THESIS

MECHANICAL AND ANTIMICROBIAL PERFORMANCE ANALYSIS OF A SHARK SKIN BIO-MIMICKED FABRIC SWATCH VIA 3D PRINTING

Submitted by

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ABSTRACT

MECHANICAL AND ANTIMICROBIAL PERFORMANCE ANALYSIS OF A SHARK SKIN BIO-MIMICKED FABRIC SWATCH VIA 3D PRINTING

Biomimicry is a long-practiced concept concerned with development of products with nature as the source of inspiration. Bio mimicked textiles is a branch of textiles wherein textile products are developed to replicate desirable elements of nature such as lotus-leaf inspired water repellent fabric, high-strength spider silk inspired by the spider web and shark skin biomimicry. The scaled texture on shark skin, known as riblet effect, exhibits drag reduction and antimicrobial properties. Accurate biomimicry of shark skin is an on-going continual process

This study is concerned with 3D printing bio mimicked fabric swatches by replication of riblet effect followed by characterization of the developed fabric swatches. The swatches were printed using Autodesk Ember photopolymer 3D printer, allowing printing of minutely detailed denticles in the base. The materials used were polycarbonate/acrylonitrile butadiene styrene (PC/ABS) and polyurethane (PU) material. PU allowed creation of rigid tough denticles embedded in flexible and soft base, indicating as a better raw material to 3D print bio-mimicked swatches for functional clothing. The PU swatches were studied further in morphological, mechanical, and antimicrobial analysis. The morphological analysis resulted into optical images exhibiting the developed texture resembling characteristic riblet effect of shark skin. Mechanical analysis in terms of tensile stress testing exhibited stronger and tougher fabric samples with thick (1.05mm) base in comparison with those having thin (0.75mm) base. Also, the mechanical analysis indicated good elastomeric properties for the fabric swatches suggesting potential in functional clothing. Lastly, the antimicrobial test conducted exhibited reduced antimicrobial

growth for samples with riblet texture against untextured samples, copper foil as well as aluminum foil thus exhibiting potential use of the textured fabric swatches as non-toxic antimicrobial material. Shark skin biomimicry through riblet effect replication has been studied majorly for hydrodynamic properties while shark skin inspired material intended for antimicrobial properties such as by Sharklet® technology is not concerned with riblet effect replication. Thus, to our best knowledge study focusing on mechanical and antimicrobial analysis of shark skin biomimicry through replication of riblet effect is missing. This study will help determine potential of shark skin biomimicry by replication of riblet effect in functional clothing, through mechanical and antimicrobial analysis.

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DEDICATION

To my dear parents Mr. Prasanna Purandare and Dr. Mrs. Rasika Purandare.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1. INTRODUCTION

Functional clothing is a significant category of textiles. Functional clothing is defined as textile and apparel products developed for a very specific application that is beyond the basic application of body covering or aesthetics (Gupta, 2011b). Being engineered for a specific function, functional clothing is also defined as "garments or accessories that protect the body or increase physical body function and hence achieve a high degree of mobility and thermal comfort" (Jana, 2011, p.380). The branch of functional clothing can be categorized into three standard classifications including sports textiles, medical textiles and protective textiles that can be further grouped into several sub-classes on the basis of design and development process (Gupta, 2011b). In recent years, there are many innovations in the design and development process of functional clothing. Examples of the innovations include e-textiles (electronic textiles), wearable technology, and smart textiles (Gupta, 2011a). One such innovation of functional clothing that is of great interest is bio-mimicked textiles. The term of biomimicry is self-explanatory, referring to imitation of natural phenomenon. Biomimicry in textile can be defined as development of textiles and apparels with elements of nature as source of information through technological advancements (Lavate, n.d.). Bio-mimicked textiles has recently become a significant part of functional clothing (Gupta, 2011a). There have been several successful bio-mimicked textiles such as lotus leaf inspired water repellent fabric, firefly inspired glowing textiles and spider silk tough yet light yarns (Lavate, n.d.).

Recently, one promising bio mimicked textile is inspired by shark skin that has many micro-scales called dermal denticles on the skin surface, thus allowing sharks to swim fast and easily in water. The dermal denticles are made up of same material as shark teeth (Das, Bhowmick, Chattopadhyay, & Basak, 2015), hence also called skin teeth. Like the teeth, dermal denticles have an inner core of pulp (made up of connective tissues, blood vessels, and serves), covered by a layer of dentine (hard calcareous material) (Kennedy, 2019). The texture on the skin surface of shark covered by dermal denticles is known as riblet effect. The riblet consists of V-shaped regularly aligned scales in a dimension of “0.2-0.5 mm wide” and “regularly spaced at distance 30-100 μm” (Das et al., 2015, p.896), creating a low-velocity channel in the riblet valleys. Therefore, shark skin is capable of reducing drag that is the force acting in direction opposite to the direction of motion of the moving body. In addition, the dermal denticles on shark skin exhibit parallel ridges that effectively prevent sharks from becoming fouled, resulting in antimicrobial and antifouling properties (Dean & Bhushan, 2010). Inspired by the structure of shark skin, recently, fabrics have been preliminarily engineered to demonstrate riblet effect and found various potential industrial applications (Das et al., 2015). First, shark-skin mimicked fabrics can be used in aircraft and watercraft to reduce drag and thus to reduce cost of transportation. Second, the use of the shark-skin mimicked fabrics can allow fast flow of liquid when they are applied on inner layer pipes. Third, the anti-microbial properties exhibited by shark skin mimicked fabrics promote their applications in medical textiles preventing bacterial growth (Das et al., 2015). Lastly, shark skin mimicked fabrics are used in functional clothing such as swimsuits, resulting in drug reduction, speed increase, and a cutting-edge competition (Lavate, n.d.).

Development of shark skin biomimicry has been attempted through various technologies such as Computer Numerical Control (CNC) through milling or molding and 3D printing (Wen, Weaver & Lauder, 2014). Recently, the 3D printing technology is considered to be an effective method if a good 3D model and a good printing material are utilized. The 3D printing method allows creation of the complexed shaped rigid denticles embedded in flexible base layer along with high efficiency (Wen et al., 2014). There have been several attempts in the past for developing shark skin mimicked fabrics. The preliminary attempts were dated back 1980's when "vinyl-film saw-tooth riblets" were used in ships and racing boats and thus to reduce cost of transportation. (Das et al., 2015, p.896). Also, there were recent claims regarding development of biomimicry of the riblet effect on fabric and its applications in swim wears (Oeffner & Lauder, 2011). However, the development of shark skin biomimicry was still preliminary and beyond practical application in functional clothing because the textured fabrics were still not appropriate to be used in designing functional clothing due to many challenges. The challenges revolved around mimicking the shark skin structure. Shark skin consist of rigid denticles with "fine rib-like surface geometries with sharp surface ridges" anchored in flexible dermis. However, most of previous studies included simplified surface roughness on rigid plates (Oeffner & Lauder, 2011, p. 785; Wen et al., 2014). The studies; Oeffner and Lauder (2011), Wen et al. (2014) and Domel et al. (2018) addressed this problem by being able to replicate the shark skin structural details in terms of complex-shaped denticles embedded in flexible base layer, in place of surface roughness. These studies based on replicating the riblet effect via 3D printing technology, made use of a multi-nozzle 3D printer and rubber alone or in combination with silicone as the printing material. The studies build strong ground in terms of 3D modelling the riblet effect with dimensions allowing efficient drag reduction followed by use of said printer and material to mimic the riblet effect. Thus, directing at future scope in terms of manufacturing methodology allowing simultaneous creation of detailed small-sized denticles embedded in the flexible base and in terms of material to better mimic required properties of the shark skin structure. On the other hand, the studies being primarily based on analysis of hydrodynamic properties of riblet effect, has created future direction for material characterization in terms of other crucial properties for possible end applications in the area of functional clothing (Oeffner & Lauder, 2011; Wen et al., 2014; Domel et al, 2018). The mechanical properties of developed 3D printed samples is crucial to ensure that the samples can withstand mechanical and environmental stresses during its potential applications as functional textile. Previous studies also indicated that the shark skin riblet effect exhibits anti-biofouling property and thus can be used as a nontoxic antimicrobial agent.

RESEARCH OBJECTIVE:

The objective of this research is to develop fabric-like shark skin bio mimicked swatches via 3D printing technology for functional clothing applications and to characterize the bio mimicked swatches in terms of microstructures, mechanical performance, and antimicrobial properties.

RESEARCH HYPOTHESIS:

3D printing method can be used to replicate micro scales that mimic the riblet effect on shark skin on a thin substrate, resulting in a fabric-like material with shark skin biomimicry. Resembling of riblet effect is expected through a detailed 3D modelling with denticle size reported to be most efficient for drag reduction (Domel et al. 2018). Autodesk Ember photopolymer printer will allow creation of small-sized detailed denticles embedded in a based layer simultaneously and polyurethane printing material will be capable of developing the denticle scales rigid enough and providing flexibility on the base due to its tough as well as elastomeric nature (Zhang, 2014).

After the fabric is 3D printed, characterization to study surface morphology, mechanical testing, and antimicrobial properties, are expected to provide useful information to determine if the fabrics have the potential in developing textile products such as functional clothing.

TASK 1: 3D modelling riblet effect based on dimensions of the denticles proved to be drag efficient in Domel et al. (2018).

TASK 2: 3D printing fabric-like material with riblet effect using Autodesk Ember photopolymer printer and polyurethane material.

TASK 3: Analysis of the developed fabric swatches in terms of; surface morphology through optical imaging, mechanical properties through tensile stress testing, and antimicrobial properties to determine the riblet effect's anti-biofouling behavior.

CHAPTER 2. LITERATURE REVIEW

The chapter begins with an overview of functional clothing, bio mimicry in textiles, and shark skin riblet effect. Further in a summary of existing work, the chapter consists of an analysis of the development and evaluation of shark skin mimic fabrics. After the summarization, the paper concludes with a critical evaluation of existing work.

2.1 Functional Clothing and Bio-mimicked Textiles

Functional clothing is a significant branch of textiles that is designed to serve a specific function ranging widely from simplifying daily life activities to saving life in extreme situations (Gupta, 2011b). Under the umbrella of three standard categories of functional clothing that is protective textiles, sports textiles and medical textiles, there are several sub-categories of functional clothing on basis of design and development process (Gupta, 2011b). The classes possess variation in terms of factors such as fiber material, user requirements, activity in which the functional clothing is expected to perform, fit requirement, and construction methodology (Gupta, 2011b). There have been innovations in design and development process, resulting in new classes of functional clothing such as electronic textiles (E-textiles), wearable technology, smart textiles, and bio-mimicked textiles (Gupta, 2011a).

Although it was until recent when bio-mimetics was recognized as a class of functional clothing, the concept of bio-mimicry has long been used by our ancestors to develop products wherein nature is the source of inspiration (Eadie & Ghosh, 2011). The term of bio-mimetics is self-explanatory, which refers to imitation of natural phenomenon. Biomimicry in textiles is the development of textile and apparel products wherein nature is the source of inspiration (Lavate, n.d.). Nature has been a major source of inspiration for textiles owing to the advanced biological structures exhibiting diverse properties and functions. An example is the basic originating principle of woven fabrics that was inspired by weaver bird's nest (Das, Shanmugam, Kumar & Jose, 2017). In fact, textiles exhibit vast opportunities of imitating elements of nature thus there have been a great number of bio-mimicked textiles. For example, a lotus leaf inspired fabric possesses water-repellency as well as dirt-repellency; a spider web inspired filament is as strong as steel but yet lightweight; and firefly inspired textiles are capable of glowing in dark through use of light emitting diodes and electronic circulations (Eadie & Ghosh, 2011). Bio-mimicked textiles are desirable because these nature-inspired products allow execution of multiple functionalities that are one of the goals in textile innovation (Eadie & Ghosh, 2011). In addition, biomimicry also performs well in energy conservation, cost effectiveness, and waste reduction (Omar, Rahman & Abdullah, 2015).

2.2 Shark Skin Riblet Effect

One bio-mimicked textiles of interest is shark skin inspired fabric. In biology, shark skin demonstrates drag reduction and anti-biofouling properties. The majority of shark species have a minute scaled texture on their skin surface made up of same material as their teeth (Figure. 2.1). The minute scaled texture on the shark skin is known as riblet effect that consists of V-shaped regularly aligned scales. The V-shaped scales have dimensions of "0.2-0.5 mm width" and are "regularly spaced at distance 30-100 μm" with adjacent scales (Das et al., 2015, p.896).

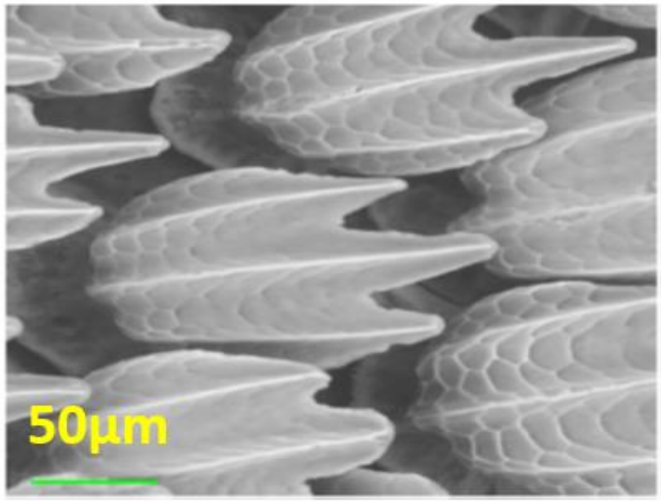

Figure 2.1. ESEM images of bonnethead shark skin riblet effect (Oeffner & Lauder, 2011).

The riblet effect is able to reduce drag when sharks swim in water, resulting in speedy mobility in water (Das et al., 2015). The riblet effect reduces the drag by reducing skin friction against the flow owing to the surface roughness (Garcia-Mayoral & Jimenez, 2011). The riblet effect exhibits anti-biofouling due to the unique scaled texture, impending bacterial growth on shark skin. The protruding scaled pattern on shark skin creates a surface roughness that is inhospitable to bacteria since the generated mechanical stress disrupts functioning of the microorganism, resulting into anti-biofouling behavior (Sharklet, n.d.). Thus, the biomimicry of shark skin demonstrates antimicrobial properties. (Das et al., 2015).

2.3 Development of Shark Skin Bio-mimicked Fabric

2.3.1 Previous attempts at shark-skin biomimicry

Replication of the riblet effect is a long-attempted biomimicry. The attempts date back to 1980 when "vinyl-film saw-tooth riblets" were attempted to be used in ships and racing boats (Das et al., 2015, p.896), providing drug reduction and hence energy saving and cost effectiveness in transportation. Later, biomimicry of the riblet effect on textiles has been experimented for multiple times. One approach of shark skin biomimicry was replication of shark skin scaled texture. There were claims regarding the successful development of accurate replication of the riblet effect on fabrics. For example, there are swim wear products commercially available in sports brands such as Speedo® and Yingfa®. A comparison study was conducted for three fabrics including; Speedo® brand's Fastskin FS II shark-skin like swimsuit fabric sample, a sample mimicking the riblet effect material and actual shark skin membrane (Figure. 2.2). The sample mimicking shark skin consisted of rubber membrane substrate decorated with a longitudinal U-shaped riblet structure made of silicone. The U-shaped structure had a dimension of 87µm height and spaced at 340µm. The actual shark skin samples were dissected from a male shortfin mako shark. The test samples were investigated in terms of surface microstructure via an environmental scanning electron microscope (ESEM) and drag reduction effect via a robotic foil flapping device. Patterned seams found on the surface of the Speedo® brand's Fastskin FS II shark-skin like swimsuit fabric were intended in the place of shark skin denticles that was expected to result riblet effect. However, consistent speed increase was not found in the commercial swimsuit fabric while the developed riblet effect material exhibited 7.2% increase in speed and the shark skin membrane exhibited 12.3% increase in speed (Oeffner & Lauder, 2011). The study concluded that swimming performance could be enhanced through bio-mimicked shark skin surface indentions and riblets accompanied by a flexible base. However, the study raised concerns on the Speedo® brand's Fastskin FS II swimsuit due to lack of resemblance with actual riblet texture and due to its incapability in improvement of swimming performance.

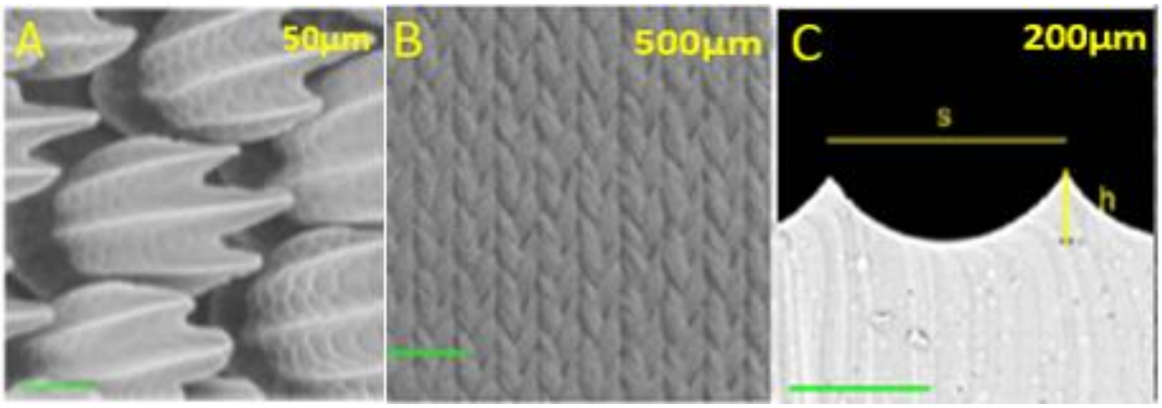

Figure 2.2. ESEM images of (A) Shark skin with scale bar 50µm, (B) Speedo® brand's Fastskin FS II shark-skin like swimsuit fabric sample with scale bar 500µm and (C) Riblet structure with scale bar 200µm (Oeffner & Lauder, 2011).

Another approach was development of textural surfaces inspired by shark skin, but not a replication. A successful example of commercialization is the Sharklet® technology that manufactures anti-microbial shark skin-inspired textural surfaces. The product specifically intended for use as antimicrobial material, is a material with patterned texture inspired by the shark skin scaled texture. The material consists of a diamond-shaped protruding micropattern of dimensions 3 microns tall and 2 microns wide, creating a surface roughness disrupting the normal functioning of microorganisms thus impending bacterial growth (Figure. 2.3). The Sharklet® materials demonstrate superior antimicrobial properties and have been used in many applications such as wound dressing, urinary catheter, phone devices, and computer screen through variation in the micropattern dimensions.

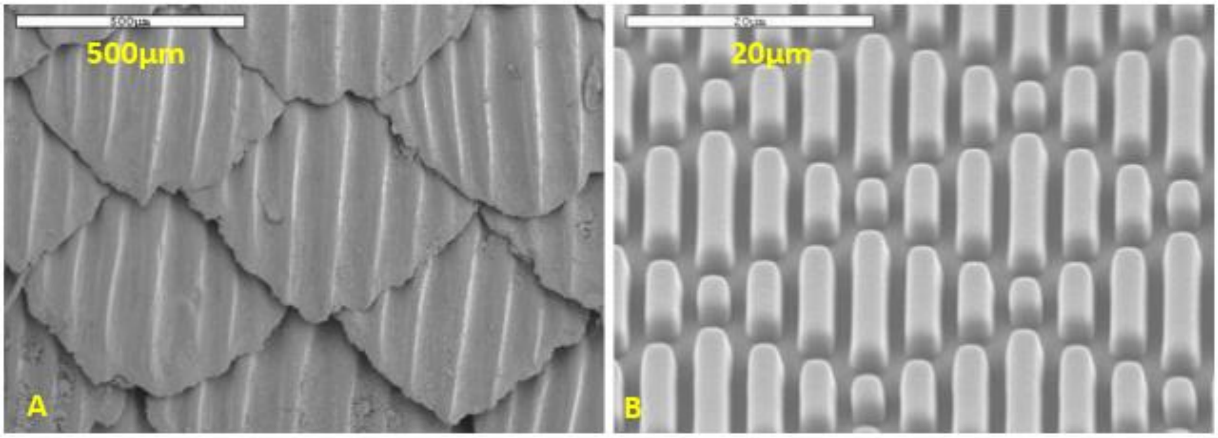

Figure 2.3. Images of (A) Shark skin riblet effect and (B) Sharklet® micropattern material (Sharklet, n.d.).

2.3.2 Methods of developing shark-skin bio mimicked fabrics

Several methods have been adopted to develop shark skin bio mimic fabrics, such as computer numerical control (CNC) milling or molding technology, and 3D printing technology. In CNC, coding is developed to run specialized machines or assemblies through either milling or molding process. The CNC milling is concerned with use of rotary cutters to obtain required shape (Jung & Bhusan, 2009) while the CNC molding is concerned with development of templates to obtain desired structure (Bechert, Bruse & Hage, 2000). In a comparison, 3D printing technology was previously reported as an effective method in creating accurate riblet effect on fabrics (Wen et al., 2014). The 3D printing technology is concerned with development of a 3-dimensional product based on a desired 3D model usually by deposition of printing material in a layer-by-layer process. The shark skin structure consists of rigid denticles embedded in flexible dermis (Meyer & Seegers, 2012; Wen et al., 2014), The surface structure requiring both rigidity and flexibility was successfully created using 3D printing technology. Further, 3D printing technology can replicate the structural details with improved accuracy and does the replication in a timely manner (Wen et al., 2014).

The development of riblet effect on fabric using 3D printing technology in the work of Wen et al. (2014) involved high resolution CT-scan images of freshly obtained shark skin followed by computer modeling of the scaled texture. Then the developed computer model was used to 3D print fabric using 3D printer. A multiple nozzle 3D printer (Object Connex500 3D Printer) was utilized so as to produce a flexible membrane substrate and subsequent rigid denticles on the surfaces. Domel et al. (2018) studied three distinct sizing for the denticles in terms of drag reduction properties. The dimensions of denticles in reference to Figure. 2.4 included as follows, for the smallest sizing set; "$lc/ls = 1.37$, $lc/lr = 1.25$, $h1/h2 = 1.2$, $lc/h1 = 1.67$, and $Ss/Sl = 1$ constant" resulting into denticles of smallest size set with h1=1.26mm, lc=2.1mm, lr=1.68mm, and ls=1.53mm (Domel et al., 2018, p.3). While for two remaining sizing sets, dimensions were increased by 1.5x and 2x. The study concluded the smallest set of dimensions to be most efficient for drag reduction. Abid et al. (2017) enumerates an angle multiplexed optical 3D printer that can print bio-mimicked textures using "two-beam interference lithography with angle-multiplexed exposures and scanning" (Abid et at., 2017, p.1). This technique is principally based on laser-printing technique wherein complex bio mimic objects can be printed by using a lateral scanning set-up with multiplexed exposure at a controlled dose (Abid et al., 2017).

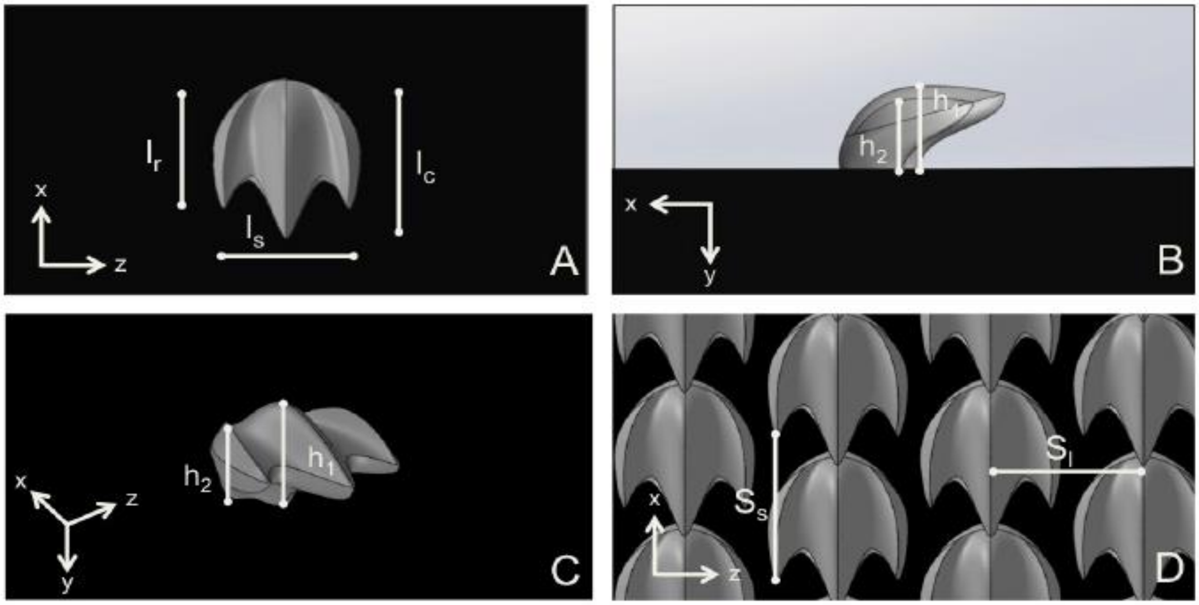

Figure 2.4. (A)Top view, (B) Side view, (C) Isometric view of 3D modelled denticles and (D) Riblet effect 3D model (Domel et al., 2018).

The final products in 3D printing are highly dependent with printing raw materials, primarily polymers. The selection of raw material should be specific to the end use of the final products. The material that has been commonly employed for the biomimicry of shark skin is 100 % rubber or rubber in combination with rigid thermoplastic polymers such as silicone for creation of material with rigid denticles and flexible base (Oeffner & Lauder, 2011; Domel et al., 2018).

2.4 Characterizations of Bio-mimicked Shark Skin Fabric

The characterization of shark skin bio-mimic fabrics is crucial to bio-mimic fabrics for the specific applications of functional clothing because it is required to ensure that the developed clothing is capable of fulfilling the purpose it is designed for. The primary characterizations of shark skin biomimicry are microstructure imaging, drag reduction measurement, antimicrobial properties, and mechanical properties.

2.4.1 Microstructure analysis

As shown in Figure 2.2, Oeffner and Lauder (2011) opted for high resolution imaging to compare the textures of real shark skin, Speedo brand's claimed shark-like swimsuit and fabric sample with shark skin like rubber denticles (Oeffner & Lauder, 2011, p. 786). In the study of Oeffner and Lauder (2011), the riblet texture involved U-shaped riblets of 87µm height and spaced at 340µm. Further, Wen et al. (2014) also described the use of microstructure analysis in order to validate a fabric with texture similar to shark skin texture. The 3D printed riblet texture in Wen et al. (2014) consisted of linear array of denticles with three surface ridges and three prongs, each denticle of height 1.4mm, length 1.87 mm and width 1.55 mm, spaced at 1.93mm from mid-center of denticles along stream-wise direction and 2.16 mm along lateral direction (Figure. 2.5).

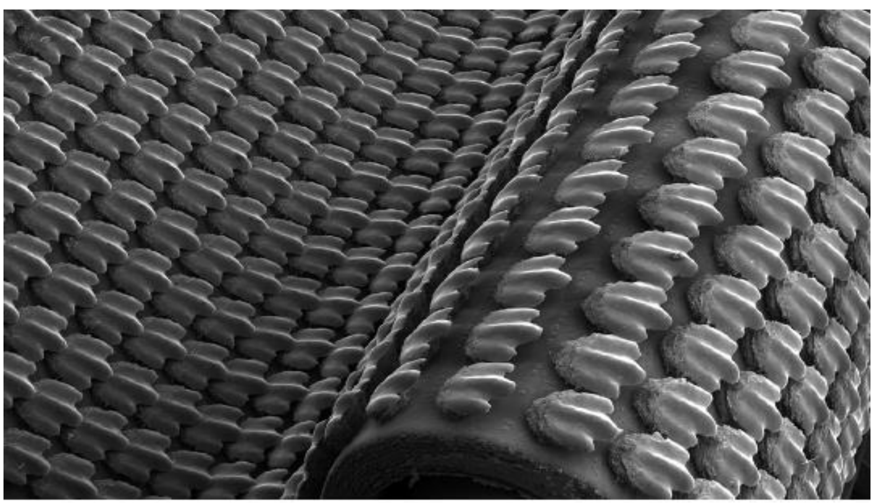

Figure 2.5.SEM image of 3D printed riblet effect with each denticles of height 1.4mm, length 1.87 mm and width 1.55 mm (Wen et al., 2014).

2.4.2 Self-Propelled Swimming Speed (SPS)

Drag reduction is a distinguishable property demonstrated by the shark skin. Therefore, drag reduction has been studied for shark skin bio-mimic materials previously developed. For example, self-propelled swimming (SPS) speed was measured in shark skin bio-mimic fabrics for potential applications in functional clothing, especially swim wear. The self-propelled swimming (SPS) speed is defined as the forward motion done with use of one's own force. A customized SPS apparatus called "flapping foil robotic device" was customized to measure drag reduction in a substrate material (Oeffner & Lauder, 2011, p.785). The flapping foil robotic device required placement of the testing sample to be tested in foils and allowed measurement of swimming speed of the foils through their own tractive power in presence of fish-like movement created by the device. (Oeffner & Lauder, 2011). The device measures the self-propelled swimming speed of the testing sample through the generation of vertical motion and rotational motion at a range of frequencies. The customized flapping foil device has been used to test hydrodynamic properties of bio mimicked shark skin fabrics (Oeffner & Lauder, 2011; Wen et al., 2014; Domel et al., 2018). The test results indicate efficient speed increase by bio-mimicked shark skin fabrics, with 6.6% increase in swimming speed and 5.6% decrease in swimming energy under specific kinematic conditions in comparison with smooth control sample (Wen et al., 2014, p.1660).

2.4.3 Anti-microbial properties

Antimicrobial textiles inhibit growth of micro-organism. The anti-microbial properties of fabrics have been greatly utilized in textile end products such as medical textiles, sports clothing, personal hygiene textiles, upholstery textiles, automotive textiles, and geotextiles (Hofer, 2006). The unique riblet effect of shark skin exhibits antifouling behavior by impending bacterial growth due to the surface roughness generated by the protruding scaled pattern creating non-suitable environment for bacterial growth. Hence bio-mimicked shark skin fabrics can be used as antimicrobial textiles (Das et a., 2015).

A common and conventional method of developing antimicrobial textiles is the use of antimicrobial agents such as quaternary ammonium, triclosan, and metallic salts. The application of these antimicrobial agents occurs either during the spinning process or after the yarn is formed prior to weaving (Shahidi & Wiener, 2012). Toxicity is a major concern when these chemicals are used to introduce antimicrobial properties to textiles. Non-toxic methods are in great need in developing antimicrobial textiles (Derin, 2017). Shark skin biomimicry has been reported as an effective and non-toxic approach of generating antimicrobial properties in textiles Mann et al. (2014). The bio mimicked shark skin fabric can play a crucial role in functional clothing. For example, Sharklet® technology manufactures anti-microbial material with a unique surface pattern inspired by shark skin. The method of evaluating antimicrobial properties of shark skin biomimicry developed in Mann et al. (2014) was to specifically test the efficacy of preventing microbial growth. Mann et al. (2014) reported a comparative analysis of colonization of methicillin-sensitive Staphylococcus aureus (MSSA) and methicillin-resistant S. aureus (MRSA) between the bio mimicked surface, control surface and copper being an anti-microbial agent. The study suggested that the bio mimicked surface reduced MSSA by 97% and MRSA by 94% in comparison with un-textured and smooth surfaces, wherein copper had no significant effect on MSSA but reduced MRSA by 80%.

2.4.4 Mechanical properties

Dizon, Espera, Chen, and Advincula (2018) suggested that it is crucial to test mechanical properties of 3D printed material. The previous studies of shark skin biomimicry have majorly focused on hydrodynamic properties of the riblet effect and measured drag reduction due to the 3D printed riblet effect. However, in the area of functional clothing it is necessary to quantify the mechanical properties of the textile since they are expected to exhibit certain properties to perform predetermined functionalities (Gupta, 2011a). Thus, it would be necessary to analyze the mechanical properties of the developed bio-mimicked fabric since it finds numerous end application as functional wear.

A common approach of analyzing the mechanical properties of a material is the tensile testing. The tensile testing is based on the principle of applying a controlled tensile force on the test specimen and subsequently to study the specimen's response under stress (Tensile testing, n.d.). Tensile testing allows the study of the mechanical properties such as breaking stress, breaking elongation, yield point and Youngs modulus/tensile modulus (Zhang, 2014). A stress-strain curve obtained in tensile testing can be used to measure these mechanical properties including breaking stress, the tensile modulus, and sure of toughness (Zhang, 2014). These properties are important of durability for functional clothing. For example, ballistic protection is expected to have high strength and high tensile modulus providing stiffness, thus involves use of polymers such as Kevlar and carbon fibers. While sports functional wear such as swim wear, scuba wear and cycling shorts are expected to exhibit high toughness and flexibility thus involves use of elastomeric polymers (Gupta, 2011a; Gupta, 2011b). Figure 2.6A exhibits stress-strain curves of various polymers and figure 2.6B exhibits stress-strain curves of different textile polymer fibers including spider silk that is a textile biomimicry known for its high strength and high flexibility.

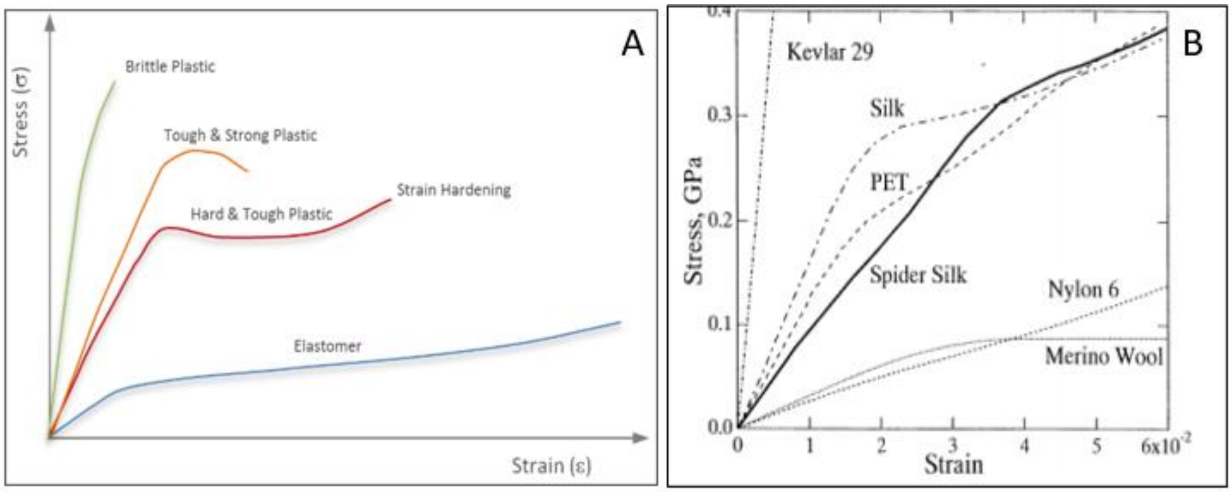

Figure 2.6. (A) Stress-strain curves of various polymers (Stress-strain behavior of polymers, n.d.) and (B) Stress-strain curves of various textile fibers (Ko et al., 2001).

2.5 Critical Evaluation of Existing Work

The positive aspect of the analyzed literature is that information regarding the concept of functional clothing as well as the concept of biomimicry in textiles was found in abundance. The analyzed literature also explores the shark skin biomimicry attempts made in past along with its methodologies. Also, the empirical studies that were analyzed involved in detail explanation about the testing methodologies employed for shark skin biomimicry.

Shark skin consists of rigid denticles embedded in linear pattern inside flexible dermis with each denticle consisting of three prongs and three surface ridges (Meyer & Seegers (2012); Wen et al. (2014). It is this structure that primarily determines drag reduction and anti-microbial properties demonstrated in shark (Das et al., 2015). Various approaches have been used in past to attempt shark skin biomimicry. The commercially available Sharklet® technology manufactures a shark skin inspired patterned material instead of replicating the actual riblet effect, resulting in superior antimicrobial performance. The diamond shaped micropatterns are about 3 microns tall and 2 microns wide (see Figure 2.2). Because the objective of Sharklet technology focuses on the development of antimicrobial materials without the application of toxic antimicrobial agents, the Sharklet technology is neither a replication of shark riblet effect nor a biomimicry of drag reduction. There is no evaluation available of drag reduction of sharklet materials. On the other hand, the replication attempts in developing shark skin fabrics via 3D printing technology was made on promoting drag reduction that could be helpful in functional clothing such as sport wear.

However, to our best knowledge antimicrobial and mechanical analysis of shark skin fabric intended for functional clothing has not been performed. Thus, literature review suggests that the mechanical and antimicrobial properties of a 3D printed shark skin mimic fabric are missing and lack in determining its potential use in the area of functional clothing. Biomimicry of shark skin is an ongoing continual process, especially in terms of replication of the riblet effect, thus directing at future scope in terms of manufacturing methodology allowing simultaneous creation of detailed small-size denticles embedded in the flexible base and in terms of material to better mimic required properties of the shark skin structure.

CHAPTER 3. MATERIALS AND EXPERIMENTS

3.1 Materials

Rigid polycarbonate/acrylonitrile butadiene styrene resin and elastomeric polyurethane photopolymer resin will be used as printing materials in the 3D printing to create shark skin bio-mimic fabric swatch. The polycarbonate/acrylonitrile butadiene styrene resin will be purchased from Chroma Strand Labs and the polyurethane photopolymer resin will be purchased from Colorado Photopolymer Solutions.

In the antimicrobial testing, *E. coli* ATCC25922 pellets will be purchased from ATCC. The Lysogeny Broth (LB) culture medium preparation involves use of tryptone, NaCl, yeast extract and DI water. All these required ingredients will be purchased from Fisher Scientific.

3.2 Development of Bio-mimicked Shark Skin Fabric

Shark skin bio-mimic fabrics will be developed via 3D printing technology using a method adopted from Wen et al. (2014). The method includes first developing a 3D model using 3D modelling software's such as Catia and Solidworks followed by 3D printing the structures that mimic shark skin.

3.2.1 3D modelling

In order to develop the 3D model of the shark skin, the software that will be used are; Onshape, MeshMixer, Catia, Fushion 360 and Cura. The 3D modelling will involve development of a single riblet model that mimics single scale of shark skin and then development of a model with line array of those riblets. During 3D modelling, graphic images of shark skin from

Wen et al.'s study (2014) will be used as a visual reference to build the required riblet 3D models for this research.

3.2.2 3D printing

After the model is developed, 3D printing will be done with a printer that can print a fabric structurally similar to shark skin that is a base layer with riblets embedded in it simultaneously. The prototyping will involve use of FDM (Fused Deposition Modelling) printer While, printing of final fabric swatch with riblet effect will be carried out by the 'Autodesk Ember photopolymer printer' in the Idea2Product (I2P) lab at Colorado State University. The reason for selecting this printer for the final product is due to its ability to simultaneously print the base layers with embedded riblets and also because of its ability to print detailed very small objects. Thus, the 3D printing will result into fabric swatches with riblet effect texture, including variation in base thickness (0.75mm and 1.05mm) to allow comparative analysis during characterization.

3.3 Fabric Characterizations

3.3.1 Morphological analysis

The biomimicry is said to be accurate when the fabric texture is similar to the shark skin texture. Thus, the evaluation will be done through optical imaging of the developed fabric swatch.

3.3.2 Analysis of mechanical properties

The mechanical properties of developed swatches will be studied by tensile stress testing using Instron low force universal testing system, Instron Corporation, Norwood, MA. This will allow determination of the developed product's applicability in functional clothing. The testing will be conducted according to ASTM D882 (Tensile properties of thin plastic sheeting), which is specifically used to determine tensile strength for thin plastic sheets with thickness less than 1mm. The expected data through this test will provide information regarding following properties of the fabric; Youngs modulus, yield point, plastic deformation strain, ultimate stress, failure stress, and elastic area.

The test samples will include untextured swatches and textured swatches (with riblet effect) each with base thickness of 0.75mm and 1.05mm. The test will be conducted in triplicate for each test sample for each base thickness allowing a comparative analysis in terms of textured verses untextured samples and in terms of variation in base thickness.

3.3.3 Analysis of anti-microbial property

The anti-microbial properties of the developed fabric swatch will be tested following the test protocol stated in Mann et al. (2014). The test will involve cultivation of the microorganism *E. coli* in LB liquid media followed by formation of its bacterial sub-culture. The samples will then be treated with bacterial sub-culture at set time and conditions followed by a PBS sterilization. Lastly, agar contact plate method will be used to observe the bacterial growth. Comparative testing will be conducted between a control sample (film with no texture), bio-mimicked samples for both base dimensions (1.05mm and 0.75mm base thickness) and aluminum as well as copper foil. The test will be conducted five times at periodic intervals.

3.4 Data Analysis

In case of morphological analysis to gauge the accuracy of the biomimicry of the 3D printed fabric samples, evaluation will involve the visual representation of the fabric samples using ESEM or optical microscope and cross-reference with the graphic images of shark skin available in Wen et al.'s (2014) published work.

In case of analysis of mechanical properties, "predetermined instrumentation will be used to yield statistical data to answer the drawn close ended research questions" (Creswell & Creswell, 2017, p. 18). Thus, the data would be analyzed through quantitative research methods and mainly through plotting tensile stress-tensile strain curves.

Lastly, in case of anti-microbial property testing, the conclusions will be derived in terms of the comparative observations obtained. The developed scaled fabric swatch will be said to have anti-microbial property if the growth of the micro-organism is lesser in comparison with the control sample as well as outsider sample (copper foil and aluminum foil).

Baxter and Jack (2005) state several ways in which the authenticity, credibility and reliability of a research can be increased. Some of those methods are clear and concise research questions and propositions, collection of data from multiple sources, collective analysis of data, triangulation of data sources, having other members audit-code the analysis, maintenance of field notes, peer examination of data and double coding. In order to increase credibility of my data, after completion of testing and analysis of obtained results the data would be audited by a fellow graduate student who is trained in fiber science and quantitative research methods.

CHAPTER 4[1]. RESULTS AND DISCUSSION

Summary: Biomimicry of shark skin has been recently found with great potential in the application of functional clothing and textiles. This study is concerned with 3D printing bio mimicked fabric swatches by replication of microstructures of shark skin and characterization of the mechanical and antimicrobial properties of the fabric swatches. The fabric swatches with denticles arranged in linear array to form riblet effect were developed using polycarbonate/acrylonitrile butadiene styrene (PC/ABS) and polyurethane (PU) in a 3D photopolymer printer. The printer and material selection for final product allowed printing of flexible base membrane and minutely detailed rigid denticles simultaneously. The PU swatches were significantly softer and more flexible than the PC/ABS swatches, suggesting that the PU was a better raw material for developing bio mimic fabrics for functional clothing using 3D printing. The PU swatches were studied further in morphological, mechanical, and antimicrobial analysis. The morphological analysis via an optical microscopy suggested a riblet texture that resembled the characteristic microstructures of shark skin. The mechanical analysis exhibited tougher and stronger fabric swatches with 1.05mm base thickness in comparison with those with 0.75mm base thickness. Good elastomeric properties were found in the fabric swatches, suggesting a good potential in functional clothing applications. Lastly, the antimicrobial test exhibited reduced antimicrobial growth for samples with riblet texture against untextured samples, copper as well as aluminum foil thus demonstrating ability of the textured swatches as non-toxic antimicrobial material. The results suggest that the PU fabric swatches with a riblet effect printed using a 3D printing have a great potential in functional clothing applications.

[1] This chapter is formatted in a manuscript for future publication

Keywords: Shark skin biomimicry, 3D printing, Riblet effect, Tensile stress test, Antimicrobial property.

4.1 Introduction

Functional clothing is a category of textiles concerned with engineering of textile and apparel products for a specific function, which is beyond the basic application of body covering or aesthetics (Gupta, 2011b). One major category of functional clothing is bio-mimicked textile that are developed by imitating elements of nature or using nature as the source of inspiration (Gupta, 2011a; Lavate, n.d.).

There are many examples of successful bio-mimicked textiles since our ancestors developed basic originating principle of woven fabrics that was inspired by weaver bird's nest (Das et al., 2017). Modern examples include water-repellent lotus leaf inspired fabric, firefly inspired textile capable of glowing in dark, super tough yet super light spider silk, and shark skin inspired textiles (Lavate, n.d.). Recently, shark skin biomimicry has been of great interest in textiles, especially functional clothing such as sport wear. Shark skin consists of V-shaped regularly aligned rigid denticles with three surface ridges and three prongs, embedded in flexible base dermis (Oeffner & Lauder, 2011; Meyer & Seegers, 2012). The denticles consists of inner core pulp and outer layer of dentine, hence termed as skin teeth (Das et al., 2015; Kennedy, 2019). The skin structure referred as riblet effect exhibits superior drag reduction and anti-biofouling properties that open a wide range of potential end applications for a shark skin bio-mimicked textile (Das et al., 2015).

Biomimicry of shark skin in engineering applications could be dated early in 1980's when ships and racing boats were covered with "vinyl-film saw-tooth riblets" to reduce transportation cost (Das et al., 2015, p.896). Biomimicry of shark skin can also serve largely for antimicrobial purpose. An excellent example is a commercial material by Sharklet® technology inspired by shark skin, promoting antimicrobial properties without toxic treatment. In the field of textiles, there were recent claims regarding replication of riblet effect for swim wear application by brands such as Speedo®. However, the attempts by the commercial brands such as Speedo® at biomimicking shark skin had some drawbacks due to lack of replication of the complex riblet effect resulting into no improvement in swimming performance thus beyond practical application in functional clothing (Oeffner & Lauder 2011).

Methodologies of creating shark skin biomimicry include computer numerical control (CNC) milling, CNC molding, and 3D printing. Recently, 3D printing is considered to be an effective method for replicating the riblet effect. The complex-shaped minutely detailed denticles with rigid structural properties embedded in flexible base can be created simultaneously in comparatively lesser time span using 3D printing (Wen et al., 2014). Domel et al. (2018) studied the variation in the sizing of riblet effect and its effect on drag reduction properties. The study involved three size sets: the smallest size as shown in Figure. 2.4 involved the dimensions as; "$lc/ls = 1.37$, $lc/lr = 1.25$, $h1/h2 = 1.2$, $lc/h1 = 1.67$, and $Ss/Sl = 1$ constant" wherein lc is 2.1mm (Domel et al., 2018, p.3). While for the two remaining sizing sets, dimensions were increased by 1.5x and 2x. The study concluded the smallest set of dimensions with each denticle of height 1.26 mm, length 2.1mm and width 1.52mm spaced equidistant along stream-wise and lateral direction to be most efficient in drag reduction of the 3D printed materials.

The studies concerned with bio mimicry of shark skin through replication of riblet effect focus on drag reduction properties of the riblet effect (Oeffner & Lauder, 2011; Wen, Weaver & Lauder, 2014; Domel et al., 2018). However, the mechanical and antimicrobial properties of shark skin biomimicry attempting at replication of riblet effect for the application of functional clothing, have not been previously understood. On the other hand, Sharklet® technology manufactures a material with protruding micropatterned texture, inspired by shark skin, specifically intended to serve as nontoxic antimicrobial material. Because of its specifically intended purpose, the material by Sharklet® technology is neither a replication of shark skin riblet effect nor does it focus on the hydrodynamical properties of shark skin biomimicry. Therefore, there is very limited knowledge about mechanical and antimicrobial performance of shark skin bio-mimic fabrics to determine its potential in functional clothing.

This work was concerned with utilization of 3D printing technology for development of fabric-like material consisting of riblet effect and material characterization through mechanical testing and antimicrobial testing, providing a better understanding if shark skin bio mimicked fabrics potentially can be used for functional clothing. Replication of riblet effect was attempted through; first, a 3D modelling based on Domel et al. (2018) study stating the dimensions of riblet effect that prove to be drag efficient. Second, the Autodesk Ember photopolymer 3D printing technology was used to allow simultaneous creation of detailed small-sized denticles embedded in base layer. Lastly, elastomeric polyurethane resin was used in 3D printing, resulting in the denticles to be rigid due to tough nature of polyurethane and at same time flexible base due to inherit elastomeric property of the polymer (Zhang, 2014). These enhancements in development of fabric like material with riblet effect are expected to closely resemble shark skin and thus have potential use in functional clothing.

The developed fabric swatches with riblet effect were characterized in the area of morphological, mechanical and antimicrobial performances that are significant for functional clothing. First, the morphological analysis through optical images of the samples allowed verification of the developed riblet effect by cross-referencing with the images of shark skin from Wen et al. (2014). Second, the samples were analyzed in terms of mechanical properties. The tensile stress test allowed comparative analysis between the samples with riblet effect and control samples, along with variation in base thickness. The results indicated tougher and stronger samples for 1.05mm base thickness and comparatively brittle samples for 0.75mm base thickness. Also, the tensile stress testing exhibited potential of samples in functional clothing applications requiring elastomeric properties. Lastly, the developed samples were tested for anti-microbial properties by following the immersion assay anti-microbial test stated in Mann et al. (2014) to explore the use of antimicrobial properties in area of functional clothing. The antimicrobial testing exhibited lowest bacterial growth for the textured samples in comparison with aluminum foil to be highest, followed by copper foil and then control samples.

4.2 Materials and Experiments

4.2.1 Materials

In terms of 3D printing the replication of shark skin riblet effect, Polycarbonate/ Acrylonitrile Butadiene Styrene (PC/ABS) resin and elastomeric polyurethane photopolymer resin were used as printing materials. The polycarbonate/acrylonitrile butadiene styrene resin was purchased from Chroma Strand Labs and the polyurethane photopolymer resin was purchased from Colorado Photopolymer Solutions.

For the characterization of the antimicrobial properties of the riblet effect, *E. coli* ATCC25922 pellets were purchased from ATCC. Tryptone, NaCl, yeast extract required for Lysogeny Broth (LB) culture medium preparation were purchased from Fisher Scientific.

4.2.2 3D printing models

The first step towards development of a bio-mimicked shark skin fabric using 3D printing methodology was development of 3D models. The 3D models for shark-skin biomimicry were developed using reference images of shark skin from previous studies (Oeffner and Lauder (2011) and Wen et al. (2014) (Figure 2.1).

For this study, initially a 3D model of single denticle was developed using various software’s such as Meshmixer, 3D builder and Catia. Followed by successful modelling of a denticle with three surface ridges and three prongs as outlined in the referenced images of shark skin, the modelling of riblet effect was completed by arranging multiple denticle in linear array, pointing in same direction embedded in a based layer. Modelling of denticles and riblet effect was entirely based on dimensions proven to be hydrodynamically efficient by Domel et al. (2018) wherein each denticle measured; height 1.26 mm, length 2.1mm and width 1.52mm. The thickness of the base layer was varied, resulting in a thin sample of 0.75 mm and a thick sample of 1.05mm.

4.2.3 3D printing

Prototypes in form of singular shark denticles were developed using a Lulzbot Mini Fused Deposition Modelling (FDM) 3D printer. In the FDM printer, multiple layers of molten plastic were deposited layer-by-layer through a head onto the bed to form the modelled product (What are the advantages of FDM technology, n.d.). The material used in the FDM printing was PC/ABS resin. Later, an Autodesk Ember photopolymer printer was used to print the swatches with riblet texture with better accuracy than the FDM printer. PC/ABS resin and PU resin were used to develop swatches with riblet effect. In a comparison, the elastomeric polyurethane resin being able to develop flexible and softer fabric-like swatches with riblet effect. Thus, using the same elastomeric resin, control samples that were swatches without the riblet texture were also developed. The PU swatches with riblet effect as well as without riblet effect, were made with variation in base thickness (1.05mm and 0.75mm) to be characterized in morphological, mechanical and anti-microbial analysis. The printing technology uses the photopolymer resins that are photo-polymerized using an ultraviolet light source in the printer. When the UV light falls on the photopolymer resins, the photo initiator activates a conversion of "light energy to chemical energy" allowing "the oligomer (also referred to as binders) and monomer mixture to form three-dimensional polymer networks" (Molitch-Hou, 2016, para. 6).

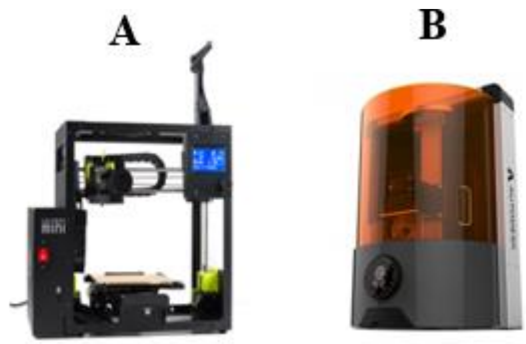

Figure 4.1. (A) Lulzbolt Mini FDM 3D printer and (B) Autodesk Ember photopolymer 3D printer.

4.2.4 Morphological analysis

The morphological analysis was made using a 40x-1000x optical microscope. The microscopic images were captured and analyzed using the software motif image plus 2.0. The biomimicry was analyzed by cross-referencing the images of actual shark skin and the images of riblet effect developed in previous studies.

4.2.5 Mechanical property analysis

The mechanical analysis of the developed fabric swatches was conducted using tensile tester (Instron 3400 series-low force universal testing system with testing capacity 2N -50N, Instron Corporation, Norwood, MA) using ASTM D882 standard (Tensile properties of thin plastic sheeting). The test involves application of a controlled force and measurement of test sample's response to the applied stress (Tensile testing, n.d.). The test was conducted for both bio-mimicked shark skin (textured) samples and untextured samples, each with 1.05mm base thickness and 0.75mm base thickness. The test was carried out in triplicate for each base dimension. The results obtained in the form of stress-strain curve provided measurements of Youngs modulus, yield point, plastic deformation strain, failure stress, and elastic area of the samples.

4.2.6 Antimicrobial property analysis

Antimicrobial test referring the 'immersion assay' test protocol stated in Mann et al. (2014) was performed on 3D printed samples including (1) untextured PU control sample, (2) thick PU textured sample, and (3) thin PU textured sample. Aluminum foil and copper foil were also used for comparison purposes. The samples were 1 cm X 1 cm. Five tests were repeated for each sample in the testing.

In the preparation of bacterial strains, Liquid LB media was prepared by combining 5 g of tryptone, 2.5 g of NaCl, and 2.5 g of yeast extract in 500 ml of DI water and sterilized by autoclaving at 250° F at 23 psi pressure for 20 min. Then, two premade Escherichia coli (*E. coli*) ATCC25922 pellets (1.0 X 10^4 cfu/pellet) were hydrated in the hydration liquid at 37° C for 30 minutes. After hydration, the mixture was vortexed and added to 250 ml liquid LB medium and incubated in a shaker incubator at 37°C for 24 h. After 24 h of incubation, the *E. coli* culture was collected and used for subsequent tests with 3D printed swatches. Since bacterial sub-culture was required, strains were sub-cultured into LB with 1:100 dilution followed by 4 hours in the shaker incubator at 37°C

For the soft agar plates required in testing, 0.9 g of LB agar powder was mixed with 100 ml LB medium and autoclaved at 250° F at 23 psi pressure for 20 min. The autoclaved mixture was poured into 4 sterile Petri dishes and kept in the room temperature until the liquid mixtures became solid. Finally, the Petri dishes with soft LB agar were stored in the refrigerator at 4° C to be used for subsequent experiments.

Prior to testing the surface properties of the test samples, the samples were adhered firmly on separate sterile petri dishes with textured side face-up. The samples were first sterilized with 95% Ethanol for 10 minutes followed by 3* rinse with deionized water and then were allowed to dry (Mann et al. 2014).

First, the samples were submerged in the dishes with bacterial sub-culture for 1 hour at room temperature. Second, the bacterial sub-culture was removed and the samples in dishes were rinsed with PBS for 3 times each for 10 seconds while simultaneously rotating the dishes. After the final rinse and drain, the samples were allowed to dry at room temperature for 1 hour. Third, the prepared soft agar plates were pressed onto the tested samples for 5 seconds without any air bubbles in between the two surfaces. The agar plates were then incubated for 24 hours at 37 ºC, after which the bacterial growth was measured and analyzed.

4.3 Results and Discussion

4.3.1 3D modelling

Initially, shark denticle 3D models were developed with Meshmixer and 3D Builder that exhibited a rough idea of the expected structure before generating final model with desired details (Figure. 4.2 A). Following the rough 3D models of the shark denticle, a 3D model of a shark denticle with the details (three ridges and pointing prongs) as exhibited in the image of shark skin was developed using Catia software (Figure. 4.2 B-E). The modelling was based on dimensions proven to be hydrodynamically efficient in Domel et al. (2018), with each denticle of height 1.26 mm, length 2.1mm and width 1.52mm (Figure. 4.3). However, there lies a difference in terms of developed model verses the shark skin riblet effect in terms of pointing of prongs (Figure. 2.1). Although the hydrodynamically proven set of dimensions stated in Domel et al. (2018) were followed, the dimensions defining the prongs in terms of distinctive between the three prongs was not specifically stated and hence the current models produced prongs without distinctive. Further, a 3D model of riblet effect with the denticles arranged in linear array similar to the arrangement on shark skin was developed wherein denticles spaced equidistant at 2mm along stream-wise and lateral direction (Figure. 4.4 A-D). The arrangement involved placement of denticles spaced equidistance from the center in lateral- and stream-wise direction with ridges faced in one direction. Two models were developed with variation in base thickness as 1.05mm and 0.75mm.

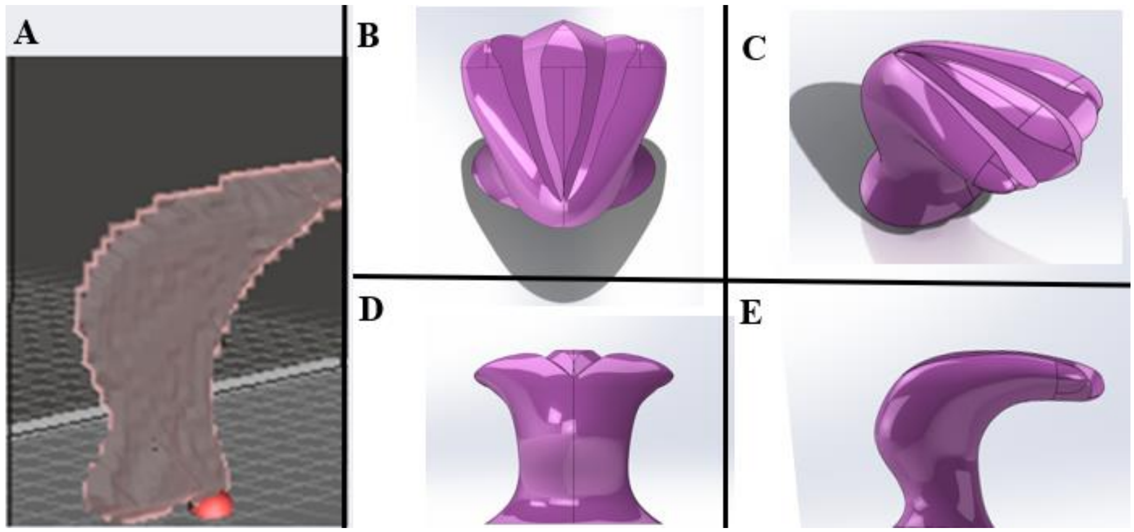

Figure 4.2. (A) Rough 3D model of shark denticle developed using Mesh mixer software and 3D Builder software. Final 3D model of shark denticle developed using Catia software (B) Orthogonal top view, (B)Isometric view, (D) Orthogonal front view and (E) Orthogonal right-hand view of the model.

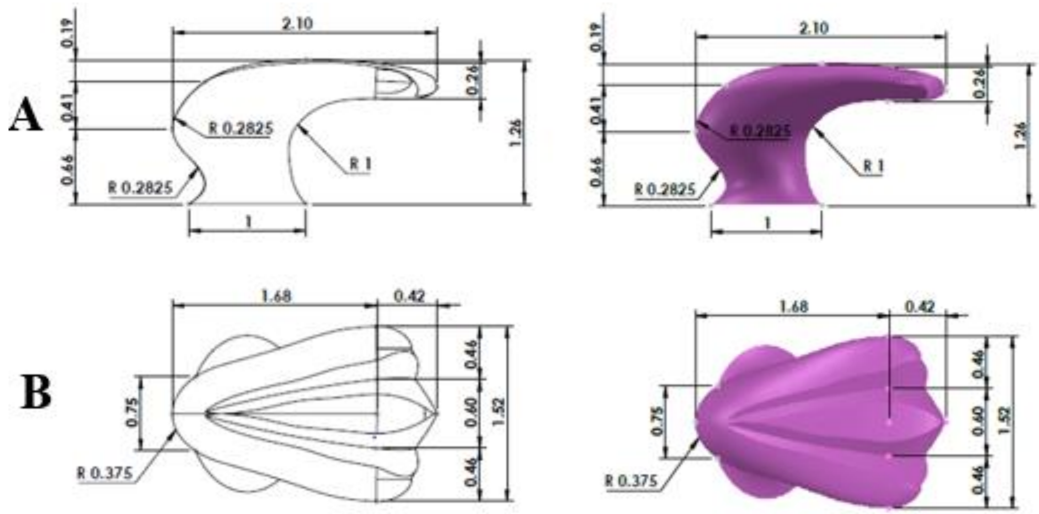

Figure 4.3. Graphical presentation of 3D model of shark denticle using AutoCAD software; (A) Orthogonal side view of the model with dimensions and (B) Orthogonal top view of the model with dimensions.

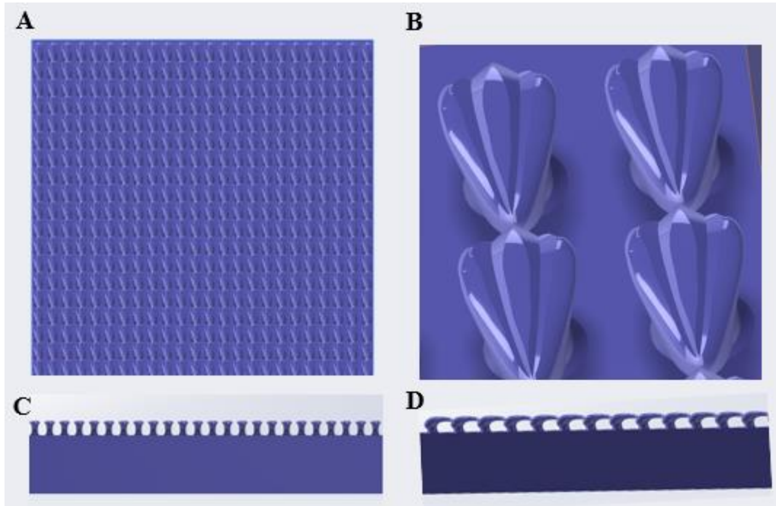

Figure 4.4. (A) Orthogonal top view, (B) Magnified image of top view, (C) Orthogonal front view, and (D) Orthogonal side view of the riblet effect model developed using Catia software.

4.3.2 3D printing

The rough 3D model as well as final detailed 3D model were used to develop prototypes; enlarged denticles of varying sizes using the FDM printer and PC/ABS material to verify the developed denticle 3D model (Figure 4.5 (A) and (B)).

The Autodesk Ember photopolymer printer was used for making films with riblet effect because it was able to print the minutely detailed denticles embedded in base layer (membrane) simultaneously and because of its ability to print detailed objects at small size scales. PC/ABS was used in the photopolymer printer, resulting in stiff swatches with riblet effect (Figure 4.5 (C)). PU resin was also used in the same printer, resulting in flexible swatches with riblet effect. The material that has been widely used in shark skin biomimicry is rubber alone or in combination with some rigid thermoplastic material like silicone (Oeffner & Lauder, 2011; Wen, Weaver & Lauder, 2014 and Domel et al., 2018). The reason for selecting polyurethane resin is because of its inherit elastomeric properties allowing formation of a flexible membrane along with allowing formation of tough denticles (Zhang, 2014). As expected, fabric-like flexible and softer films, exhibiting potential for functional clothing, were obtained from PU resin. Thus, swatches (with and without riblet effect) with variation in base thickness (1.05mm and 0.75mm) were developed using elastomeric polyurethane photopolymer resin in the photopolymer printer (Figure 4.5 (D)). The PU swatches were further used in material characterizations.

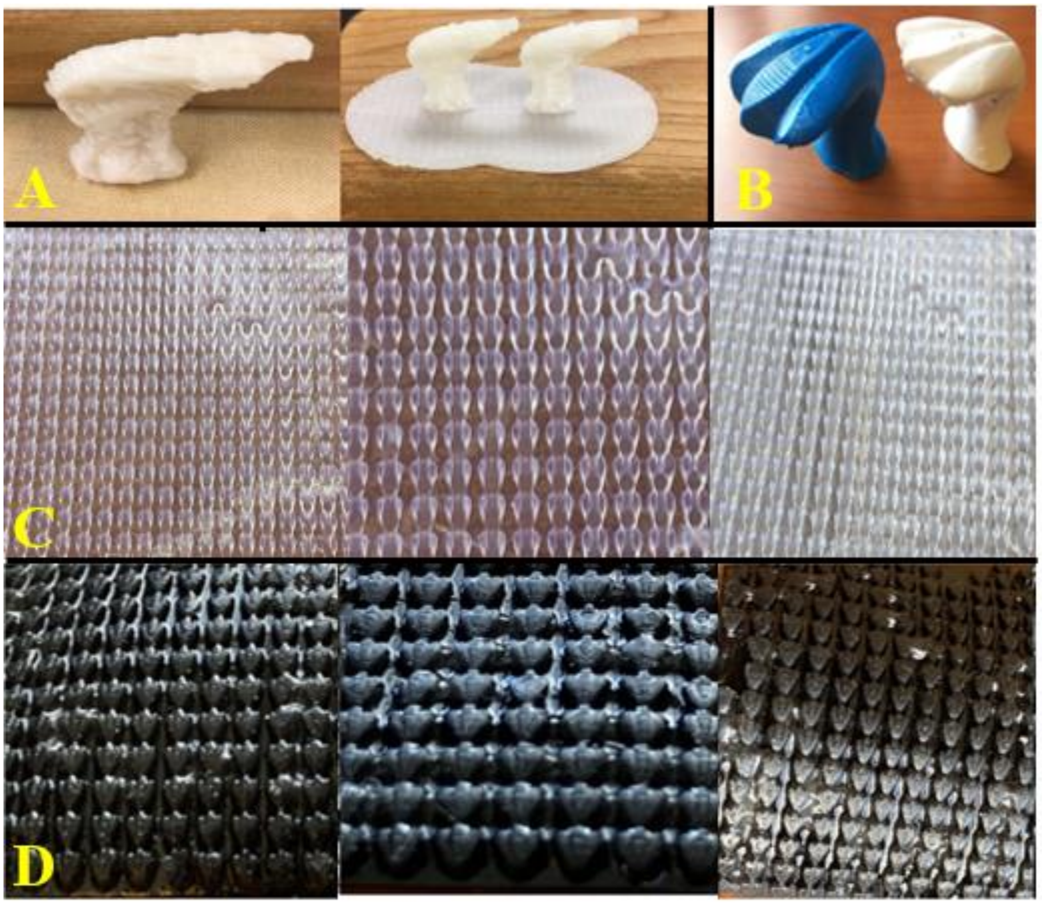

Figure 4.5. (A) Single denticle and denticles in linear array embedded in base, printed by FDM printers with PC/ABS resin (using the rough denticle 3D model), (B) Denticles with three surface ridges and three prongs printed by FDM printers with PC/ABS resin (using the detailed final denticle 3D model), (C) Riblet texture printed by Autodesk Ember Photopolymer Printer using PC/ABS resin and (D) Riblet texture printed by Autodesk Ember Photopolymer Printer using elastomeric polyurethane photopolymer resin.

4.3.3 Morphological analysis

This final product was morphologically tested using an optical microscope. The microscopic images captured by motif Image Plus 2.0 indicated detailed riblet structure consisting of "three surface ridges and three posteriorly pointing prongs" as observed in the images of shark skin captured in the studies of Oeffner and Lauder (2011) and Wen et al. (2014) (Oeffner & Lauder, 2011, p.789). The films consisted of each denticle of height 1.26 mm, length 2.1mm and width 1.52mm, spaced equidistant along stream-wise and lateral direction as proven to be hydrodynamically efficient by Domel et al. (2018). However, the riblet structure could be refined in terms of the three prongs. As per microscopic images of shark (Figure 2.1) the prongs are expected to be further distinct from each other by increasing the inner curves separating them since those dimensions were not specifically listed in the referenced work.

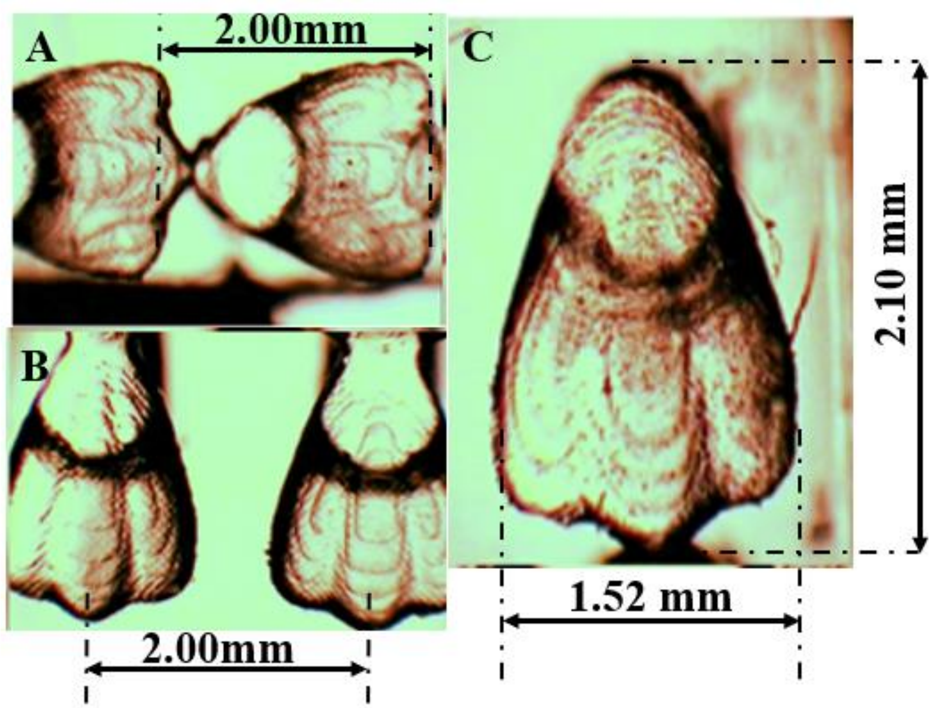

Figure 4.6. (A) Adjacent denticles along lateral direction, (B) adjacent denticles along stream-wise direction and (C) single denticle optical images of 3D printed bio mimicked shark skin films.

4.3.4 Mechanical properties

The 3D printed textured as well as untextured polyurethane films for thick base (1.05 mm) and thin base (0.75 mm) were tested each in triplicate for mechanical performance. Raw data in form of load-extension curves obtained from Bluehill 2.0 software were converted into stress-stain curves as shown in Figure 4.7.

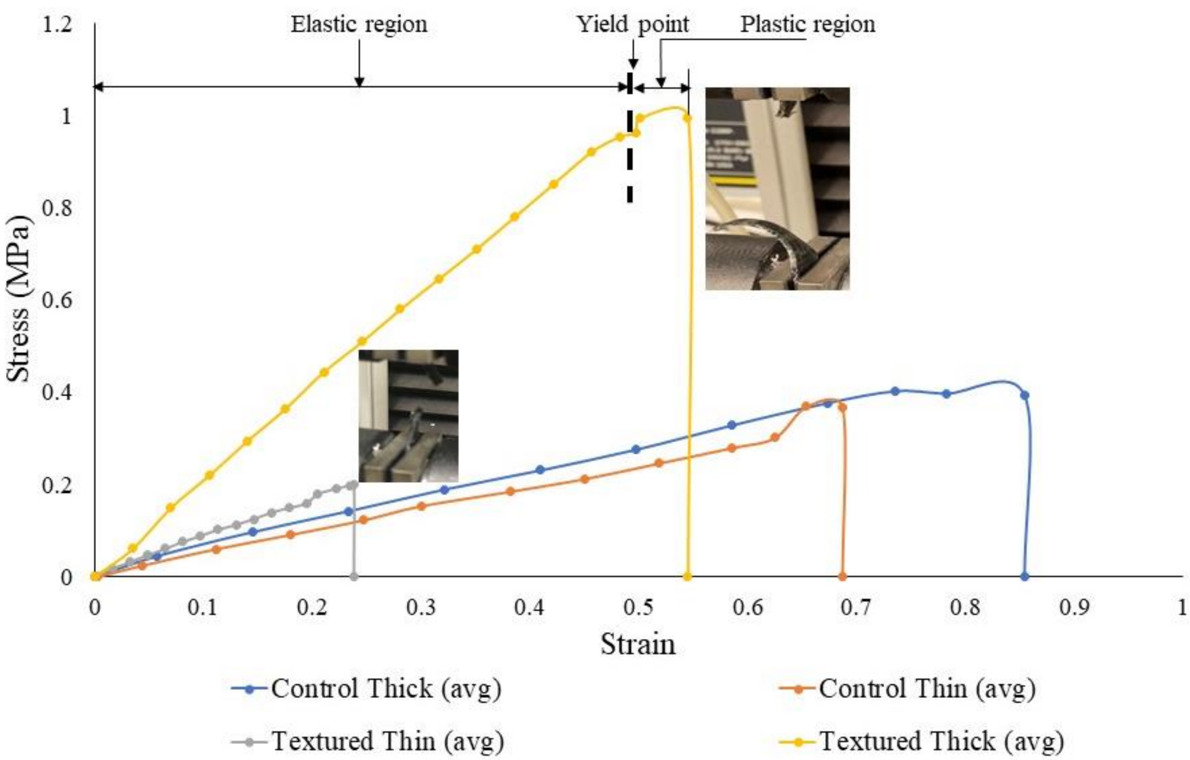

Figure 4.7. Averaged stress-strain curves for bio mimicked and untextured (control) samples for 1.05mm base and 0.75mm base along with fracture point images for the textured samples.

Table 4.1 Tensile strength, breaking elongation, and Young's modulus for the bio mimicked and untextured (control) samples for 1.05mm (thick) base and 0.75mm (thin) base.

Sample Type		Tensile Strength (MPa)	Breaking Strain (%)	Young's Modulus (MPa)
Textured Samples	Thin	0.201	23.86	0.842
	Thick	0.994	54.50	1.823
Untextured Samples	Thin	0.366	68.72	0.532
	Thick	0.392	85.45	0.458

As represented in the stress-strain curve obtained for the thick textured sample as shown in Figure 4.7, the curve observed fits into the typical curve path of a polymer fiber exhibiting an elastic area, yield point, plastic area as well as tensile strength and strain at break (Zhang, 2014). The deformation prior to the yield point was usually reversible in most of polymeric fibers (Zhang, 2014). After the yield point, the deformation was irreversible and a strain smoothening phenomenon occurred wherein stress decreases due to increase in strain (Zhang, 2014). After the strain smoothening phenomenon, the stress increased again till a point where the fiber breaks. Figure 4.7 also shows the fracture points for the texture samples, observed closer to the upper grip. The samples were tested along vertical direction of riblet effect. Fracture occurred in the area of samples clamped, which might be due to the photopolymer resins such as polyurethane used in the 3D printed films which are isotropic in nature (Fragassa and Minak 2008).

Further, the stress-strain curve of a polymer exhibits the tensile strength (stress at break), Youngs modulus/stiffness (slope of the curve) and toughness (area under the curve). Table 4.1 shows the tensile strength, breaking elongation, and Young's modulus obtained for the bio mimicked and untextured (control) samples for 1.05mm (thick) base and 0.75mm (thin) base. As per tensile strength values, thick samples have higher strength than thin samples. Further in terms of stiffness, base thickness variation did not impact the stiffness however the textured samples are stiffer than untextured samples due to higher Young Modulus values. Lastly for toughness, thick samples are tougher than thin samples due to larger area under curve. Overall, the textured thick sample was observed to have highest strength of 0.994 MPa, highest stiffness of 1.823 MPa, as well as highest toughness justified by the thick base (1.05mm) along with the protruding inbuilt riblet effect.

For functional clothing applications requiring elastomeric properties, fibers with high toughness and elasticity are utilized such as but not limited to; polyurethane (maximum elongation 35% to 204%), nylon-spandex blends (maximum elongation 27.48% to 40.49%), and spacer fabrics (maximum elongation 49.17% to 159.68%) (Lin et al., 2004; Zhu et al., 2006 and Yip & Ng, 2008). The developed textured swatches exhibit maximum elongation of 23.86% to 85.45%. Thus, stress-strain curves of the developed swatches being made up of polyurethane resin exhibit a typical elastomeric stress-strain curve path with high toughness as well as elongation (Zhang, 2014, Stress-strain behavior of polymers, n.d.). These elastomeric properties can have potential in functional clothing such as swim wear, compression wear, biking shorts, scuba wear and more, requiring flexible material that can endure high breaking energy. Further, the tensile strength of the developed swatches ranges from 0.201 MPa to 0.994 MPa. For elastomeric functional wear application such as active sports-wear, the mainly used material includes elastomeric fibers that is polyurethane (tensile strength of 0.36 MPa) or in combination with fibers such as Nylon (3.87 MPa to 5.4 MPa), Polyester (tensile strength of 2.25 MPa to 5.4 MPa), and Wool (0.9 MPa to 1.53 MPa) (Manshahia & Das, 2014; Zhang, 2014).

4.3.5 Antimicrobial properties

Figure. 4.8 shows photographs of one antimicrobial tests. The results indicated overlapped, bacterial colonies, likely to be than 300, thus the results were analyzed through quantification of the area of bacterial growth for each test sample in place of counting the bacterial colonies using ImageJ software (Viable cell counting, 2019).

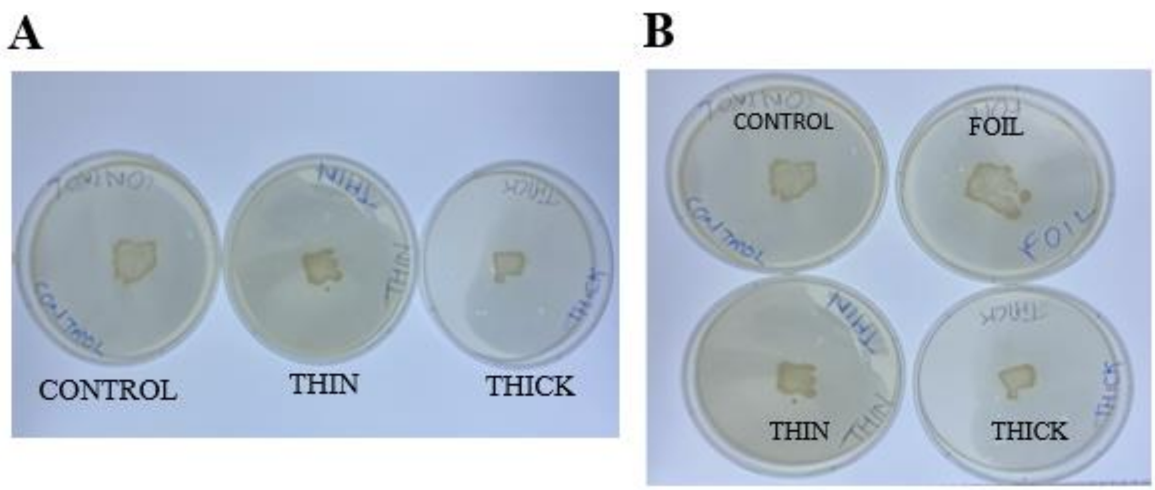

Figure 4.8. Antimicrobial test results in terms of post-incubation bacterial growth for (A) Textured sample with thick (1.05mm) base, textured sample with thin (0.75mm) base and control sample (B) Textured sample with thick (1.05mm) base, textured sample with thin (0.75mm) base, control sample and foil.

Quantification of five antimicrobial test results (n=5) was completed through individually measuring the surface area of the microbial growth for each test sample. Software employed was ImageJ to quantify the results. The results indicated reduced bacterial growth in the textured samples. Overall, as seen in Figure 4.9A, the bacterial growth was observed highest for the aluminum foil followed by the copper foil, then control sample and lowest for the textured samples. The inclusion of aluminum and copper foil for comparative analysis indicated that copper foil exhibits reduced bacterial growth as compared to aluminum foil corresponding to why copper is considered as an antimicrobial agent. Smaller the *p*-value, stronger is the evidence against null hypothesis which in this case is ‘no antimicrobial activity by test sample’ while strongly supporting alternative hypothesis which in this case is ‘demonstration of antimicrobial activity by the test sample’. As exhibited in Figure 4.9A, since the textured samples exhibited smaller comparative p-values in terms of aluminum foil, copper foil and control sample, thus said to greater support the alternative hypothesis than the rest. However, as seen in Figure 4.9B a significant difference in terms of antimicrobial property was not observed among the thick and thin bio-mimicked samples with a significantly large *p*-value of 0.841106 (>0.05). Thus, the thickness of the base does not play a vital role in antimicrobial property of the sample.

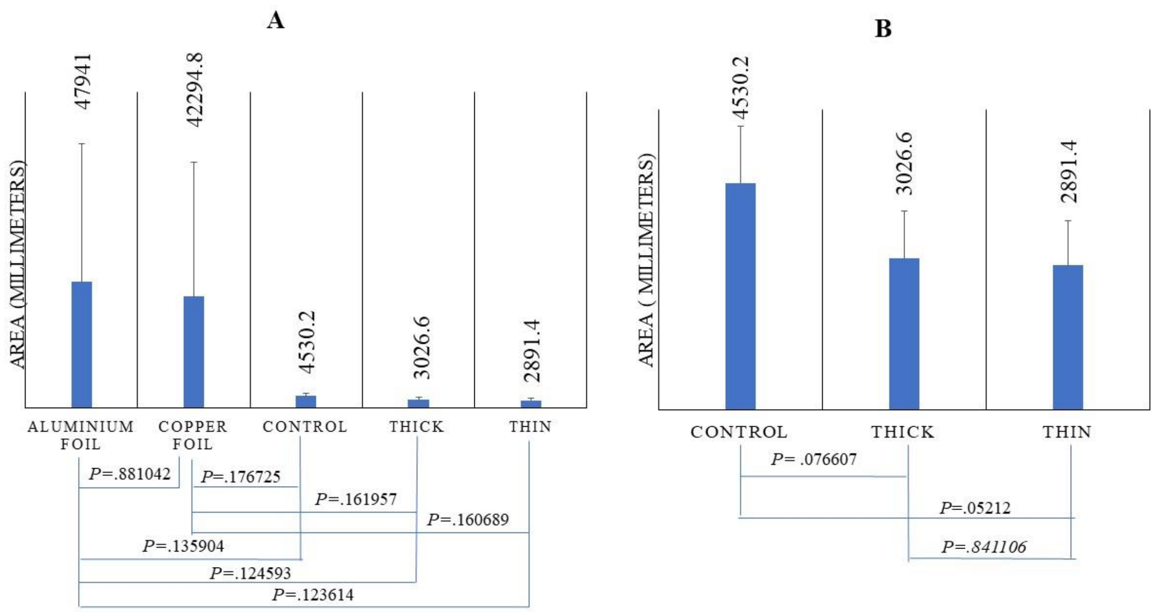

Figure 4.9. (A) Histogram representing average of the area of observed bacterial growth for each test sample (B) Histogram representing average of the area of observed bacterial growth for control (untextured) and textured samples.

As emphasized in Irene et al. (2016), copper coating for textiles is proven to be an effective antimicrobial agent for textiles used in health-care setting. Our results indicated that the developed textured swatches exhibited 92.8% (thick base) and 93.1% (thin base) bacterial reduction in comparison with copper foil. Further, due to growing need of non-toxic antimicrobial agents, several studies have focused on eco-friendly methods of generating antimicrobial textiles for applications such as medical textiles and textiles worn close to skin (Joshi, Ali, Purwar & Rajendran, 2009). As per the study of Thilagavathi & Kannaian (2010), the microencapsulation of geranium leaves extract into textiles finish exhibited reduced bacterial growth for *E.coli* as; microencapsulated geranium extract by coacervation spray drying method (55%) and microencapsulated geranium extract by spray drying method (60%). However, the reduction in bacterial growth dropped to 25%, and 40% respectively after 15 washes. Similarly, Thilagavathi, Bala & Kannain (2007) studied antimicrobial properties of textiles through microencapsulation of herbal extract for *E.coli*. The results indicated reduction in bacterial growth as; microencapsulated neem extract (55.21%) and microencapsulated Mexican daisy (53.85%), while the directly applied extracts got removed post wash resulting into no antimicrobial property. The developed textured swatches in this study exhibit 33.16% (thick base) and 36.17% (thin base) bacterial reduction in comparison with control swatches. The inbuilt riblet effect responsible for antimicrobial properties can ensure consistent antimicrobial behavior sustaining the wash cycles through the period of use, thus it can exhibit potential in functional clothing as non-toxic antimicrobial material.

4.4 Conclusion

Fabric swatches with riblet texture and variation in base thickness (1.05mm and 0.75mm) were successfully printed using polyurethane material via an Autodesk Ember photopolymer printer. The developed riblet texture demonstrated denticles each measured; height 1.26 mm, length 2.1mm and width 1.52mm reported to be efficient in drag reduction (Domel et al., 2018).

The selection of this particular 3D printer and polyurethane resin as the material allowed production of the flexible base membrane with minutely detailed rigid denticles embedded in it simultaneously. The study conducted material characterizations including morphological analysis, analysis of mechanical properties and antimicrobial property allowing determination of the developed swatches' potential for use in the area of functional clothing. The morphological analysis exhibited the resemblance in the developed texture and 3D models of shark skin. Analysis of mechanical properties was conducted through tensile stress testing, providing comparative analysis of the samples with variation in base thickness in terms of Youngs modulus, yield point, plastic deformation strain, failure stress, and elastic area of the samples. The comparative analysis in terms of base thickness indicated that thick samples were tougher while thin samples were brittle. The variation in thickness also indicated strong samples with thick base. Further, it was observed that the textured samples have higher stiffness than the untextured samples. The stress-strain curves following elastomeric path, possessing high toughness and flexibility, indicate potential of the textured swatches in functional clothing such as swimwear, scuba wear and biking shorts. Lastly, the antimicrobial tests indicated a reduction of bacterial growth for the textured samples against the control (untextured) samples and against aluminum as well as copper foil. Thus, the test results indicate potential of the textured swatches for use as non-toxic antimicrobial material.

Studies concerned with replication of riblet effect mainly focused on hydrodynamic properties while the shark skin inspired material intended for antimicrobial purpose as by Sharklet® technology is not concerned with riblet effect replication. Thus, to our best knowledge, study focusing on mechanical and antimicrobial analysis of shark skin biomimicry through replication of riblet effect for functional clothing application was missing. Thus, this study helped in determining potential of replicated riblet effect in functional clothing through the characterized morphological, mechanical and antimicrobial properties. As confirmed by the morphological analysis, the developed samples possess riblet effect with dimension proven hydrodynamically efficient as per Domel et al. (2018). The mechanical analysis indicated better strength and toughness for 1.05mm base thickness, while also indicating potential of the developed samples in functional clothing requiring elastomeric applications. Further, the antimicrobial analysis shows reduced bacterial growth for the riblet effect, showing potential as a non-toxic antimicrobial material.

CHAPTER 5. CONCLUSION AND FUTURE WORK

The research aimed at (1)3D printing bio-mimic shark skin fabric swatches, (2) characterization of the developed swatches in terms of morphological, mechanical and antimicrobial property analysis to determine the developed swatches' potential for use in the area of functional clothing. The development of fabric swatches with shark skin riblet effect involved 3D printing the swatches using elastomeric polyurethane resin and Autodesk Ember Photopolymer 3D printers. The selection of this particular 3D printer and polyurethane as the material allowed production of the flexible base membrane with small-sized detailed rigid denticles embedded in it simultaneously. The developed riblet effect's dimensions were based on proven hydrodynamically efficient sizing criteria with each denticle of height 1.26 mm, length 2.1mm and width 1.52mm, spaced equidistant along stream-wise and lateral direction (Domel et al., 2018). Further, to allow comparative analysis, the samples were developed with variation in base thickness (0.75mm and 1.05mm)

The developed swatches were analyzed in terms of morphological, mechanical and antimicrobial properties to determine the developed swatches' potential for use in the area of functional clothing. First, the morphological analysis indicated riblet shark skin texture on developed swatches. Second, the mechanical analysis conducted through tensile stress testing allowed comparison between the variation in base thickness in terms of strength as well as toughness of the samples. It was found that the variation in thickness resulted into higher strength and toughness for thicker samples than the thinner samples. It was also observed that the untextured samples have higher toughness in comparison with untextured samples. While the textured thick sample recorded the highest strength among all the samples.

Lastly, contact agar plate antimicrobial test conducted indicated reduced antimicrobial growth for the shark skin textured swatches in comparison with the untextured swatches, copper foil and aluminum foil. Since, limited information was available in terms of mechanical and antimicrobial analysis of shark skin biomimicry through replication of riblet effect. Thus, this study can help fill the gap of understanding the shark skin bio mimic fabrics in the use of functional clothing.

The attempt of biomimicry of shark skin in textiles is an on-going continual process. Future studies could be directed to better replicate the shark skin riblet effect in terms of structural details and properties. This study builds ground to attempt at developing a fabric with riblet effect, large enough to be converted to a functional cloth. This study also directs towards testing of riblet effect in terms of other properties such as but not limited to; fabric hand and breathability to better study its potential in the area of functional clothing. Lastly, because the focus of this study has been to explore the use of a bio-mimicked 3D printed fabric in functional clothing, a possible future scope would be construction and evaluation of a functional wear utilizing a 3D printed fabric with riblet effect.

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