THESIS

STUDY OF MECHANICAL AND ANTIMICROBIAL PROPERTIES OF BIOMIMETIC SHARK SKIN FABRICS WITH DIFFERENT DENTICLE SIZE VIA 3D PRINTING TECHNOLOGY

Submitted by

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ABSTRACT

STUDY OF MECHANICAL AND ANTIMICROBIAL PROPERTIES OF BIOMIMETIC SHARK SKIN FABRICS WITH DIFFERENT DENTICLE SIZE VIA 3D PRINTING TECHNOLOGY

Previous studies have shown that biomimetic shark skin fabrics can reduce water drag and increase swimming speed. It was also known that the smaller the denticle was, the higher water drag reduction was. In nature, the size of the denticles on shark skin is between 100 µm and 500 µm. However, the minimum size of the 3D printed denticles on a biomimetic shark skin fabric previously reported was about 2mm, which was still much larger than the natural size. In this study, different sizes of denticles ranging from 0.65mm to 1.30mm were fabricated using a Form3 3D printer and Flexible80A resin, and the effect of denticle size on mechanical properties and antimicrobial properties of biomimetic shark skin fabric were evaluated for the applications in functional clothing. The results suggested that when the size of the denticle was decreased, the stiffness of the fabrics was increased. In the tensile testing, the tensile strength and the breaking elongation of the 3D printed fabric with 1.04mm denticles were largest in the tested fabrics, which was larger than those of some common fabric materials used in commercial swimwear, suggesting great potential of functional clothing applications. In addition, mechanical anisotropy was observed in the 3D printed fabrics, which is commonly seen in textile fabrics. In antimicrobial testing, the shark skin fabrics with 0.65mm and 1.04mm denticles were found to be less susceptible to bacterial attachment, suggesting good potential for functional clothing applications.

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CHAPTER 1 INTRODUCTION

Biomimicry is a technology which creates innovative solutions for human challenges by emulating nature's time-tested strategies (Lurie-Luke, 2014). It has been recently recognized as an innovative strategy for developing functional clothing and textiles. Using biomimetics in a textile design system is the newest area in high-tech fabric with little research, but it has great significance (Kapsali, 2015). In one biomimetic development, tooth-like structures, called denticles, covering the shark skin surface are believed to play an important role on hydrodynamic effects that may benefit the design of functional clothing (Oeffner & Lauder, 2012). Studies have found that the denticles on shark skin can especially reduce fluid drag and increase thrust (Domel et al., 2018). It has been reported that shark skin biomimetic materials have played an essential role in the aerospace industry, medical field, and functional textile industry (Montazer & Harifi, 2018; Pu et al., 2016). In the competitive swimming sector, drag reduction properties are especially valued for the design of swimwear. The integration of shark skin characteristics into fabrics that are used in full-body suits can help athletes reduce water drag and speed up swimming (Kapsali, 2015). In order to realize the application value of drag reduction properties, man-made materials are produced to imitate the structure of shark skin and to develop biomimetic shark skin fabric (Oeffner & Lauder, 2012). Thousands of denticles on shark skin are arranged in a regular distribution that requires fabrication techniques capable of fine work in imitating the denticles and their arrangement. One of the promising fabrication techniques is digital 3D printing that can print the prototype of a 3D model generated by computer aided design (CAD) programs (Gross et al., 2014). 3D printing technology can help fabricate unique shapes such as shark skin denticles, suggesting an effective method for making biomimetic shark skin fabrics.

The performance of shark skin biomimetic fabrics is associated with important factors such as denticle size, space between adjacent denticles, denticle arrangement, and base thickness. Wen et al. (2015) 3D printed shark skin fabrics with a denticle size of about 1.87mm and studied different arrangement of denticles to examine the influence of denticle arrangement patterns on its hydrodynamic performance. The result showed that the staggered-overlapped pattern had superior water drag reduction. In addition, Domel et al. (2018) investigated the effects of denticle size on hydrodynamic properties of shark skin fabrics. The results suggested that the smallest denticles of 2 mm promoted the most drag reduction. In fact, the size of the denticle found in shark skin in nature is between 100μm and 500μm (Domel et al., 2018). The denticle size that has been studied to date for biomimetic fabrics was approximately ten-time larger than that in nature. In addition, the use in functional clothing requires flexibility and good hand in textile fabrics, suggesting that the biomimetic shark skin fabrics decorated with small denticles would be preferable due to good hand and flexibility. With the increasing demand for functional clothing, shark skin fabric has great potential for development in future market. Therefore, exploring the performance of biomimetic shark skin fabrics with denticles close to the size found in nature can help maximize the benefits of biomimicry for functional clothing applications.

In this study, biomimetic shark skin fabrics will be created using a commercial resin (Flexible80A, Formlabs, Somerville, MA) that contains 75%-95% methacrylate monomer, 3%-6% urethane dimethacrylate, and <1.5% photoinitiator and Form3 SLA 3D printer (Somerville, MA). The fabric will be made by a thin film with denticles simultaneously printed on the film via 3D printing. The denticles of interest will be designed in sizes smaller than 2mm as well as infinitely close to the natural size in CAD models. The thickness of the thin films is set at 0.3mm which is found in the range of fabric thickness of commercial swimsuits. Therefore, the thin films likely simulate fabric swatches for the purpose of functional textiles. The printed fabric swatches will be characterized for three textile performance properties, including stiffness performance, mechanical properties, and antimicrobial properties. The primary objective is to provide implication of the effects of small denticle size on the fabric performance characteristics of stiffness, mechanical, and antimicrobial properties.

Hypothesis of this study: When the size of the denticles printed on the thin films decreases, the stiffness and antimicrobial performance of the films would be increased, but the tensile strength of the films would be reduced.

The hypothesis will be tested in the objectives as follows:

1. To develop 3D models of individual denticle and biomimetic shark skin fabrics with different denticle sizes.

2. To print biomimetic shark skin swatches using a 3D printer.

3. To measure and evaluate stiffness, tensile strength, Young's modulus, elongation at break, and antimicrobial properties of biomimetic shark skin fabrics with different denticle sizes.

CHAPTER 2 REVIEW OF LITERATURE

2.1 Biomimicry

Sharks swim fast in the ocean, which has inspired researchers to pay attention to shark skin and discovered excellent hydrodynamic properties due to the unique surface structure of the shark skin (Wen et al., 2014). Shark skin surface is not that smooth at all at macroscopic view, but shows so called riblet structures (Fu et al., 2017). The surface of shark skin is regularly covered with scales that are called dermal denticles. The denticles contain riblet structures with specific size and spacing between adjacent denticles, parallel to the swimming direction (Bixler & Bhushan, 2013). This structure can prevent vortexes from forming as well as staying on the shark skin surface and hence reduce the dragging force when sharks move in the water (Fu et al., 2017). A single dermal denticle generally has a three-prong structure as shown in Figure 1 that gives a close look of the replica shark skin of Spiny Dogfish.

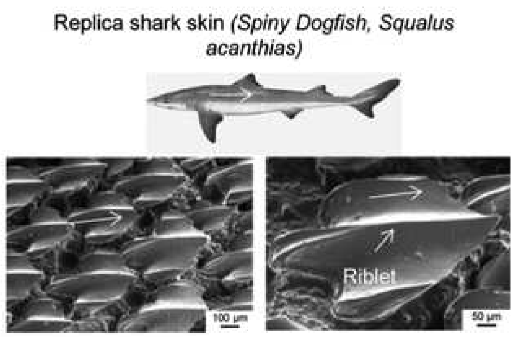

Figure 1. Dermal denticle of replica shark skin. The left image shows the denticle structure of the replica Spiny Dogfish under the microscopic lens (Bixler & Bhushan, 2013). The arrows on the right image represent the direction of liquid flow.

2.2 Shark Skin

Biomimicry is the process of creating new things by imitating nature and improving human life (Rawlings et al., 2012). The goals of biomimicry are to make alternative products and to deepen the understanding of biology. Deepening the understanding of biological and natural working principles through biomimicry can help humans use natural mechanisms and create products with natural characteristics (Rawlings et al., 2012). Scholars have studied and used biomimicry to develop new products and technologies for a long time, and there are many successful examples in different fields (Hwang et al., 2015). Human development and progress have benefited a lot from the mystery and diversity of nature and will continue to explore and develop in the field of biomimicry in the future.

For example, the Japanese bullet train is inspired by the kingfisher's beak (Hwang et al., 2015). The original train leaving the tunnel was accompanied by a loud roar, engineers simulated the front of the train based on the long and narrow beak of the kingfisher to solve this problem. This model reduced noise and increases speed, saving them 10-15% of energy (EarthSky, 2012). Another example is morphotex fabric that was developed from the Morpho butterfly (Frumkin & Weiss, 2012). The iridescence on the wings of this butterfly comes from its micro-laminated structure (Rawlings et al., 2012). The Morphotex fiber uses nylon and polyester to mimic this structure to achieve the same vibrant blue color on fabrics without textile dyeing.

2.3 Biomimetic Shark Skin Fabrics

The superior hydrodynamic effects by shark skin riblet have inspired the application of shark skin's unique structures in textiles, especially in functional clothing such as swimwear. In order to apply water drag reduction properties to functional clothing, researchers have attempted to develop shark skin fabric and imitated the dermal denticles as well as its riblet structures on fabric surface. Using biomimetic shark skin fabrics in swimsuits can reduce drag forces in water and hence increase swimming speed (Kapsali, 2015). Recently, the demand for the production of this smart textile has increased as textile innovation and sustainability have played greater roles in market. The application of biomimicry to swimsuits has become crucial. It is a high-tech fabric application that has a significant effect on the improvement of functionality. Biomimicry follows the path of least energy consumption, combines with the principle of promoting sustainability, and solves problems from the perspective of the environment and humans (Suresh Kumar et al., 2020). It establishes an economy that follows natural development and evolution, creates stable productivity, and reduces the negative impact on the environment (Hwang et al., 2015). However, there are still many challenges that make mass production of shark skin fabrics difficult for functional clothing applications. The challenges include fabric flexibility, comfort, fabric hand, cost, and efficiency requirement of shark skin fabric fabrication (Thompson, 2014).

2.4 Current Technologies to Develop Shark Skin Fabrics

After many years of research on imitating shark skin on fabrics, a few commercial textile products that are inspired by shark skin have gradually emerged and begun to grow in textile industry. For instance, Speedo is a functional clothing company who dedicates to the production and innovation of swimwear that helps swimmers move fast in the water (Swimswam, 2019). Inspired by the texture of the shark skin, Speedo developed and commercialized a product line of Fastskin® suit. The Fastskin® suit uses knitted super-stretch polyester fabric with V-shaped ridges and denticle surface print as shown in Figure 2 (Science in The News, n.d.). The product mimics the microstructure of shark skin surface by superimposing resin material on the surface of the fabric to achieve the drag reduction (Singh et al. 2012). However, the V-shaped ridges in suits like Speedo's Fastskin® II didn't truly mimic the denticles of shark skin. The lack of true biomimicry of shark skin denticles in Fastskin® swimsuits is unable to promote drag reduction effectively (Oeffner & Lauder, 2012), suggesting that there is still great potential of applying true shark skin riblet effects to functional clothing, especially to swimwear. Innovation in fabrication technologies is of great interest in developing true shark skin fabrics for functional clothing.

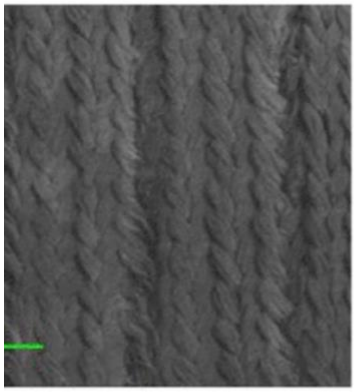

Figure 2. Speedo fabric under the microscope level. It only has some grooves instead of riblets, which cannot increase swimming speed effectively (Oeffner & Lauder, 2012).

2.5 3D Printing Technology

3D printing technology is a flexible way of making parts and can develop parts that cannot be produced by traditional manufacturing technology (Lee et al., 2016). 3D model of the unique part is developed by using computer-aided design (CAD) programs, and then 3D printer is utilized to fabricate the part. The 3D printing technology is highly efficient, especially for unique, special, and sophisticated geometry and shapes.

Many different 3D printing methods have been developed so far. Melt extrusion and photopolymerization (PP) are two categories of methods that have been used widely. In the melt extrusion method, a heated nozzle is used to extrude a semi-melted thermoplastic material onto a computer-controlled stage and hence create a 3D printed model (Chatterjee & Ghosh, 2020). The printed parts are usually post-processing, such as sanding, to reduce surface roughness (Yadav yet al., 2020). In the photopolymerization method, photopolymerization is utilized to develop 3D objects simultaneously during printing. For example, digital light processing (DLP) is a popular PP methods. In DLP, a small amount of resin is poured into a build chamber. The building platform is immersed in the resin from above, and the ultraviolet (UV) light is projected from below the build chamber to cure the resin (Bagheri & Jin, 2019).

Parts made by PP methods tend to have a smooth surface finish compared with those made by melt extrusion. Therefore, no subsequent sanding and polishing process is required in PP methods. In addition, PP methods provide high resolution, which makes them suitable for producing intricate details. The layer thickness of a part can be printed as low as 25 microns (Bowmanap, n.d.). Another benefit of the PP methods is that the photopolymer resins can be rigid or flexible, brittle or highly stretchable. Therefore, different resins can be chosen according to the requirements of the printed parts. When a 3D printing method is considered for creating shark skin fabrics for textiles such as swimsuits, flexibility and strong tensile properties are necessary. Resins with this characteristic would be desirable for printing shark skin fabric. However, the photopolymer resins used in PP methods are more expensive than the raw materials used in melt extrusion methods (Innotech, 2016). In addition, parts produced by photopolymerization become brittle over time and are not friendly to prolonged use (Bowmanap, n.d.).

Typical photopolymer materials used for PP 3D printing are acrylic resins. With acrylates, an anionic photoinitiator produces free radicals when exposed to UV light. These free radicals react with monomers to induce polymerization, which proceeds to create parts (PolymerDatabase, n.d.).

2.6 Biomimetic Shark Skin Fabric via 3D Printing

In the work that has been reported to develop 3D printed biomimetic shark skin fabric, the shark skin denticles were usually printed using rigid materials (Wen et al., 2014), while the base layer was printed using a flexible rubber-like material (Domel et al., 2018; Wen et al., 2014). This approach ensured that the denticles on the sample surface were not deformed, while the entire sample was flexible.

It was reported that Objet Connex500 3D printer has been used to create biomimetic shark skin fabrics with denticle size approximately of 2mm (Wen et al., 2015; Domel et al., 2018). An Autodesk Ember photopolymer printer was also used to make shark skin fabrics that had denticle size of 2.1mm (Purandare, 2020). Denticle size smaller than 2 mm has not been reported in literature. Therefore, there is a critical need to investigate the feasibility of printing small size denticles on a base layer that can be used as shark skin fabrics for innovative textile applications.

2.7 Characteristics of Biomimetic Shark Skin Fabric

2.7.1 Drag force reduction

The microstructure of the denticles on shark skin is able to change the water vortex structure and the flow of the water surrounding it, thereby reducing drag forces. The reduction of drag forces is critically associated with two characteristics of the denticle microstructure. The two characteristics are the denticle arrangement and the denticle size.

2.7.1.1 Effect of denticle arrangement and spacing

Wen et al. (2015) developed three 3D printed shark skin fabrics with different denticle arrangement and spacing and studied the impact of the denticles arrangements, or patterns, on drag reduction. Figure 3 shows three arrangements of denticles, in (B) staggered-overlapped pattern, (C) linearly-overlapped pattern, and (D) linearly-non-overlapped pattern. A set of static and dynamic tests were designed and carried out using a robotic flapping foil apparatus that evaluates hydrodynamic performance of biomimetic shark skin fabric models (Oeffner and Lauder, 2012). The results suggested that the different patterns showed different performance of resistance to water flow. The static force resistance results showed a 3.2% reduction by the staggered-overlapped pattern and a 0.9% reduction by the linear-non-overlapped pattern, but a

9% increase by the linear-overlapped model, respectively (Wen et al., 2015). In the dynamic test, the resistance is evaluated by self-propelled swimming (SPS) speed that is measured by time-averaged zero force self-propelling device developed by Wen and Lauder in 2013. Fast swimming speed is promoted by low drag that corresponds high drag reduction. On the other hand, slow swimming speed indicates that the surface of the model receives large fluid drag, and the effect on the drag reduction is not significant. The SPS results showed that the staggered-overlapped shark skin had the highest SPS, and the SPS speed of the other two patterns was even lower than the smooth surface mode (control surface) Therefore, the staggered-overlapped arrangement demonstrated the most efficient drag reduction performance.

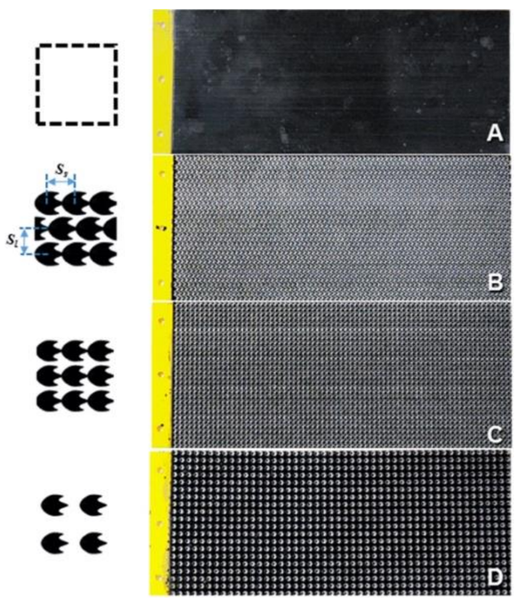

Figure 3. Control surface (A), staggered-overlapped pattern (B), linearly-overlapped pattern (C), and linearly-non-over-lapped pattern (D) arrayed shark skin fabric (Wen et al., 2015).

2.7.1.2 Effect of denticle size

The size of denticles on shark skin fabric is a decisive factor affecting swimming speed. Domel et al. (2018) studied the effect of denticle size on swimming performance. Three models with denticles in different sizes (2.10 mm, 3.15 mm, 4.20 mm, respectively) were arranged in a staggered-overlapped manner. The same robotic flapping foil apparatus as that used in the previous studies (Wen et al., 2015; 2018) was also used in static and dynamic tests. The results showed that the smallest denticle size shark skin (2.1mm) showed the most significant drag reduction effect than the smooth control model. The other two models with larger denticles than the first one showed greater water resistance compared to the smooth control model, resulting in low drag reduction. Therefore, the conclusion was that the larger the denticle, the less conducive to the drag reduction effect (Domel et al., 2018).

2.7.2 Mechanical properties

Mechanical properties are extremely important to functional clothing and have a significant impact on functionality of clothing. The mechanical properties that are commonly evaluated for textiles and fabrics are tensile strength, abrasion resistance, and stiffness (Munikenche Gowda et al., 1999).

Purandare (2020) studied the impact of different thicknesses of 3D printed shark skin fabrics on mechanical properties of the fabrics. Fabric swatches covered by 2mm denticle with 1.05mm base and 0.75mm base were created via an Autodesk Ember photopolymer 3D printer using elastomeric polyurethane photopolymer resin. The results suggested that thick shark skin fabric (1.05 mm thickness) had higher tensile strength of 0.994 Mpa than the thin fabric (0.75 mm thickness) when the denticle size was the same. In a comparison, the tensile strength of nylon fabric that is commonly used in swimwear is found to be as high as 5.4Mpa (Lin et al., 2004). Therefore, it is still challenging to develop 3D printed sharkskin fabrics with desirable strength (Sabantina et al., 2015).

2.7.3 Antimicrobial property

The riblet structure on the surface of shark skin does not have the effect of killing bacteria, but the microstructure has the characteristics of preventing external objects from being attached, resulting in prevention of bacterial contamination and breeding (Rettner, 2014). The principle of antimicrobial property on riblet structure is that it has a thin air layer hidden in the surface of shark skin due to the microstructure. The air layer can act as a physical barrier to prevent the adhesion of bacteria (Pu et al., 2016). Antimicrobial properties are of great value in textile and apparel because antimicrobial textiles cut off the transmission of pathogens and effectively prevents pathogens from contacting humans (Montazer & Harifi, 2018), resulting in promotion of safety and health. The swimming pool is a space where bacteria spread quickly. Although there is chlorine to kill bacteria, it still takes a while to take effect (Wallentine, 2017). Rostami et al. (2021) prepared a mixed solution using chitosan solution, lactic acid, ampicillin sodium salt, and caffeic acid phenethyl ester and used a solution casting method create a biomimetic shark skin surface, which was found to reduce the growth of bacterial biofilm by 11.1% to 19% compared with the control group. Purandare (2020) reported that 3D printed sharkskin fabrics made by polyurethane photopolymer resin demonstrated more than 14% bacterial reduction compared with copper foil that is a common antimicrobial agent used in health-care industry. The previous research suggested potential of significant antimicrobial properties that may be beneficial to functional clothing by 3D printed biomimetic shark skin fabrics. The antimicrobial effect of this physical barrier is non-toxic and harmless to the environment, and it plays a positive role in the friendly development of the environment.

CHAPTER 3 MATERIALS AND EXPERIMENTS

3.1 Materials

Flexible 80A resin (75%-95% methacrylate monomer, 3%-6% urethane dimethacrylate, and <1.5% photoinitiator, Somerville, MA), the resin for Formlabs SLA Printers, was purchased from Formlabs.

For the antimicrobial testing, *E. coli* ATCC25922 was purchased from ATCC (Microbiologics, St. Cloud, MN). Tryptone (Fisher, Waltham, MA), sodium chloride (NaCl) (Supelco, Bellefonte, PA), yeast extract (Fisher, Waltham, MA), and deionized (DI) water were the ingredient chemicals for preparing lysogeny broth (LB) culture media solutions. LB agar powder (Becton, Dickinson and Company, Sparks, MD), potassium chloride (KCl) (Fisher, Waltham, MA), sodium phosphate dibasic (Na_2HPO_4) (Sigma-Aldrich, St. Louis, MO), potassium phosphate monobasic (KH_2PO_4) (Sigma-Aldrich, St. Louis, MO), and 95% ethanol (Fisher, Waltham, MA) were also used in the tests. All the chemicals and solvents were used without further purification.

3.2 3D Models Development and 3D Printing

In the study of Wen et al. (2014), the Environmental Scanning Electron Microscope (ESEM) was used to photograph the mid-trunk position morphology and dimensions of freshly dead mako shark (Isurus oxyrinchus) denticles. In this study, Wen's model was adopted to create 3D models of sharkskin fabrics with denticles. First, Autodesk Fusion360 (Autodesk, Inc.) was used to develop a 3D model of a single denticle with precise parameters. Second, the single denticle model was arranged into the staggered-overlapped pattern (Wen et al., 2015) on a 50 mm × 30 mm × 0.3mm (length × width × height) film that creates the base layer of shark skin fabric. Biomimetic shark skin fabric swatches with the same staggered-overlapped pattern, but different denticle sizes were developed via Autodesk Fusion360. Figure 4 and Table 1 show the dimensions of large, medium, and small denticles and spacing dimensions between denticles. The spacing between two adjacent denticles was shortened as the denticle size was decreased. The range of denticle sizes and the density of denticles per square millimeter on real sharks were measured in Kanagusuku et al.'s (2021) study. Based on denticle size and density reported in the article, the denticle sizes the spacing in this study was proportionally determined as shown in Table 1.

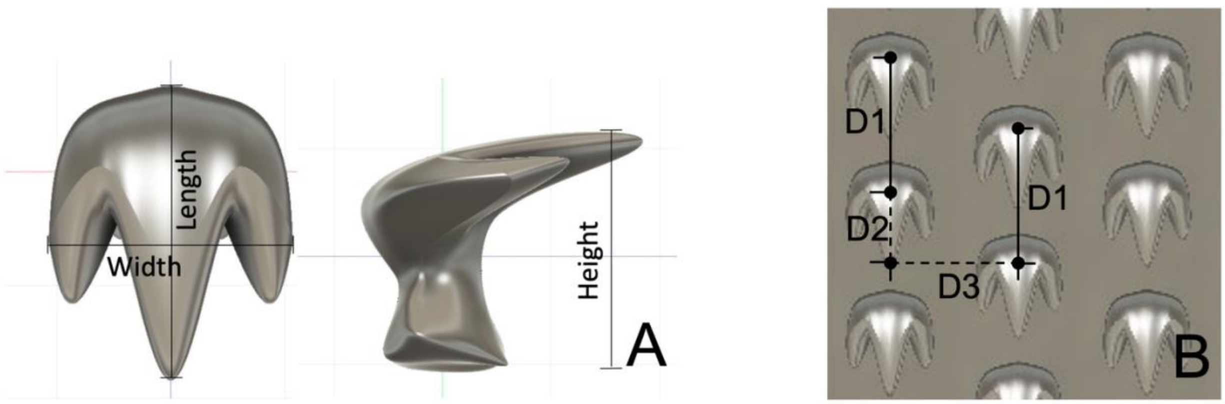

Figure 4. Three-dimensional image of a single denticle (A) and spacing between denticles (B).

Table 1. Denticle dimension and spacing between denticles on the 3D printed biomimetic shark skin fabric model.

	Large	Medium	Small
Length (mm)	1.30	1.04	0.65
Width (mm)	1.06	0.85	0.53
Height (mm)	1.02	0.82	0.51
D1 (mm)	1.50	1.25	0.80
D2 (mm)	0.75	0.625	0.40
D3 (mm)	1.50	1.25	0.80

In the printing, a Form3 3D printer (Formlabs, Somerville, MA) was used to print shark skin fabric swatches. A photopolymerization method is utilized in the Form3 printer. The surface of shark skin fabric is covered with complex microstructure, so the photopolymerization method is more suitable for establishing it. The Form3 printer has a resolution of 25 microns on the Xaxis and Y-axis, which can ensure that the details of the shark denticle are printed to the greatest extent. The film and denticles that made up the biomimetic shark skin membrane were simultaneously printed with Flexible 80A resin using the Form3 printer. Flexible 80A is an acrylate-based resin, which is commonly used and already is compatible with most 3D printers using a photopolymerization mechanism on the market. Thiol–ene-based photocurable resin is another commonly used material for PP printing. However, both resins have short shelf life and unpleasant smell (Bagheri & Jin, 2019), they were not used in current tests. After printing was completed and supports were removed, the part was washed with tripropylene glycol monomethyl ether (TPM) for 10 minutes to remove waxiness, and then cured for 10 minutes at 60°C in the Form Cure (Formlabs, Somerville, MA).

3.3 Fabric Performance Characterizations

3.3.1 Morphological analysis

Investigation of biomimetic shark skin fabric swatch morphology was carried out using an optical microscope (Motic Digital Microscope DMB3-223). The motif image plus 2.0 software (Motic Instruments Inc, Canada) was used to capture images for morphological analysis. Denticle size and spacing were measured using Image J software.

3.3.2 Stiffness testing

According to AATCC TM66 (Test Method for Wrinkle Recovery of Woven Fabrics: Recovery Angle) and ASTM standard D1388-08 (Standard Test Method for Stiffness of Fabrics), a customized method was developed to test stiffness of the biomimetic shark skin fabric swatches using wrinkle recovery tester (Momsanto Chemicals, Leverkusen). In sample preparation, fabrics were cut into a rectangle shape (15 mm × 40 mm). The three-prong of the denticle was parallel to the long side of the sample. Most of fabric samples were not flat so a 4 pound weight was used to flatten fabric samples for 24 hours. Figure 5 is a photograph that shows a sample being tested. In the stiffness testing, first, a sample was aligned to square in a 15 mm wide clamp to ensure it was aligned with the edge of the clamp. Second, as shown in Figure 5, the short straight line of the circular dial was oriented to the 90° indicator on the dial to ensure the clamp was completely perpendicular to gravity's force. When the sample on the clamp fell due to gravity, a mark was made on the circular dial where the sample hanged before the clamp was removed. And then, a straight line was drawn to connect the mark with the center point of the dial and extended to the edge of the dial. The dial was rotated 180° to enable the short straight line to eclipse the original straight line below the tester; the position of the clamp remained horizontal. The angle indicated by the numbers on the dial was recorded to measure the stiffness of the fabrics. The smaller the angle of bending, the stiffer the sample was. Each result was the average mean of five specimens (N=5).

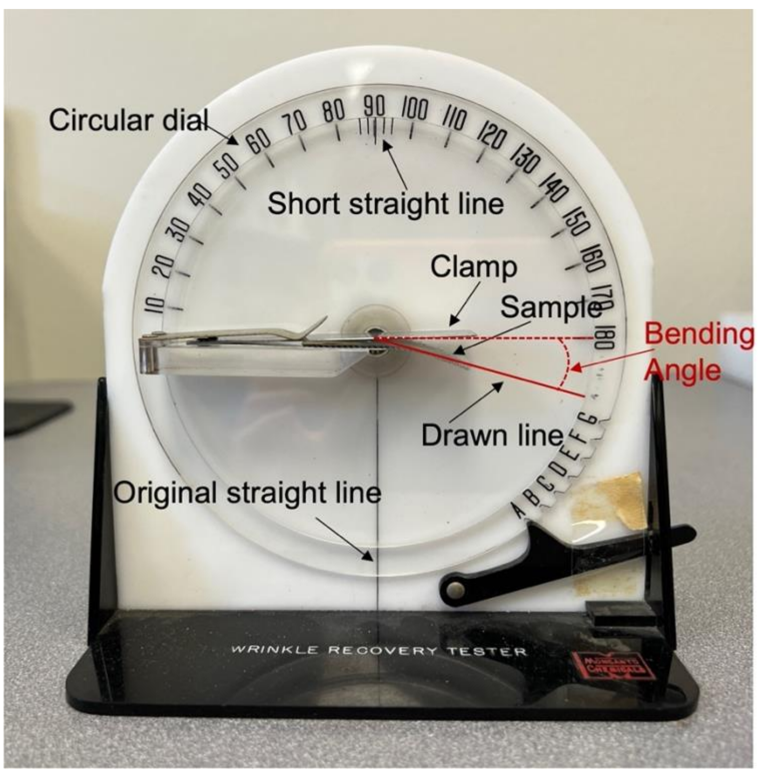

Figure 5. Wrinkle recovery tester for stiffness testing.

3.3.3 Mechanical properties analysis

In this study, the tensile strength, elongation at break, and Young's modulus of biomimetic shark skin fabrics were tested using Instron 4442 Mechanical Tester (Norwood, MA). The testing was conducted by following ASTM standard D412-16 (2021) (Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension). The samples were cut into dumbbell shapes and placed on the grips. The grip separation speed was 500±50 mm/min. The stress-strain curve and load-extension curve were recorded by Bluehill 2.0 software. As shown in the Figure 6, the fabrics were tested in both lengthwise (3 prongs of the denticle parallel to vertical direction) and widthwise (3 prongs of the denticle parallel to horizontal direction) directions. In Figure 6A, the denticles are all aligned in a straight line in the lengthwise direction, while the denticles are arranged in a zigzag pattern in the widthwise direction. Each result was the average of five specimens (N=5). All 30 tests (3 denticle sizes × 2 directions × 5 repetitions) were conducted at room temperature.

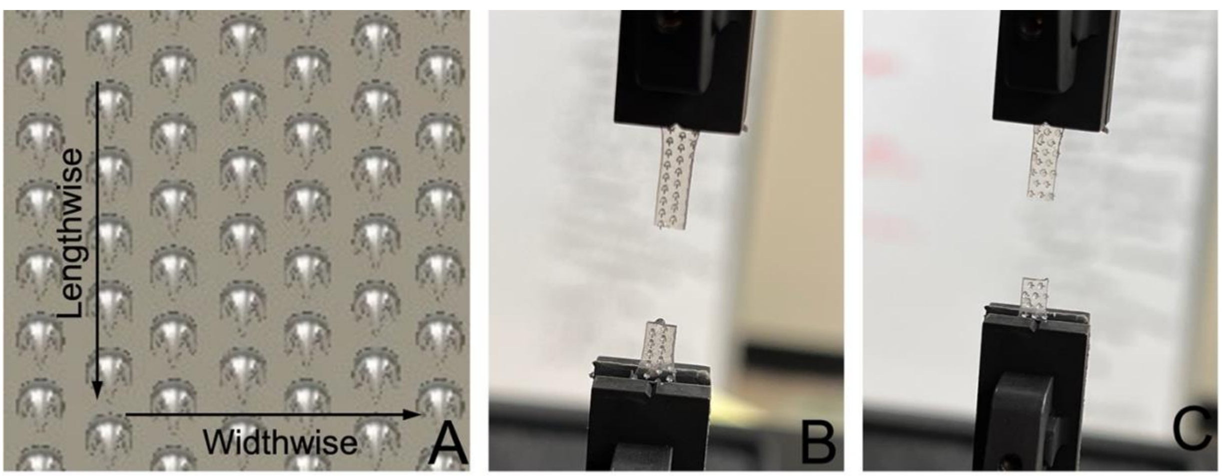

Figure 6. Directions of lengthwise and widthwise (A). Biomimetic shark skin fabric samples were tested in lengthwise (B) and widthwise (C) directions in mechanical testing.

3.3.4 Antimicrobial properties analysis

The antimicrobial properties of the biomimetic shark skin fabrics with different denticle sizes were evaluated using an immersion inoculation assay method described by Mann et al. (2014) and Purandare (2020). The prevention of bacterial growth was measured by the area occupied by bacteria after incubation. Statistical analysis was performed using ANOVA tests. 3D printed fabrics with smooth surfaces (no denticles) were used as control samples in the tests. All 3D printed fabric samples were cut into 1 cm × 1 cm squares. Five replicates (N=5) for each sample were used for statistical analysis.

First, a LB culture media solution was used to inoculate and to dilute *E. coli* ATCC25922. The LB solution was made of 950ml of DI water, 9.5g of tryptone, 4.75g of NaCl, and 4.75g of yeast extract. The solution was autoclaved at 273°F and 28 psi for 1 h. Two premade *E. coli* ATCC25922 pellets (1.0 × 104 cfu/pellet) were put in a hydration liquid and mixed by shaking, and then hydrated for 30 min at 37°C. The hydrated *E. coli* was inoculated in a 250ml LB media solution. The mixture was incubated at 37°C for 24 h in an incubator (Fisher, Waltham, MA). The incubated *E. coli* bacteria were diluted at 1:100 with a 700 ml LB media solution. The diluted solution was sub-cultured in the incubator at 37°C for 4 h.

A soft agar solution was made by mixing 700ml of DI water, 7g of tryptone, 3.5g of NaCl, 3.5g of yeast extract, and 6.3g of agar powder. The solution was autoclaved at 273°F and 28 psi for 1 h before the solution was added into 20 sterile petri dishes, covered and kept at room temperature for 2 h. The soft agar plates were stored in the refrigerator at 4°C for the following experiments.

1L of 1 × phosphate buffered saline (PBS) was made using 800ml of DI water, 8g of NaCl, 0.2g of KCl, 1.44g of Na_2HPO_4, and 0.245g of KH_2PO_4 mixed well. The solution was adjusted to pH value 7.4. More DI water was added to increase the volume to be 1L.

Before *E. coli* was introduced to shark skin fabric samples, each sample was firmly adhered to the bottom of a petri dish with the side with denticles facing up. The sample fixed in the petri dish was sterilized with 95% ethanol for 10 m and then was rinsed 3 times with DI water before it was allowed to air dry for 1 h. According to the immersion inoculation assay method (Mann et al., 2014), first, a sub-cultured *E. coli* solution was poured into petri dishes and samples were submerged and stored at room temperate for 1 h. Second, the *E. coli* that were not attached to the samples had to be removed by rinsing with 1 × PBS for 10 s while rotating for 3 times. Third, the petri dishes with samples were allowed to air dry at room temperature for 1 h. Each sample was taken off from the petri dish and pressed onto a soft agar plate for 5 s to minimize air bubbles between the sample surface and the agar. The soft agar plates then were incubated at 37°C in the incubator for 24 h. After the incubation, each soft agar plate was photographed. The area covered by the bacteria were measured using Image J software (Purandare, 2020).

CHAPTER 4 RESULTS AND ANALYSIS

4.1 Morphological Analysis

The 3D model of the denticle is a 3-prong structure that consists of a centrally-located long prong paralleled by two short prongs, one on either side, extending from the same main body creating a trident shape. Figure 7 shows optical images of shark skin fabrics with different size denticles. Table 2 shows denticle size and spacing of the fabrics as printed. It was found that the large denticles of 3D printed shark skin fabric (Figure 7A) had same morphology as the 3D model that contains the completed 3-prong structure. However, the medium denticles as shown in Figure 7B were not printed as precisely as large ones according to the 3D models. Although the 3-prong structure was still presented, the two laterally-located short prongs were not fully printed, resulting in shorter prongs than what were shown in the 3D model. The denticle was proportional imbalance, which resulted in a longer centrally-located prong that is highlighted. It was most likely due to the printer resolution limitations. When the denticle size continued to decrease, the 3D printed shark skin fabrics started to lose the unique 3-prong structures. Figure 7C shows the morphology of small denticles, which is quite different from the 3D model. The resolution of Form3 3D printer limited the presentation of such a small 3-prong structure. Only the most obvious feature, the central long prong, was presented. The prongs on both sides of the small-sized denticle merged to the prong in the middle, and the printed denticle no longer resembled the shape of the designed 3-prong structure.

Table 2. Denticle dimension and spacing between two adjacent denticles on the 3D printed biomimetic shark skin fabric

	Large	Medium	Small
Length (mm)	1.30	0.90	0.62
Width (mm)	1.04	0.84	0.50
D1 (mm)	1.68	1.47	0.87

D2 (mm)	0.84	0.735	0.435
D3 (mm)	1.89	1.60	1.10

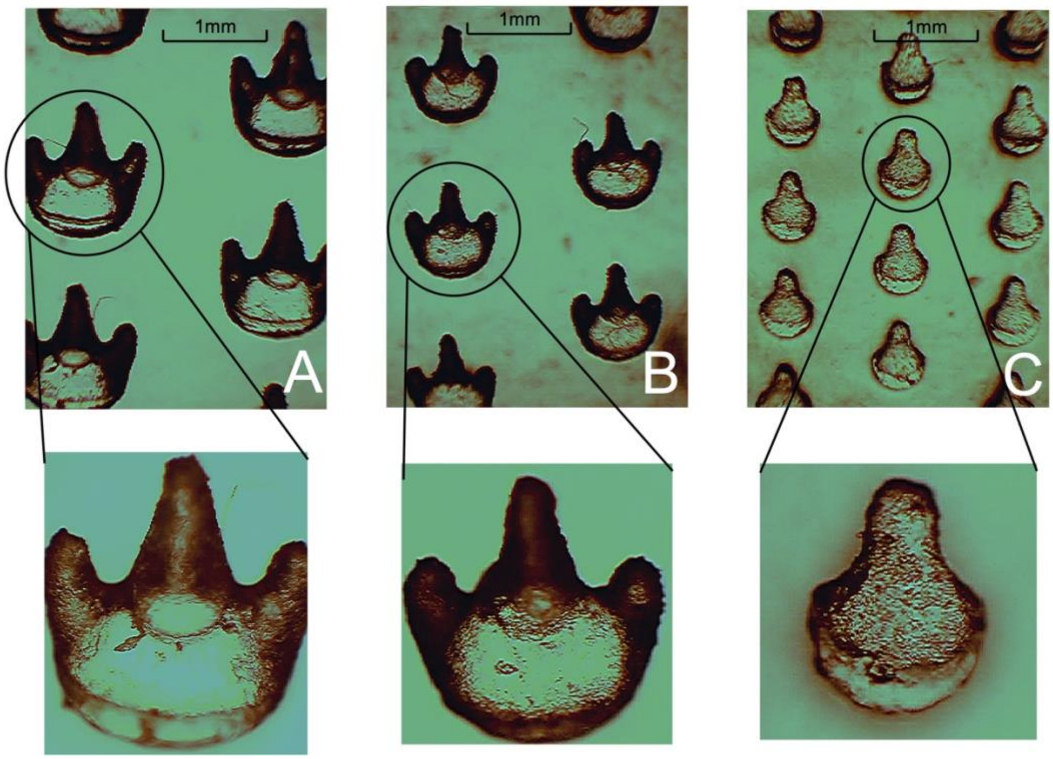

Figure 7. Images of large- (A), medium- (B), and small- (C) sized denticles on biomimetic shark skin fabrics captured by an optical microscope.

4.2 Stiffness Performance Analysis

Figure 8 shows bending angles of shark skin fabrics pressed illustrated the effect of denticle size on stiffness of shark skin fabric. Data was analyzed by ANOVA at 5% significance level. There was statistical significance for the main factor denticle size ($p<0.05$) and denticle density ($P<0.05$), for stiffness. The fabric with large denticles had the largest bending angle, suggesting that it was the softest fabric. The fabric with small denticle showed the smallest bending angle, suggesting it was the stiffest fabric. The results may be greatly associated with different denticle density due to different denticle size. Denticle density is defined by the number of denticles in one cm^2 of the fabric as shown in Figure 9. It was found that the fabrics with small denticles had a high denticle density and small space between denticles. Therefore, the overall thickness of the fabrics with small denticles may be larger than the other two fabrics, which made it difficult to bend, resulting in small bending angle in the fabrics with small denticles. On the other hand, medium- and large- sized-denticle samples had less denticle density and more space between denticles, which lent to the softness of the sample. In a summary, the large denticles that had large spacing between adjacent denticles promoted the stiffness of the shark skin fabrics. To compare the shark skin fabrics with currently used fabrics in swimwear, a nylon spandex blend was also measured using the same testing method and the bending angle was 53.8 ± 3.03 degrees. Therefore, the 3D printed fabric in this study was much stiffer than the nylon spandex blend. The stiffness of shark skin fabric may reduce the comfort level of the functional clothing if it was used, which is still a challenge of using 3D printed shark skin fabric in functional clothing.

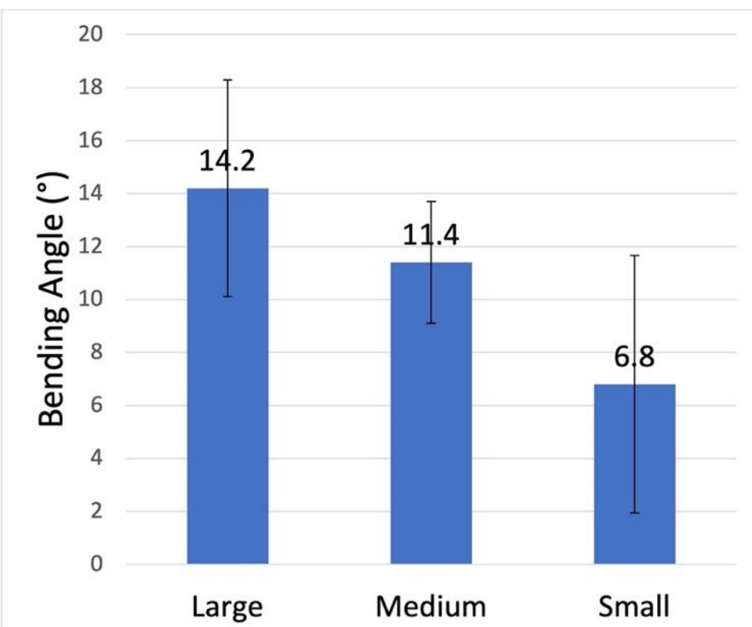

Figure 8. Bending angle for biomimetic shark skin fabric with different denticle sizes ($p<0.05$).

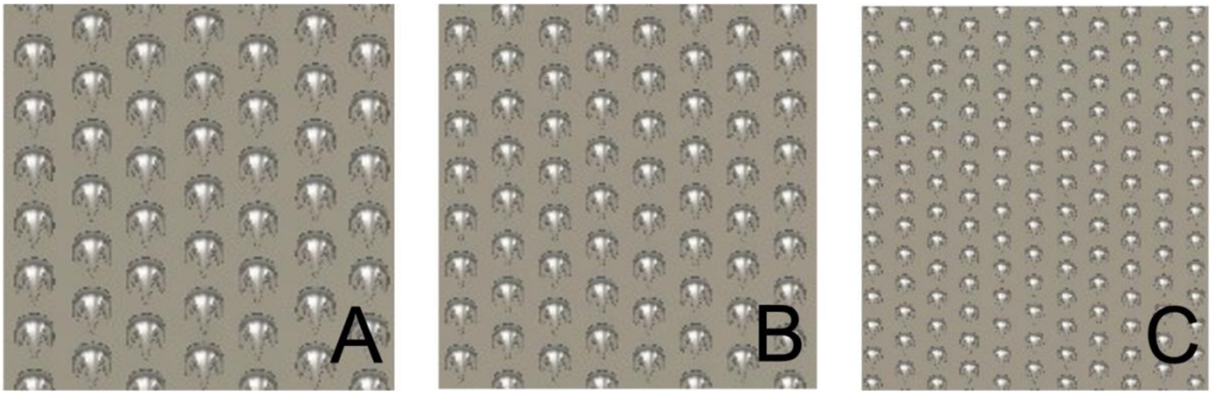

Figure 9. Denticle density of biomimetic shark skin fabric with large-sized denticles (A. 49 denticles/cm2), fabric with medium-sized denticles (B. 72 denticles/cm2), and fabric with small-sized denticles (C. 154 denticles/cm2).

4.3 Mechanical Properties Analysis

Figure 10 shows the stress-strain curves of biomimetic shark skin fabrics with different denticle sizes in lengthwise and widthwise directions. Table 3 shows three mechanical properties including tensile strength, young's modulus, and breaking elongation, which are critical to textile performance testing.

Table 3. Tensile strength, Young's modulus, and elongation at break of biomimetic shark skin fabric with different denticle sizes in lengthwise and widthwise directions ($p<0.05$).

Size (direction)	Tensile Strength (Mpa)	Young's Modulus (Mpa)	Elongation at Break (%)
Large (Length)	4.21±0.58	8.68±1.91	155.57±31.09
Large (Width)	6.14±0.61	12.20±2.00	154.12±18.03
Medium (Length)	5.74±1.47	9.84±2.23	185.24±34.78
Medium (Width)	7.52±1.82	15.42±0.83	156.86±29.40
Small (Length)	5.62±1.08	8.83±3.15	172.84±15.36
Small (Width)	8.75±1.13	12.80±1.52	200.13±13.39

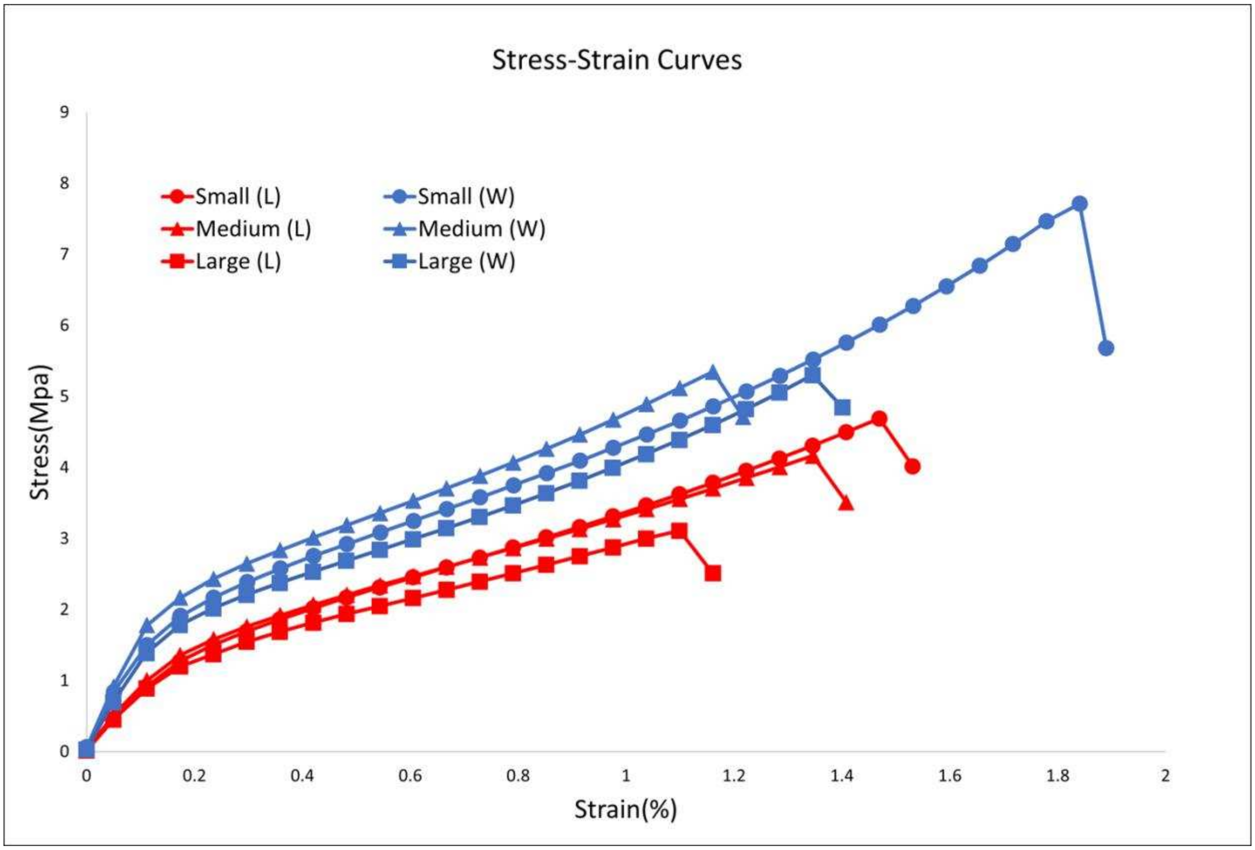

Figure 10. Stress-strain curves of biomimetic shark skin fabrics with different denticle sizes in the lengthwise and widthwise directions.

First, in a comparison of lengthwise and widthwise directions, all of the fabrics in widthwise direction had higher tensile strength, Young's modulus, and breaking strength, than those in the lengthwise direction, suggesting mechanical anisotropy in the 3D printed fabrics. The mechanical anisotropy of the biomimetic shark skin fabrics with different denticle sizes was about 31%-56%. Mechanical anisotropy was previously reported in polymeric parts fabricated using additive manufacturing techniques, such as DLP (Zohdi & Yang, 2021). Zohdi and Yang (2021) have reported that the mechanical anisotropy of a part made by DLP was around 5%. The higher percentage was given because denticles were arranged in the staggered-overlapped pattern, which included the denticles aligned in a straight line in the lengthwise direction and the denticles arranged in a zigzag pattern in the widthwise direction. The denticles in a zigzag pattern in widthwise direction might be more favorable in mechanical testing than the denticles in a straight line in lengthwise direction, resulting in better mechanical properties in widthwise direction than in lengthwise direction. Additionally, the mechanical anisotropy might be due to the uneven thickness of the fabric base layer produced during 3D printing.

Figure 11 shows tensile strength (a), Young's modulus (b), and breaking elongation (c) of the shark skin fabrics with different denticle sizes. The tensile strength of the fabric with the small denticles in the widthwise direction was found greatest as shown in Figure 11(a). When the denticle size was increased by 50% from small denticle to large denticle, the tensile strength was decreased by 29.67% from 8.73 MPa to 6.14 MPa. When the denticle size was increased by 20% from medium denticle to large denticle, the tensile strength was decreased by 18.35% from 7.52 MPa to 6.14 MPa. Therefore, the tensile strength increased with a decrease in the denticle size. On the other hand, in the lengthwise direction, the fabric with medium denticles had the highest tensile strength and breaking elongation. The shark skin fabric with medium size denticles had the most elongation at the point of structural failure in the lengthwise direction. The small denticle size could help increase the breaking elongation of the fabrics in the widthwise direction more than the other sized denticle fabrics. Therefore, there may be a certain denticle size that capitalizes on the opposing properties of smaller and larger denticles' tensile strength. However, this critical point was not easily recognizable within the measured size range in the widthwise direction due to its mechanical anisotropy. The shark skin fabric with medium size denticles had the highest Young's modulus as shown in Figure 11(c), suggesting high tensile stiffness in lengthwise and widthwise directions (Zukor, 1960). The Young's modulus of the shark skin fabric with the same size denticles was different in two directions, due to the mechanical anisotropy.

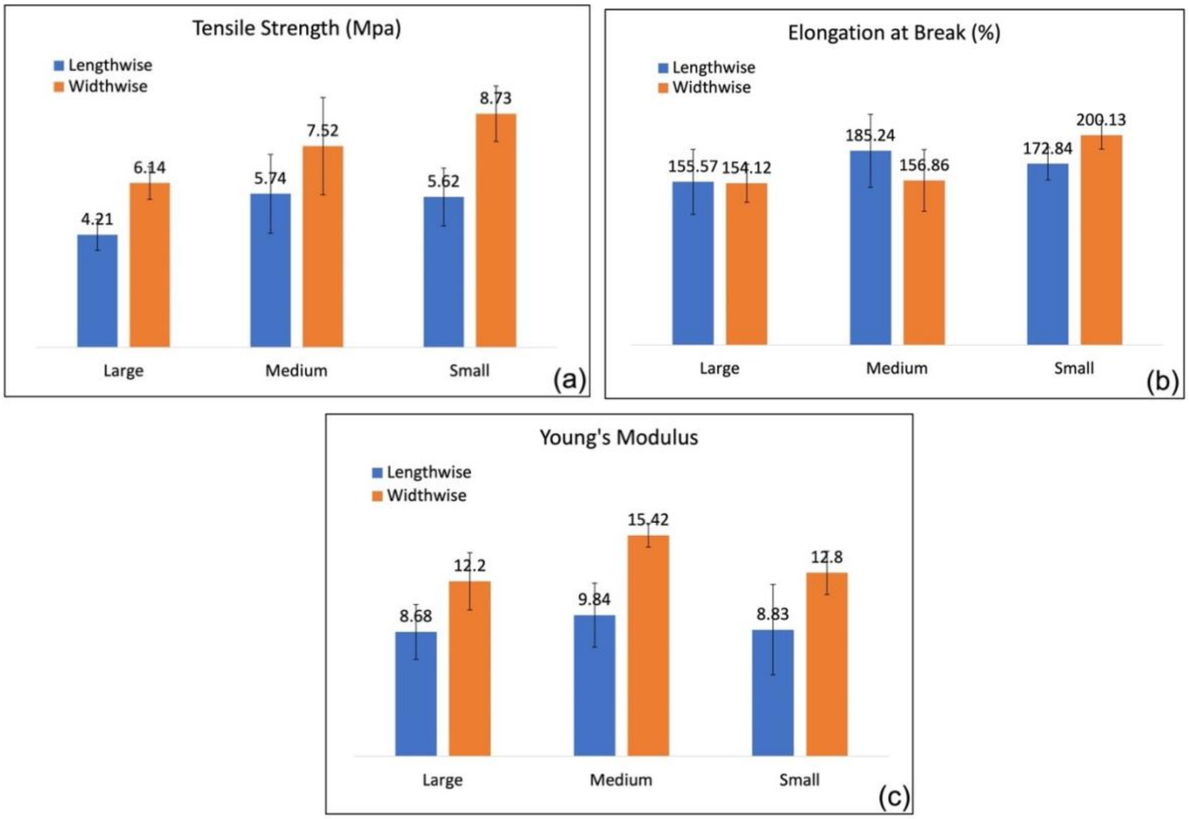

Figure 11 Results of tensile strength (a), elongation at break (b), and Young's modulus (c) for biomimetic shark skin fabric with different denticle sizes ($p<0.05$).

The results of mechanical testing of biomimetic shark skin fabric suggested that the 3D printed shark skin fabrics had the potential to be integrated into competitive and commercial swimwear designs. Typically, fabrics are mechanically anisotropic due to the weaving structure and fiber content (Klevaitytė & Masteikaitė, 2007). Therefore, mechanical anisotropy of 3D printed shark skin fabrics would not be an obstacle to potential applications in functional clothing. According to Manshahia and Das (2014), nylon or polyester fibers that are often used in swimwear have a maximum tensile strength around 5.4 Mpa. The minimum tensile strength of the medium-sized-denticle shark skin fabric was 5.74 Mpa. The biomimetic shark skin fabric elongation was between 154.12 % and 200.13% while the maximum elongation of commonly used nylon-spandex blends is only 40.49% (Zhu et al., 2006). Therefore, the 3D printed biomimetic shark skin fabrics had higher tensile strength and elongation than the fabrics currently used in the swimwear market, indicating a great potential as an innovative fabrics for sportswear applications.

Statistical analysis

The testing results were subjected to 2-way ANOVA tests and hence assessed to determine the effects of denticle size and mechanical anisotropy on the properties of biomimetic shark skin fabric. It was found that there were significant differences in tensile strength, Young's modulus, and elongation at break showed by the fabrics with different denticle sizes at 0.05 level ($p<0.05$). However, the differences between lengthwise and widthwise directions of the fabrics were not statistically significant ($p\geq0.05$). In addition, the denticle size and the direction did not have combined effect on the tensile strength, Young's modulus, and elongation at break ($p\geq0.05$).

4.4 Antimicrobial Properties Analysis

The immersion assay method was used to evaluate antimicrobial properties of the 3D printed shark skin fabrics. Figure 12 shows the results of a typical antimicrobial testing. The photographs show *E. coli* growth area after 24 hours for the fabrics with different size denticles as well as the control fabric sample (without denticle). The control fabric sample's *E. coli* growth area was larger than the fabrics with medium and small denticles, but smaller than the large-sized denticle fabric. The *E. coli* growth area for the fabric with large denticles was found large as shown in Figure 12B, while the fabrics with medium and small denticles had the small *E. coli* growth area. Figure 14 shows the average of bacterial growth area on each sample. The area of bacteria growth was reduced from $6.01cm^2$ in control groups to $2.41cm^2$ in samples with medium denticles and to $2.25cm^2$ in samples with small denticles, respectively. The reduction in comparison with the control sample was 60 % ($p<0.05$) with medium denticles and 63%

($p<0.05$) with small denticles, respectively. The results were statistically significantly different, suggesting good antibacterial properties demonstrated by the small and medium denticles. On the other hand, the bacterial growth area was increased by 42% ($p \geq 0.05$) on the 3D printed shark skin fabric with large denticles compared to smooth controls. However, the increase in bacterial growth was not statistically significant, suggesting that the antimicrobial behaviors of the control group and the sample with large denticles were not significantly different. In addition, the statistical comparison between the medium denticles and the small denticles was not significantly different ($p \geq 0.05$) either. It was probably because the 3-prong structure of the small-sized denticle was not completely and accurately printed due to the resolution of the printer as shown in Figure 14. Another hypothesis to explain the differences is associated with actual surface area of the 3D printed shark skin fabrics. When the denticle size of the fabrics was changed, the surface area of the fabric was also changed. The surface area for each fabric shown in Figure 13 was theoretically estimated according to the 3D model with different denticle size. Overall, the biomimetic shark skin fabrics had higher surface areas than the control sample without denticle ($1cm^2/1cm^2$). Although the fabric with large denticles had the largest surface area, no statistically significant changes in antimicrobial properties were found in the fabric with large denticles in comparison with the control smooth sample. On the other hand, when the size of the denticle was changed from large, to medium and then small, the surface areas of the fabrics with medium and small denticles were slightly smaller than that of the fabrics with large denticles, but still larger than that of the control sample. The spacing between adjacent denticles was reduced when the denticle size was decreased, resulting in small spaces between denticles for the fabrics with small denticles. When bacteria were introduced on the fabrics, the attachment of the bacteria to the fabric surface was likely reduced due to the steric hindrance, which was able to improve antimicrobial properties of the fabrics (Pu et al., 2016). It is in a good agreement of the antimicrobial property results. In a comparison of the fabrics with medium denticles and small denticles, however, the differences in the surface area were trivial, which might explain why no statistical differences were found in their antibacterial testing. The results suggested that steric hindrance is essential for improving antimicrobial performance of the 3D printed shark skin fabrics. The steric hindrance was determined by the denticle size as well as the spacing between adjacent denticles. Future work could include a study of steric hindrance on antimicrobial performance of the shark skin fabrics.

In summary, samples with large-sized denticles did not demonstrate antimicrobial properties, while samples with medium- and small-sized denticles exhibited significant antimicrobial properties.

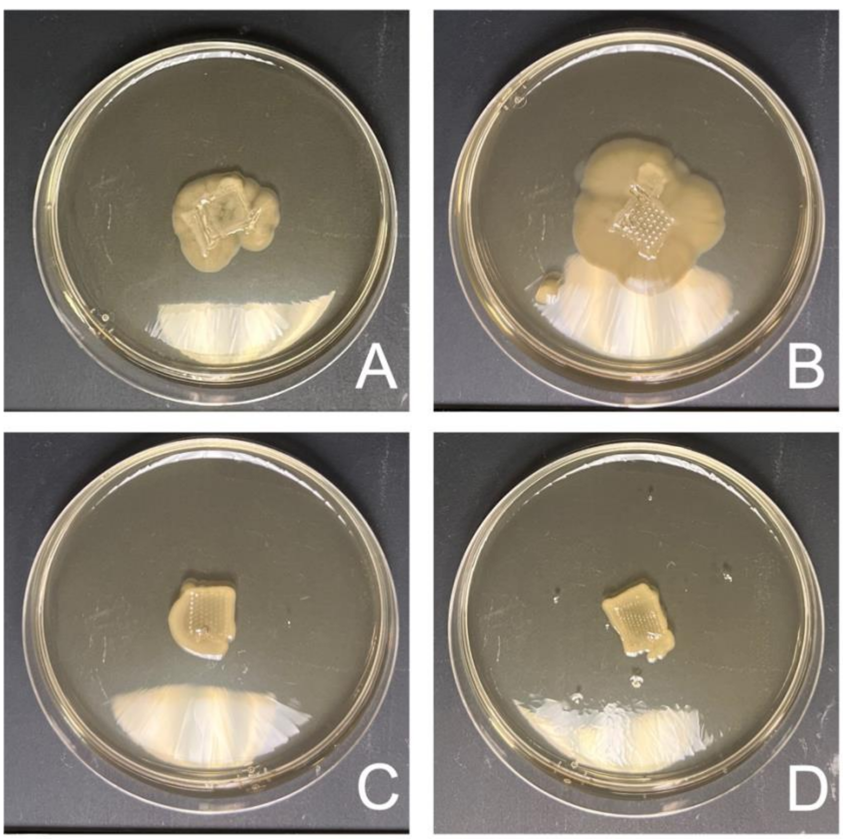

Figure 12. Antimicrobial testing results of *E. coli* growth for control sample (A), sample of fabric with large-sized denticles (B), sample of fabric with medium-sized denticles (C), and sample of fabric with small-sized denticles (C).

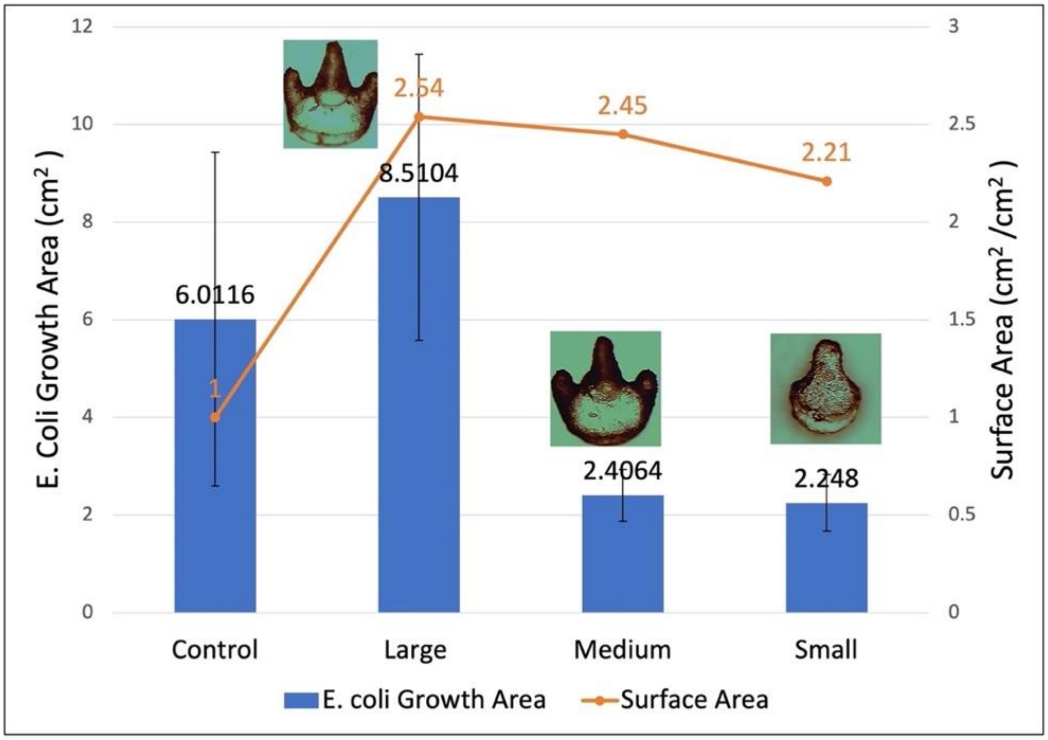

Figure 13. Antimicrobial testing results and surface area, average (n=5) of *E. coli* growth area in smooth control groups (Control), 3D printed shark skin fabrics with large size denticles (Large), medium size denticles (Medium), and small size denticles (Small).

CHAPTER 5 CONCLUSION

5.1 Conclusion

This study investigated the fabric performance properties of 3D printed biomimetic shark skin fabrics decorated with different size denticles (small, medium, large), including (1) stiffness performance, (2) mechanical properties, and (3) antimicrobial properties of 3D printed biomimetic shark skin fabrics. When the size of the denticle was decreased, the stiffness of the biomimetic shark skin fabric was increased. All of the tested fabrics exhibited mechanical anisotropy to different degrees. The biomimetic shark skin fabric in the widthwise direction (3 prongs of the denticle parallel to horizontal direction) had higher tensile strength, Young's modulus, and breaking strength than those in the lengthwise direction (3 prongs of the denticle parallel to vertical direction). Additionally, the fabric with the small denticles had the largest tensile strength and breaking elongation. The fabric with medium- and small-sized denticles showed good antimicrobial properties, while fabrics with large denticles did not demonstrate antimicrobial properties.

In a summary, the biomimetic shark skin fabrics with small denticles showed great potential for integration into swimwear production in terms of mechanical properties and antimicrobial properties, The results provided guidance in continuous development of 3D printed shark skin fabrics for functional clothing and textile innovation.

5.2 Future Work

The application of 3D printed shark skin fabrics in functional clothing and textile innovation is still challenging. First, hydrodynamic properties of the biomimetic shark skin fabric developed in this study was not conducted in this study. Therefore, future study of hydrodynamic properties of the fabrics with different denticle size can be conducted so the effect of denticle size on hydrodynamic properties of the fabrics can be evaluated.

Second, the denticle size in the current and previous studies was still larger than that found in nature. With advancement in 3D printing technology, printing the shark skin fabric with denticle size close to nature permit the exploration of the hypothesis that fabric performance would be increased. Future work can focus on creating small denticles with precise dimensions using a printer with high resolution.

Third, in the current model design of biomimetic shark skin fabrics, the spacing between the denticles on the fabric shrank with a decrease in the denticle size. Future study may include a study on different denticle size with constant spacing between adjacent denticle.

Last, fabric comfort is critical to functional clothing and is usually assessed using hand value measurement. Kawabata Evaluation System (KES) is commonly used to evaluate hand value of performance fabrics. Therefore, future work may include KES measurements for 3D printed biomimetic shark skin fabrics.

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