IMPACT OF PROCESS PARAMETERS ON MECHANICAL PROPERTIES OF 3D PRINTED POLYCAPROLACTONE PARTS

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Hootan Mehraein
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IMPACT OF PROCESS PARAMETERS ON MECHANICAL PROPERTIES OF 3D PRINTED POLYCAPROLACTONE (PCL) PARTS

The following faculty members have examined the final copy of this thesis for form and content, and recommend that it be accepted in partial fulfillment of the requirement for the degree of Master of Science with a major in Industrial Engineering.

___________________________________________
Anil Mahapatro, Committee Chair

____________________________
Nils Hakansson, Committee Member

____________________________
Ramazan Asmatulu, Committee Member
ABSTRACT

Rapid prototyping is an innovative and powerful technology used for direct layer-by-layer fabrication of parts from a 3D computer model. Fused Deposition Modeling (FDM) is the most common and simplest technique in 3D printing technology. Fused Deposition Modeling technology has attracted many researchers, especially those interested in its biomedical applications in custom-made implants, tissue and cell printing and the dental industry. Polycaprolactone (PCL) is both biocompatible and biodegradable and generally used in fabrication of scaffolds in tissue engineering using FDM technology. Although there are reports on impact of different process parameters on parts performance of Polylactic Acid (PLA) using FDM technology there is no study about the effect of process parameters on 3D printed PCL parts. Therefore, in this thesis, we report the effects of 3D-printing parameters such as nozzle temperature and flow rate on a polycaprolactone tensile specimen fabricated by fused deposition modeling. Printrbot Simple metal heated bed 3D printer was used to optimized with polycaprolactone. Seven nozzle temperatures (115°C, 125°C, 135°C, 145°C, 155°C, 165°C and 175°C) and with optimized nozzle temperature remain constant six different flow rates (100% [2.2 mm³/s], 105% [2.31 mm³/s], 115% [2.53 mm³/s], 125% [2.75 mm³/s] 135% [2.97 mm³/s] and 145% [3.19 mm³/s]) were tested to determine optimal parameters. Also with optimal printing condition remaining constant various process parameters such as infill density, layer height, and shell perimeters were investigated to find the optimal process parameters. The result suggested nozzle temperature of 165°C and flow rate of 135% results in a defect free PCL 3D printed test specimen and infill density of 90%, layer height of 0.1 mm and shell perimeter of 2 produce the strongest test specimen. Therefore, these data can assist designers to better understand the behavior of 3D-printed PCL material.
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CHAPETER 1: INTRODUCTION

1.1 Background to the Study

Rapid prototyping (RP) is defined as a set of technologies that produce three-dimensional (3D) objects based on design data with the aid of a computer [1]. The common materials used in RP equipment are varied depending on the type of technology from polymer (synthetic or natural) [2] to ceramics [3] and metals [4]. Rapid prototyping process begins by drawing the model in any CAD professional software, followed by slicing the model layer by layer, generating the g-code and finally 3D printing the part [5]. There are different types of additive manufacturing technologies, however, the most common types are Fused Deposition Modeling (FDM) [6], Stereolithography (SLA) [7], Selective Laser Melting (SLA) [8]. Additive manufacturing technologies also referred to as 3D printing. 3D printing has contributed to many industries such as automotive, aerospace and biomedical fields. Freedom of design and limited material selection are some of advantage and limitation in 3D printing. 3D printing in biomedical industry has a wide range of applications in consumer goods and medical prosthesis. The applications include the production of customized hearing aids, dental crown bridges, surgical instruments [9], artificial joints [10] and artificial hips [11] and tissue and cell printing [12].

Polylactic acid is a polymer that is both biocompatible and biodegradable used in FDM 3D printing technology for biomedical devices. Therefore, within the last decade, there has been significant research using FDM technology as opposed to traditional methods in biomedical applications such as tissue engineering. Tissue engineering aims to regenerate or repair damaged or lost tissues using stem cells, a scaffold and a growth factor. An ideal scaffold is necessary to achieve this goal [13]; therefore, developing scaffolds that resemble tissue extracellular matrix is necessary. FDM technology has
contributed to tissue engineering by helping researchers fabricating different types and shapes of scaffold.

With advancements in material technology, medical devices have benefitted from different types of materials such as different polymers. The most common polymers that can be used in FDM technology are polyethylene, polypropylene, polyamide, ABS, polyester, polycarbonate and polycaprolactone. However, among these materials only a few such as polylactic acid and polycaprolactone are both biodegradable and biocompatible, therefore they have the potential to be used in biomedical devices that require these properties. Fabricated 3D printed part using aforementioned polymers result in different mechanical properties from those of materials manufactured using traditional methods such as injection molding or Computer Numeric Control (CNC). Even though polycaprolactone has been studied in tissue engineering, there have been limited studies in regard of effect of process parameter on the mechanical property of PCL 3D printed parts. Rapid prototype failure could be a major issue, especially in biomedical applications. Therefore, a good understanding of a material’s physical properties greatly enhances a designer’s confidence in developing more reliable objects.

1.2 Motivation

Rapid prototyping is used in many fields such as aerospace, automotive and medical fields. Accurate understanding of mechanical behavior of materials is a critical requirement for any industry that uses RP, especially the biomedical field. In the biomedical field, much research has been conducted to investigate the effects of different parameters on the strength of different thermoplastic polymers such as Polylactic Acid (PLA) Acrylonitrile Butadiene Styrene (ABS) using FDM technology. However, Polycaprolactone (PCL) which is one the material that is both biocompatible and biodegradable with the melting temperature of 60°C which makes it an ideal material for FDM technology hasn’t gained lots of attention by researchers. Examining the literature shows that there have been no studies on the effect of
different process parameters on mechanical properties of PCL made parts by a 3D printer using FDM technology. Thus, there is a need for further research in this area because these properties will assist designers to better understand the properties of 3D-printed PCL. In order to accomplish our objective, the specific aims of the thesis are as follows:

1.3 Study Objective

Specific Aim 1: Optimize 3D printing parameters to fabricate a defect-free polycaprolactone tensile specimen.

Specific Aim 2: Study the impact of process parameters on mechanical properties of 3D printed polycaprolactone parts.

1.4 Thesis Outline

This thesis is divided into four chapters. Chapter 1 presents the introduction, and Chapter 2 presents the literature review, and its applications and limitations. Chapter 3 presents thesis. In Chapters 4, future research is discussed.
2.2 Introduction to 3D Printing

Rapid prototyping, also known as additive manufacturing or 3D printing [14], is described as a quick process for creating a prototype based on design data with the aid of a computer [1]. Since the introduction of 3D printing technology in the 1980s, the term “3D printing” has been used in many industries such as the automotive [15], aerospace [16], electronics and biomedical [17] industries. 3D printing was developed in the 1980s when Charles W. Hull invented the early 3D printing prototype called Stereolithography (SLA), which was based on laser technology and a photo-curable polymer. Since then, many other scientists and researchers have been developing other forms of 3D printing technologies such as SLS by Deckard [18], FDM by Crump [18] and many other technologies.

2.2.1 The Rapid Prototyping Process

Rapid prototyping involves steps starting from creating a 3D-model CAD file to 3D printing the final object. Gibson, in his work entitled “Additive Manufacturing Technology” [5], summarized the process as follows:

Step 1: CAD – To start the process, all parts should start from a software that describes the geometry of the model. Any professional CAD solid-modeling software can be used.

Step 2: Conversion of the model into an STL file – Most CAD softwares are capable of converting the model into an STL-file format, which has been the standard for 3D printers. There is a variety of 3D-printing slicer software available on the market. Some of them are Cura, Slic3r, Repetier and FreeCAD [19].
Step 3: Transfer of the STL file to an Additive Manufacturing Machine and manipulation of the STL file – The STL file must be transferred to the machine. At this stage, it may be necessary to change the file to the correct size, position and orientation.

Step 4: Machine Setup – At this stage, the machine must be set up prior to the building process. Parameters that must be set are layer thickness, nozzle and bed temperature, infill density, etc.

Step 5: Building – This step is mainly an automated process, and the machine can carry on with minimal human control; however, the only human contribution required for this stage is making sure that the machine has enough material and reliable power.

Step 6: Removal – Once the machine has completed the 3D object, the object must be removed for the machine.

Step 7: Post-processing – At this stage, the completed object may require additional cleaning up or removing the support material, which may require more time and attention.

Step 8: Application – At this stage, the part is ready to use; however, the part may require additional treatment such as priming and painting depending on its usage.

2.2 Additive Manufacturing Technology

There have been several types of additive manufacturing technologies such as Stereolithography (SLA), Digital Light Processing (DLP), Fused Deposition Modeling (FDM), Selective Laser Sintering (SLS), Selective Laser Melting (SLM), Electronic Beam Melting (EBM) and Laminated Object Manufacturing (LOM). However, in recent times few technologies such as FDM, SLA, SLS, and LOM have gained commercialized significance by different industries and researchers [4].
Fused Deposition Modeling

Fused deposition modeling (FDM) [6] is the most common and simplest techniques in 3D printing technology that was developed by Stratasys in 1989. Fused deposition modeling uses a wide range of thermoplastics, such as ABS, nylon, PLA and their blends [20]. The FDM process starts when a thermoplastic filament is melted in a liquefier at a temperature above its melting point and then pushed through a nozzle of a given diameter. As the nozzle moves, the molten thermoplastic filament is deposited layer by layer in the horizontal direction on a heated bed (figure 2.1). Once the first layer is printed, the nozzle starts printing the second layer on top of preceding layer and continuing until the completion of the objects.

FIGURE 2.1. Fused Deposition Modeling Technology.
(Image courtesy: http://www.custompartnet.com/wu/fused-deposition-modeling)
2.2.2 Stereolithography

Stereolithography (SLA) [7, 21] is a method of 3D printing that uses laser and a photo-curable liquid resin. It was developed by Charles W. Hull in the 1980s and is commonly called the first commercial 3D printer. The SLA process begins when the liquid resin is filled in the vat of the 3D printer and the elevator table set just below the surface of the liquid resin (figure 2.2). Then the optical scanning system that is controlled by a microchip directs the laser beam to solidify the resin in appropriate areas. The elevator tables then lower and the laser draws the next layer; this process continues building a part from the bottom up until the part is finished. There are various materials that can be used using SLA technology giving a wide range of properties, for example “3D system company” specialized in SLA technology sells various inks that provide similar properties to thermoplastics counterparts, although composition of these ink haven’t been disclosed, they’ve classified these material based on properties such as Polypropylene-like class, Tough/Durable class, ABS-like class, casting class, and high-temperature and composite class [22].
2.2.3 Selective Laser Sintering

Selective laser sintering (SLS) is a method of 3D printing developed and patented in 1999 by Dr. Carl Deckard. It uses laser to sinter a powdered material by fusing the powder to form a solid mass [8]. Selective laser sintering is similar to SLA in that both use laser to solidify a material. However, in SLS, the material that the high-energy laser fuses is in powder form. The high-energy laser selectively fuses a section of a powder material according to customer specification, and then a new layer of powder is deposited on top as the entire bed drops and the laser does another sweep, fusing the single layered powder until the part is finished [4].
2.2.4 Laminated Object Manufacturing (LOM)

One of the earliest AM technologies was the Laminated Object Manufacturing (LOM) system from Helisys, USA. This technology used a laser to cut out profiles from sheet paper, supplied from a continuous roll, which formed the layers of the final part. Layers were bound together using a heat-activated resin that was coated on one surface of the paper. Once all the layers were bound together, the result was very like a wooden block. A hatch pattern cut into the excess material allowed the user to separate away waste material and reveal the part [5].
2.2.5 Advantages of 3D Printing

Many fields have been benefited from 3D printing technology. The advantages of 3D printing have been summarized below:

1. Freedom of design: 3D printing can produce any shape of any complexity at a fraction of the cost compared with traditional methods.

2. Reduction of fabrication time: A 3D printer can produce objects relatively faster than traditional methods such as CNC [9].

3. Assist in prototyping: 3D printing can assist in detecting errors and flaws at an early stage of production [23]. For example, by 3D-printing a prototype, designers and engineers can detect any flaws before the production of any parts.
4. Waste reduction: 3D-printing a prototype can significantly reduce material waste as the price of the material is relatively cheaper than traditional methods.

5. Eliminate tooling: The 3D printing process, unlike traditional methods, does not require tooling as 3D printers are capable of quickly producing complex solid objects directly from a computer model without the need for tools [24].

2.2.6 Limitations of 3D Printing

Even though 3D printing has contributed to the many fields; however, there are still some limitations which need to be addressed and further researched to overcome them.

1. Poor surface finish: Since their surface is usually rougher than machined surfaces, 3D-printed objects could have a poor surface finish [25].

2. Limited material selection: Three-dimensional printers are limited in material selection. For example, FDM technology is limited to materials that fall within the temperature of nozzle, therefore very limited materials are available given the range of melting point.

3. Anisotropic property: The strength of RP parts is weaker in the Z-direction than in other directions or the printed objects are anisotropic [26, 27]; therefore, the accuracy of 3D printing in the Z-direction is typically worse than that in either the X- or Y-direction [28].

2.2.7 Applications of 3D Printing

Rapid prototyping has been widely used in many industries such as the automotive, aerospace and electronics industries [27]. In this section, we review some of the 3D printing applications using different technologies.

One of the advantages of using FDM technology is geometric flexibility in the design. Bourell and coworkers studied the design of an intake manifold for a formula SAE car using FDM technology and a
composite material. The freedom in the design helped the researchers to create a unique geometry that
featured a tapered plenum and tapered runners. The results suggested that the new design provided more
torque and reduced total pressure drop [29].

Rapid realization of designs without going through conventional techniques such as machining or
photolithography is one of the advantages of 3D printing. Liang and coworkers investigated the design
and fabrication of a 3D printed microwave patch antenna. A microstrip patch antenna working around
7.5 GHz 3D printed using FDM technology. The results presented a good resonance peak at 7.5 GHz,
which was similar to a conventional method [30].

Gas chromatography (GC) is a technique that is used to distinguish different molecular compounds
and is usually composed of an injection unit, a separation column, and a non-selective gas sensor. The
design of GC the column is a critical aspect in GC. Lucklum and coworkers designed and fabricated a
miniature separation column for a compact, mobile gas chromatography using the SLA technique. The
results suggested that a 3D-printed column when compared to rectangular geometry fabricated in silicon
showed improved separation capabilities due to design freedom in 3D printing technology that helps
create 3D printed column with round cross-section geometry and subsequently optimizing the design.
Therefore, 3D printing is one of the techniques that should be considered for further research in this area
[31].

The ability of a researcher to choose the right 3D-printing technology is very important in
aerodynamic testing. Olasesl and Wiklak manufactured four models of a standard NACA0018 aerofoil
for aerodynamic testing in different methods (Multi Jet Modeling (MJM), SLS and FDM). The results
showed that surface roughness in MJM method is the lowest, while surface roughness is higher in FDM,
also suggested small holes quality is worse in SLS and better in MJM while it was omitted from design
for FDM due to its limitation. Therefore, it was possible to 3D-print small holes with a very high length-
to-diameter ratio as well as a very small object. However, certain 3D-printed models such as FDM technology are not yet suitable for small dimensions holes [15].

With the advancement in the precision of 3D-printing technology, especially SLA, scientists have investigated fabrication of microfluidics. Using SLA, Kanai and Tsuchiya fabricated microfluidic devices with 3D flow channels and three coaxially cylindrical channels [32]. Hydrophilic silica coating was used to control the wettability’s of oil-in-water-in-oil (O/W/O) channels. Silicone oil was also used for the inner and outer phases, and ultrapure water and a surfactant as a middle water phase. The results suggested that monodisperse O/W/O double emulsion could be generated by the fabricated device with precise control of the size and number of the encapsulated droplets. Therefore, the authors concluded that this method could potentially be useful in drug delivery, food, etc [32].

2.3 3D Printing for Biomedical Applications

3D printing in the biomedical industry has a wide range of applications in consumer goods and medical prosthesis. The applications include the production of customized hearing aids, dental crown bridges, surgical instruments [9], artificial joints [10] and artificial hips [11] and tissue and cell printing [12].

2.3.1 Dental Industry

Orthodontics in dental industry has benefited from rapid prototyping. One of the areas that have benefitted from 3D printing is bracket production and positioning. Using a stereolithography 3D printer, bracket production and positioning are fused together as one unit [33]. Creation of 3D physical models of a patient’s skull and other structures gives an oral surgeon a realistic impression of structures before surgery. This method helps in minimizing the extra-oral time and injury while translating teeth [33]. Another application of 3D printing in dental industry is implant surgery simulation. The author of a paper entitled “Rapid Prototyping Mandible Model for Dental Implant Surgery Simulation” investigated the
usage of Polyjet technology in production of a real-mandible model for implant surgery simulation. The result suggested that 3D printing technology can produce a real mandible to guide researchers and dental surgeons to simulate a surgery [34].

With the advancement of 3D printing technology, dental laboratories are now looking for ways to save cost and increase the quality of their products. Using ultraviolet light technology to cure resin, authors of the paper entitled “New Design for Rapid Prototyping of Digital Master Casts for Multiple Dental Implant Restorations” produced dental casts without analogs and compared them with a conventional master cast. The results showed a faster, more accurate and lower-cost process with higher quality. Therefore, it is possible to use the rapid prototyping technology to produce digital dental casts to reduce cost and improve quality [35].

2.3.2 Prosthesis

3D printing technology has benefitted prosthesis fabrication by reducing the time needed to obtain patient-specific anatomical structure image which leads to patient-specific need such as size and shape of the design. Jardini and coworkers studied the potential of additive manufacturing in treating a patient with a cranial defect. A 3D scan of a patient’s skull with a cranial defect was obtained and sliced, then a cranial section was successfully 3D-printed using direct metal laser sintering (DMLS) with Titanium Ti64 ELI material. The result indicated that DMLS technique has potential in the production of patient-specific implant [36]. A factor that plays a vital role in knee implant is kinematics and retropatellar pressure. Shroder et al. compared the effect of 3D printed prostheses made of photo-polymerized rapid prototype material with that made of cobalt-chromium alloy prostheses as a validation standard by measuring the sliding friction and knee kinematics and retropatellar pressure on a knee rig [37]. The result showed that prostheses obtained using 3D printing technology are comparable to standard prostheses.
Customization in 3D printing is one of the advantages over traditional methods. Fan et al. investigated the manufacture of customized titanium prostheses for limb salvage surgery using electron beam melting technology. Results from this experiment indicated that 3D prostheses had no surgical complications such as screw loosening and implant breakage. Therefore, the researchers suggested that electron beam melting technology was a useful technique of manufacturing customized titanium prostheses [38].

2.3.3 Tissue and Cell Printing

The goal of tissue engineering is to restore or replace damaged tissue. Cells and artificial Extracellular Matrix (ECMs) also known as scaffold are the important components in tissue engineering. Scaffold provides appropriate condition that support cellular growth, differentiation and proliferation. 3D printing has allowed researchers to create variety of scaffolds using different materials [39].

Some of the important factors for 3D-printed scaffolds that are used in tissue engineering are strength, cytotoxicity, and printability. Aldemir et al. investigated the biocompatibility of modified calcium-sulfate-composite-powered (MCSCP) powder-based 3D-printed scaffolds and compared it with that of salt-coated MCSCP powder-based 3D-printed scaffolds using a Visijet powder based printer for artificial bone application. Mechanical and cell viability tests were performed to determine the strength and cytotoxicity of the printed samples. The mechanical testing result showed MCSCP samples were significantly stronger in comparison to salt coated MCSCP, also cell viability tests suggested that 3D-printed MCSCP had higher cell number and proliferation without any toxic effect compared to salt-coated MSCP scaffolds. Therefore, 3D printed MCSCP scaffolds were biocompatible, non-cytotoxic and printable, whereas salt-coated MCSCP did not have any significant positive effect on the scaffolds. The author suggested for future study of more samples should be used for better interoperation [40].
Bone disorder is expected to double in the next decade [41]. The most common treatments for bone disorders are autografts, allografts, and xenografts that treat different types of bone injuries. However, immune rejection remains one of the drawbacks for different treatments, although the main cause of such a rejection is bacterial colonization. Correia et al. used a Fab@Home syringe-based 3D printer to produce Tricalcium Phosphate (TCP)/Sodium Alginate (SA) 3D scaffolds with silver nanoparticles to overcome bacterial contamination. The results suggested that the scaffolds had mechanical properties that were suitable for bone application [41].

The different 3D printing technology such as powdered based and syringe-based deposition system have been discussed. Although each technology has its own advantages and disadvantages, applications of 3D printing in tissue and cell printing have been rapidly growing in the last decade due to advantages of 3D printing technology.

2.3.4 Training/Planning

Another important area in which 3D printing technology has helped is the training of students in the medical field. Khan and his coworker successfully 3D-printed an unruptured paraclinoid aneurysm of a 40-year-old female using a stereolithographic technique. This type of modeling can be used in biomedical research, patient education, and training of medical students [42].

Surgical training is one of the fields that have benefited from 3D printing technology. Rose and coworkers investigated a temporal bone model using the SLA technique of 3D printing using multiple colors and materials [43]. An Objet350 SLA based 3D printer was used to 3D print temporal bone using multiple colors with different material to distinguish soft from hard tissue. The results indicated that the 3D-printed temporal bone had significant potential in otological training and education. The authors concluded that 3D printing technology’s advantages such as using multiple colors and material outweigh other methods such as injection-molding plastic in surgical training [43].
Guiding surgical treatment with a patient-specific prototype from the fracture site could potentially improve the quality of patient-physician communication and surgery. Yang et al. evaluate the effect of a 3D-printed prototype of trimalleolar fracture and its role in doctor-patient communication by printing actual fracture. FDM technology using PLA material with the layer height of 50 µm and infill of 20% was used to 3D print the ankle joint fracture. In order to validate the design, a questionnaire for doctors was designed and the response showed the prototype met the doctor’s specifications. The results showed the successful 3D printed prototype with detailed anatomical structure of the fractured joint. Therefore, 3D printed patient-specific prototypes can help the surgeon to plan ahead of time and better communicate with patients [44].

2.4 3D Printing Using FDM Technology for Biomedical Devices

FDM printing technology has attracted researchers in biomedical applications due to its availability and price. Therefore, within the last decade there has been significant improvement of using FDM technology as opposed to traditional methods.

To investigate the feasibility of FDM 3D printing in the manufacturing of a capsular device for a drug delivery system using hydroxypropyl cellulose (HPC), Melochi and coworkers studied the possibility of fabricating hollow structures using FDM. In order to make the filament, HPC was used in Turbula with Polyethylene Glycol (PEG) using Hot Melt Extrusion (HME). Then PLA filament used to 3D print hollow structure followed by 3D printing capsular device using in-house HPC filament. Then capsular devices were filled with acetaminophen to determine pulsatile-release profile. The result showed pulsatile-profile was consistent in comparison to injection molding method. Therefore, FDM 3D printers were capable of manufacturing capsular devices for drug delivery system [45].

Pre-formed titanium meshes have been used to assist parenthetically guided bone regeneration of atrophic maxillary arches. Fantini and coworkers manufactured patient-specific biomodel to guide pre-
formed titanium mesh. A 3D 768 Soluble Support Technology (SST) printer, which is based on FDM technology, was used to manufacture a patient-specific biomodels of maxillary arches to build the prototype. In the first approach, the model was filled with ABS material, however, in the second approach honeycomb structure was used to 3D print interior part. Then commercial flexible titanium mesh was customized and shaped with the help of biomodel. The researchers found that both techniques were sufficient to fix the implants in the planned position, thus both techniques can be used for bone augmentation in atrophic maxillary arches [46].

Fabrication of custom-made implants is one of the advantages of 3D printing; however, there is a need to investigate the feasibility of commercial-scale applications of this technique. Jumani and coworkers investigated commercial aspects of the FDM technique with ABS for manufacturing custom-made foot orthoses. They performed an analysis considering machine cost, material cost and production overhead per year to find out the best case for production of custom-made foot orthoses using Dimension SST 768 which uses FDM technology. The model gives the cost of production per pair in 2014 as £182.33 ($285.45) compared to current cost in the market from £150 ($234.84) to £200 ($313.12). The higher cost of FDM is attributed to higher cost of material compared to traditional method. Therefore, the author concluded that in order to drive the cost down there is a need to develop new cost-effective material for the fabrication of custom-made devices such as orthotics and prosthetics. Even though still the cost of fabrication using FDM technology exceeded the conventional methods the FDM technique have some advantages over conventional methods: such as lower lead time, better fitting and improved the final quality of custom-made foot orthoses that can compensate for the slightly higher cost, which the authors did not consider in their model [47].
2.4.1 Tissue Engineering

Tissue engineering aims to regenerate or repair damaged or lost tissues using stem cells, a scaffold and a growth factor. An ideal scaffold is necessary to achieve this goal [13]; therefore, developing scaffolds that resemble tissue extracellular matrix is necessary. Rosenzweig and his coworkers investigated the usage of a 3D printer with FDM technology to develop a scaffold using PLA and ABS. Primary bovine chondrocyte, Primary articular chondrocytes and nucleus pulposus cells were cultured for 21 days on ABS and PLA scaffold printed with a low-cost 3D printer. Primary bovine chondrocyte and NP cells showed high viability on either construct type. Primary articular chondrocytes and NP cells proliferated and filled the space. The result suggested there was no difference between ABS and PLA scaffolds when comparing viability and proliferation. Also, mechanical testing was performed on both scaffolds and the result suggested mechanical properties of scaffolds after 21 days remain unchanged [12]. The authors then concluded that 3D printed PLA and ABS scaffolds both provided good mechanical behavior for tissue engineering applications.

The design and fabrication of porous scaffolds remain a major challenge in bone tissue engineering. Zhou and coworkers introduced an integral manufacturing method using FDM technology with PLA and gas foaming to fabricate hierarchical porous polymer scaffolds. The FDM technique was used to rapidly fabricate macroporosity scaffolds, and the CO₂ gas-foaming technique proved a useful approach to generating micropores, which usually do not form though 3D printing techniques. The results showed positive cooperative effects to fabricate hierarchical porous scaffold for tissue engineering [48].

To fabricate a coronary artery bypass graft (CABG), Owida and coworkers devised a new combined method of FDM and electrospinning to fabricate a CABG. Coronary bypass grafting is a procedure to treat coronary artery disease. However, in some cases, surgeons cannot use their patient’s vessel and there is a need to use artificial grafts which require special design to ensure revascularization and anti-
thrombogenicity. A physical model mold for electrospinning was 3D printed using ABS material and polyurethane (PU) nanofibers was electrospun on the mold to fabricate the CABG graft. The results showed a successful fabrication of a CABG, suggesting that this method could be used for the fabrication of artery vessel [49].

Improving the scaffold in tissue engineering is beneficial in long-term clinical applications. A study by Paricio and coworkers investigated the effect of PLA addition to PCL matrices to determine chemical, mechanical and biological performances of a 3D-constructed scaffold using FDM technology. Melt blending and solvent casting were used to blend PCL and PLA and BioCell printing system was used to produce PCL/PLA scaffolds. To make sure that no chemical modification was induced during the process UV, β and γ radiation were used. Mechanical testing showed addition of PLA to PCL greatly improved the performance of scaffolds under compressive loads. Also, biological evaluation of both scaffolds revealed all scaffolds were able to sustain cell proliferation and adhesion. The results showed that the addition of PLA to PCL scaffolds using solvent casting strongly improved the biomechanical performance of the constructs in comparison to PCL scaffolds alone. The author concluded that addition of PLA improved the mechanical property of PCL [50].

2.4.2 Medical Polymer for FDM Technology

With advancements in material technology, medical devices have benefitted from different types of materials such as different polymers. Examples of medical devices include but are not limited to stent, pacemaker, implants, surgical instruments and imaging machines [51]. A material to be used for 3D printing with the FDM technique is determined based on several factors such as melting temperature, mechanical properties, biocompatibility and biodegradability. The current range of materials that are used directly in FDM technology are polyethylene, polypropylene (PP), polyamide, ABS, polyester, polycarbonate (PC) and polycaprolactone (PCL) [27].
Polyethylene (figure 2.5) can be classified into six categories depending on how it is made: ultra-low-density polyethylene (ULDPE), very low-density polyethylene (VLDPE), linear low-density polyethylene (LLDPE), low-density polyethylene (LDPE), medium-density polyethylene (MDPE) and high-density polyethylene (HDPE) [51]. Among different types of polyethylene, HDPE is commercially available as a filament with a melting point of 130°C [52]. Polyethylene has been used in biomedical implants as the wear bearing surface of knee total joint replacement [53]. Suwanparateeb and coworkers successfully 3D printed Zygoma bone using HDPE material [54].

![Polyethylene Structure](image)

FIGURE 2.5. Polyethylene Structure

Polypropylene (PP) (figure 2.6) has three main types: homopolymers, random copolymers and impact copolymers. The most common types used in FDM technology is the homopolymer, which is available for commercial use and melts at 230–260°C [6]. PP is generally used in syringe, suture and medical trays in medical device application [51]. PP has been used in fabrication of custom made orthoses. Prefabricated orthotic devices are designed to fit patients without considering patient-specific comfort and function, also creating custom-fit orthosis is a very time consuming task using traditional method, however by using 3D printing technology Mavroidis et al. demonstrated successful fabrication of custom made orthoses using PP according to patient specification [55].
Polyamide or nylon (figure 2.7) is one of the materials that have been used in medical devices, is also available in filament form and has a melting temperature of 235°C–270°C [56], which makes it a good candidate for FDM. Polyamide has been used in biomedical devices such as packaging, syringe pump component and scalpel blade holder [51]. Kettner et al. successfully fabricated skull model in a case of fatal hammer impact to the head using SLS 3D printing technology. Digital CT scan of the patient was obtained and Fine polyamide was used to 3D print model of damaged skull [57].

Acrylonitrile Butadiene Styrene (ABS) (figure 2.8) is an amorphous polymer and is made of three monomers: Acrylonitrile, Butadiene and Styrene. ABS have been used in several applications including automotive, electronics and biomedical applications. ABS has good strength and toughness with a melting temperature of around 230°C and a glass temperature of 107-115°C [5]. To determine the feasibility to 3D print patient-specific mold structure and catheter channels in a brachytherapy treatment planning, Harris and coworkers successfully 3D printed ABS using FDM technology [58].
Polyester are polymers of a semi-crystalline structure with two types: thermoplastic and thermosetting polymer [5]. Polylactic acid (PLA) is a polyester that has been used extensively in research and industry. Polyester has been used in sterilizable trays, dental tool, surgical device control cable, surgical tubing and cannula in biomedical applications [51]. PLA is derived from renewable resources, such as corn starch. PLA (figure 2.9) polymers are both biocompatible and biodegradable, therefore, it can be used within human body as it has been proven to be safe in surgical implantation. PLA is available in commercial form with a melting point of 160°C–220°C depends on the printer settings [14].

Polycarbonate (PC) (figure 2.10) is a type of polyester that is formed from the reaction of bisphenol A and carbonic acid. PC is available for commercial use in the FDM technique and has a melting
temperature of 255–300°C and a glass temperature of 145-148°C [5, 59]. PC has been used in medical apparatus, reservoirs, high pressure syringe and dialyzer housing [51]. Smith and coworkers investigated the effects of different orientations on the mechanical properties of 3D-printed PC using FDM technology to understand the material’s properties for designers. The results showed that orientation had a significant effect on mechanical properties; therefore, designers should investigate different parameters to understand the material’s behavior [60]. Chan and coworkers successfully fabricated PC mandible templates using FDM technology [61].

![Polycarbonate Structure](image1)

**FIGURE 2.10. Polycarbonate Structure**

Polycaprolactone (PCL) (figure 2.11) is another biodegradable and biocompatible polyester. PCL filament has a melting temperature of 150°C–250°C and a glass transition temperature of -60°C with a melting point of 60°C [62].

![PCL Structure](image2)

**FIGURE 2.11. PCL Structure**
In this section different types of medical polymers available for FDM technology were discussed such as polyethylene, polypropylene (PP), polyamide, ABS, polyester, polycarbonate (PC) and polycaprolactone (PCL), however not all of them are biocompatible and biodegradable. Among them PLA and PCL are both biocompatible and biodegradable, therefore they can be used in applications that require these properties.

2.4.3 Fused Deposition Modeling Using PCL Material

Polycaprolactone has received much attention from researchers in biomedical applications due to its biocompatibility and biodegradation properties. The most common application of PCL in 3D printing is in tissue engineering [63]. PCL has a low transition temperature of −60°C and a melting temperature of 60°C [64] which makes it an ideal material for FDM technology.

Xu et al. used computed tomography (CT) with the FDM technique to fabricate PCL and PCL/hydroxyapatite (HA) artificial bones to mimic goat femurs. A 3D scan of a normal goat leg was obtained by X-ray CT; subsequently, the 3D model (of artificial bones) was imported into Mimics software and exported in an STL format to be used by the FDM printer. Counter-rotating internal mixer (Rheomix 600 and Rhecord 9000) were used to blend PCL with HA at 100°C for 10 minutes. Using FDM technology, PCL/HA and PCL bone were fabricated. Mechanical testing determined that PCL/HA bone had a higher compressive strength and elastic modulus as compared to PCL fabricated bone. Also, cell proliferation was better in PCL/HA scaffold as compared to PCL scaffolds. Tests also showed PCL/HA results in better bone osteoconduction ability in comparison to PCL. Therefore, PCL/HA 3D artificial bones had several advantages such as superior biometric properties, shaping ability and biomechanics, biocompatibility and easier producibility for manufacturing and operability for a surgeon. The authors suggested that PCL/HA 3D artificial bones could potentially be used to treat load-bearing bone defects and benefit bone repair in comparison to PCL 3D artificial bone [63].
To enhance cartilage tissue engineering, Wang and coworkers prepared a 3D composite scaffold composed of a PCL backbone and poly(lactide-co-glycolide)-block-poly (ethylene glycol)-block-poly(lactide-co-glycolide) (PLGA–PEG–PLGA) thermogel surface. Medical-grade PCL was used at 130°C to fabricate PCL scaffolds using the FDM method. The scaffolds were printed with fiber spacing of 300 µm and a Z axis interlayer increment of 300 µm in order to make the penetration and exchange of nutrient easier. In order to obtain PCL/GEL composite scaffolds, PCL was impregnated into the PLGA–PEG–PLGA solution at a low temperature and incubated at body temperature (37°C) to fabricate 3D thermogel scaffold. To compare mechanical properties of the PCL and thermogel scaffolds with behavior of the real bone, rabbit osteochondral (OC) plug was obtained from femoral condyles. The mechanical testing showed that the thermogel was slightly lower but negligible compared to other scaffolds, and no differences in compressive strength and elastic modulus were observed among the other scaffolds. Moreover, the 3D thermogel composite scaffold showed greater number of cell proliferation compared to other scaffolds. Therefore, the author concluded that thermogel scaffold could be a better scaffold for tissue engineering since it showed better cell proliferation. In this paper, the author used the FDM technique to fabricate PCL scaffolds without considering the effects of different process parameters on mechanical properties of PCL [65].

Steffens studied the interaction between mesenchymal stem cells (MSC) and 3D-printed poly(ε-caprolactone) using a Fab@CTI printer. PCL was fabricated through an extrusion process of additive manufacturing using the FDM technique. Scaffolds were printed with different air-gaps (layer thickness), which are spaces found among successive layers, as follows: 0.1, 0.15, 0.2, 0.3 and 0.4 mm. Different air-gaps were selected in order to see the how well cells proliferate and differentiate in different settings. MSC were seeded for 21 days on PCL scaffold. Cellular viability test showed all the scaffolds have the same behavior with no significant difference. Also, confocal microscopy showed MSC density
increased over a 21-day period in all the groups. The author concluded that a 3D-printed PCL structure could potentially be used in tissue engineering as a good substitute for other material [66].

To investigate the efficiency of coating on biodegradable PCL scaffolds made using FDM, Choong and coworkers investigated the effect of a calcium-phosphate coating on cell attachment, proliferation and differentiation of PCL. PCL material is both biodegradable and biocompatible. However, PCL material exhibit hydrophobic properties which make it hard to grow cell as they don’t allow bonding on the surface of polymer. To overcome this problem the author introduced osteoconductive particles into the scaffold. In this study calcium phosphate used to coat PCL using biomimetic method. The results suggested that cells in coated scaffolds had a higher proliferation rate compared to those in uncoated scaffolds. The author concluded that calcium phosphate coating improved proliferation and differentiation, which resulted in a better 3D PCL scaffold for bone tissue engineering [67].

2.4.4 Effects of 3D Process Parameters

As elaborated in the previous section, researchers have directly applied PCL material and used it, however, the effect of process parameters has never been investigated. To have successful implementation of PCL printed material, the effects of different process parameters on mechanical properties of 3D printed PCL needs to be investigated. This section elaborates on various reports that suggest the importance of optimized FDM parameters to achieve optimal part performance.

Wu et al. investigated the effect of layer thickness and raster angle on mechanical properties of a Polyetheretherketone (PEEK) material using FDM technology. Layer thickness is defined as the thickness of each layer and Raster angel is the direction of raster. PEEK samples with three raster angles (0°, 30° and 45°) and three layer thickness values (200, 300 and 400 µm) were built by 3D printer using FDM technology and tensile, compressive and bending strength were tested. A 400 µm thickness resulted in the lowest tensile strength and bending strength; however, 300 µm layer-thickness samples
had the highest tensile strength and bending strength. Raster angle of 0° had the highest tensile strength and bending strength, while 45° had the lowest tensile strength and bending strength. The result suggested that with 300 µm layer thickness and raster angles of 0° had the greatest tensile strength, thus it is clearly evident that the PEEK were greatly affected by layer thickness and raster angle [68].

Two most common materials used in 3D Printing are PLA and ABS. Tymrak et al. investigated the effect of layer heights of 0.2mm, 0.3mm, 0.4mm and orientations of 0°, ±45° and 90° on the mechanical property of the PLA and ABS. Mendel RepRap, Pursa Mendel and Lulzbot Prusa RepRap were used to 3D print samples at 100% infill. The mechanical testing showed that ABS samples with 0.2 mm had the highest tensile strength, while 0.4 mm samples had the highest elastic modulus and orientations of ±45° were the strongest, while 0° and 90° had the greatest elastic modulus. PLA samples printed with 0.2 mm layer height had the greatest tensile strength and elastic modulus and samples with 0° and 90° had the greatest tensile strength and at 0.3 mm the tensile strength reduced by 22%, while samples with ±45° had then greatest elastic modulus. Therefore, in both PLA an ABS layer thickness of 0.2 mm results in the greatest tensile strength while layer thickness of 0.3 mm results in lower tensile strength, however at layer thickness of 0.4 mm there is an increase in strength compare to 0.3 mm [69].

Another factor which plays an important role on the mechanical property of material is number of shell perimeters. Study by Lanzotti and coworkers investigated the effects of number of shell perimeters on mechanical properties of 3D printed parts. Shell perimeters is defined as number of shells of the exterior skin of the 3D printed parts. Four different shell perimeters such as 3, 4, 5 and 6 were used to 3D print PLA specimen using RepRap 3D printer. The mechanical testing showed the number of shell perimeters is directly correlated to ultimate tensile strength (UTS) of printed specimens. Shell perimeters with the value of 3 had the lowest UTS and value of 6 had the highest UTS. Therefore as the shell perimeters increase the UTS value increases as well [70].
Another study by Wittbrodt and coworkers investigated the effects of different colors of PLA on the ultimate tensile strength. A Lulzbot TAZ 4 was used to 3D print samples in the following colors: natural, black, white, blue and silver at 190°C with 100% infill. Additionally, more white samples were 3D printed with varying temperature between 190°C and 215°C to study the effect of temperature. The result showed that natural color had the highest ultimate tensile strength and maximum strain with the lowest percent crystallinity, while gray color had the lowest ultimate tensile strength and maximum strain with higher percent crystallinity. Also, different temperature resulted in different percent crystallinity, for instance for white samples as the temperature increase from 190°C to 210°C the percent crystallinity increase; however, at 215°C is back down to a lower value, therefore, temperature can change the crystallinity rate. The result suggested some of the coloring agents may be acting as crystallization rate modifier, Therefore, color can significantly impact mechanical strength of 3D printed PLA material [71].

Infill density and infill pattern are other important factors that determine the strength of 3D printed objects. Tsoukindas and coworkers investigated the effect of infill density and infill pattern on mechanical property of PLA 3D printed specimens. MendelMAx 2 3D printer was used to 3D print the samples. The result showed as infill density increases the maximum impact force increases as well also octagonal infill pattern showed higher impact resistance for lower infill density in comparison to rectilinear and concentric filling. Therefore, infill density and infill pattern can determine strength of 3D printed materials and should be considered in 3D printing process [72].

2.5 Research Motivation

Previous literature showed that different process parameters in 3D printing have significant effect on the property of 3D printed PLA objects. However, there is no study on the effects of different process parameters on mechanical properties of 3D printed PCL that was printed using FDM
technology; therefore, there is a need for further research in this area because these properties will assist designers to better understand the properties of 3D-printed PCL. In order to accomplish our objective, the specific aims of the thesis are as follows:

Specific Aim 1: Optimize 3D printing parameters to fabricate a defect-free polycaprolactone tensile specimen.

Specific Aim 2: Study the impact of process parameters on mechanical properties of 3D printed polycaprolactone parts.
3.1 Abstract

Rapid prototyping is an innovative and powerful technology used for direct layer-by-layer fabrication of parts from a 3D computer model. Fused Deposition Modeling technology has attracted many researchers, especially those interested in its biomedical applications in custom-made implants, tissue and cell printing and the dental industry due to its price and availability. Polycaprolactone (PCL) is both biocompatible and biodegradable and generally used in fabrication of scaffolds in tissue engineering using FDM technology. Although there are reports on the impact of different process parameters on parts performance of PLA using FDM technology there is no study about the effect of process parameters on 3D printed PCL parts. Therefore, this manuscript reports the effect of nozzle temperature and volumetric flow rate for the fabrication of defect-free 3D-printed Polycaprolactone test specimens using FDM technology. The impact of process parameters such as infill density, layer height and the number of shell perimeters on the mechanical properties of PCL 3D-printed parts were investigated. Printrbot Simple Metal FDM 3D printer was used to fabricate tensile test specimens. Five specimens were produced for each experimental run, totaling 60 specimens. The results showed nozzle temperature of 165°C and volumetric flow rate of 2.97 mm³/s results in a defect free PCL 3D printed test specimen. Also, an infill density of 90%, layer height of 0.1 mm and shell perimeter of 2 resulted in the maximum tensile strength using the chosen optimization methods.
3.2 Introduction

Rapid prototyping is an innovative and powerful technology used for direct layer-by-layer fabrication of parts from a three-dimensional (3D) computer model [27]. Three-dimensional printed parts are fabricated based on an additive fabrication process using different techniques such as fused deposition modeling (FDM), stereolithography (SLA), selective laser sintering (SLS) and laminated object manufacturing (LOM) [8]. These techniques have been used in many fields such as electronics, aerospace, automotive and medicine. Fused deposition modeling developed by Stratasys in 1980s is the most commercialized and accessible technology [6]. Fused deposition modeling technology has attracted many researchers, especially those interested in its biomedical applications in custom-made implants, tissue and cell printing and the dental industry [46, 47, 67].

Fused deposition modeling uses a wide range of materials such as Polyester (Polylactic acid and Polycaprolactone), Polyamide, Polycarbonate, Polyethylene, Polypropylene and Acrylonitrile-Butadiene Styrene (ABS) whose melting points fall within the temperature range of the FDM printer’s nozzle which liquefy the material [6, 27]. However, in manufacturing implantable medical devices such as tissue engineering, material selection plays an important role because materials should be both biocompatible and biodegradable [73]. One of the commercially available modern implantable material that has been used for biomedical devices and implants is Polylactic-acid (PLA). Currently, limited materials are available that can be used by FDM technology that are biodegradable and biocompatible. PLA has been used widely in biomedical applications such as cell and tissue engineering, dental industry and prostheses. However, one of the limitations of 3D printing of medical devices is lack of available material. Polycaprolactone (PCL) is one of the material that meet the criteria for implantable material. The most common application of PCL in 3D printing is in fabricating scaffolds for tissue engineering [63]. PCL has a low transition temperature of −60°C and a melting temperature of 60°C [62] which makes it a good material for FDM technology.
Previous research on PLA has shown different parameters such as orientation [74], layer height [69], shell perimeters [70], colors [71] and infill density [72], have significant effect on the mechanical property of 3D-printed PLA. PLA has been used in biomedical application using FDM technology mostly in tissue engineering field such as studying the effects of primary bovine chondrocyte, primary articular chondrocytes and nucleus pulposus cells on PLA scaffold [12] or investigating the effect of PLA is used, in addition to PCL to fabricate new scaffolds [50]. Also, most of the reports on PCL are about tissue engineering applications such as evaluation of different types of PCL scaffolds for artificial bones [63], interactions between mesenchymal stem cells and PCL scaffold [66]. Although, there are reports on evaluations of 3D printed PCL scaffolds, however to the best of our knowledge there are no fundamental studies in the literature that investigates the effect of process parameters on the mechanical properties of RP fabricated PCL parts.

The present study reports the effect of nozzle temperature and volumetric flow rate to fabricate defect-free 3D printed Polycaprolactone tensile test specimens using FDM technology. The impact of process parameters (infill density, layer height and the number of shell perimeters) on the mechanical properties of PCL 3D printed parts was also investigated. The purpose is to assist designers in biomedical field to select the optimal parameters using PCL fabricated part by FDM technology.

3.3 Materials and Methods

To determine the mechanical properties of 3-D printed parts, all the specimens were produced in a Printrbot Simple Metal with a 1.75 mm nozzle diameter using 1.75 PCL filament from 3D4MAKER in natural color with the working temperature of 115 °C to 145 °C. Tensile specimens were designed according to ISO 527-2:1996 which was downloaded online, however, in order for the test specimen to have a good grip in the machine the length at both ends of the test specimen was increased by 10 mm. Cura software was used to generate G-code files and control the movement of the nozzle.
The tensile testing was performed by a Sintech 5/g test machine (figure 3.1) with crosshead speed of 0.05 in/min and a load cell of 20 kN. To calculate the strain, images of the test specimen were captured using Infinity camera from Lumenera while performing the test and exported into GOM 2D software for further analysis.

As there is no report on the optimal printing condition to fabricate defect-free test specimen using PCL such as nozzle temperature, volumetric flow rate, speed and bed temperature, three specimens per parameter were fabricated to investigate the effect of these parameters to fabricate a defect free specimen. In this study, volumetric flow rates can be simply calculated by multiplying nozzle diameter (width) with layer height, print speed and the flow rate percentage. Table 3.1 shows the summary of all the parameters that were investigated.
TABLE 3.1. Summary of process parameters that were investigated to obtain a defect free tensile test specimen.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nozzle temperature (°C)</td>
<td>115, 125, 135, 145, 155, 165, 175</td>
</tr>
<tr>
<td>Volumetric flow rate (mm$^3$/s)</td>
<td>2.20, 2.53, 2.75, 2.97, 3.19</td>
</tr>
<tr>
<td>Bed temperature (°C)</td>
<td>Room temperature (21) and 30</td>
</tr>
<tr>
<td>Print speed (mm/s)</td>
<td>55, 65, 75, 85</td>
</tr>
</tbody>
</table>

Also, five specimens per experimental runs and a total of 60 specimens were fabricated to investigate the effect of infill density, layer height and shell perimeters. Table 3.2 shows the different parameters used. Nozzle temperature, Volumetric flow rate, print speed, infill pattern and bed temperature remained constant while other parameters were changed.

Six infill density of 10%, 30%, 50%, 70%, 90% and 100% were investigated, whereas five layer heights ranged from 0.10 mm to 0.30 mm considered. Finally, three number of shell perimeters of 1, 2 and 3 were used to fabricate the test specimens. Each fabricated test specimen was held in place using the grips of the machine and loaded along the vertical axis until the necking was observed. Table 3.2. shows the summary of different process parameters used in this study.

TABLE 3.2. Different process parameters used to fabricate test specimen

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Infill density (%)</td>
<td>10, 30, 50, 70, 90, 100</td>
</tr>
<tr>
<td>Layer height (mm)</td>
<td>0.10, 0.15, 0.20, 0.25, 0.30</td>
</tr>
<tr>
<td>Shell perimeter</td>
<td>1, 2, 3</td>
</tr>
</tbody>
</table>
All the values presented in this paper are represented as the average of five measurement for each parameter with error bars represent ± the standard deviation. To analyze the data and determine which is statistically significant, analysis was performed using Minitab (Fisher LSD method) to analyze mean values using significant level (α-level) of 0.05 and confidence interval of 95%.

3.4 Results and Discussion

As mentioned, there is no study in optimal printing condition for PCL material, therefore print parameters such as bed temperature, print speed, nozzle temperature and volumetric flow rate were evaluated.

Since nozzle temperature was viewed as a parameter that had the most impact it was investigated first as compared to other parameters. Test specimens were created with the nozzle temperature within the range of 115–175 °C. The lower temperature of 115 °C was selected based on recommended temperature settings provided by the PCL filament manufacturer. As reported in the literature nozzle temperature has a significant impact on the physical characteristics of the 3D-printed parts [71] hence various temperatures up to 175 °C were evaluated to obtain a defect free tensile specimen. Default values of other parameters (print speed: 55 mm/sec, volumetric flow rate 2.2 mm³ / sec and bed temperature: room temperature at 21 °C) were used for initial investigation of effect of nozzle temperature.

A Nozzle temperature of 115 °C resulted in a deformed and defective print and at 125 °C (figure 3.2), there is stringing with more holes on the surface. At temperatures between 165 °C (figure 3.3) and 175 °C (figure 3.4), there was a reduction in the number of holes on the surface, as well as stringing. To determine why there was a deviance between the manufacturer recommended temperature and the optimal printing condition obtained by this study, an infrared camera was used to determine the actual temperature of the hot end at various temperatures. The results showed that at stated nozzle temperature
of 115 °C the actual temperature of the nozzle (determined by infrared thermal camera) was reached approximately 96.8 °C. Thus, the actual temperature was way below the recommended nozzle operating temperature for PCL. Similarly, at stated temperature of 165 °C the actual nozzle reached approximately 140.4 °C (figure 3.5).

FIGURE 3.2. Visual picture of a representative specimen fabricated at temperature 125 °C: (a) front view and (b) back view

FIGURE 3.3. Visual picture of a representative specimen fabricated at temperature 165 °C: (a) front view and (b) back view
FIGURE 3.4. Visual picture of a representative specimen fabricated at temperature 175 °C: (a) front view and (b) back view

FIGURE 3.5. Representative image using IR camera indicates different in nozzle temperature (NZ) and actual temperature (AT)

Visual appearance was used to determine the operating temperature of PCL. Qualitative determination of samples (visually observed) was carried to ensure presence of minimal voids and stringing. Since increasing temperature beyond 165 °C did not eliminate presence of voids and didn’t produce a better specimen (determined visually, figure 3.3 – 3.4) it was determined that a different parameter was to be investigated to further refine the 3D printed part. Also, since the defects (voids) on the test specimens were spread out randomly on repeat runs, therefore, quantitative comparison of mechanical testing at this stage was determined to be rudimentary. It was thought that there would not be a consistent failure mechanism (due to random voids) within different runs of the same setting hence mechanical testing was deferred until a defect free test specimen was obtained.
Since the appearance of voids (holes) on the surface indicates a lack of material on the surface; therefore, the volumetric flow rate was investigated for potential reduction of voids on the test sample. With the nozzle temperature remaining constant at 165 °C, different volumetric flow rates were investigated (2.2 – 3.19 mm$^3$/s, table 3.1) to reduce the number of holes on the surface. The number of holes on the surface decreased as the volumetric flow rate was increased from 2.2 mm$^3$/s. At volumetric flow rates of 3.19 mm$^3$/s (figure 3.6) stringing and inconsistency of the material around the edges of the specimen occurred due to overflow/excess of material. At the lower volumetric flow rate of 2.2 mm$^3$/s (figure 3.7) printed specimen showed significant number of holes on the surface. For the volumetric flow rates studied it was found that at 2.97 mm$^3$/s volumetric flow rate (figure 3.8), the specimens seem to be defect free without any inconsistency or stringing (determined by visual observation). Figure 3.9 shows the specimen printed at 165 °C and volumetric flow rate of 2.97 mm$^3$/s which is devoid of stringing and surface voids. These conditions were subsequently used for further investigation.

Investigations into effect of bed temperature (room temperature: 21 °C and 30 °C) showed no difference in print quality of the tensile specimens. Similarly, effect of print speed (55,65,75,85 mm/s) showed no difference in print quality of the tensile specimens. Hence, default (based on initial setting of the printer software) was used for subsequent studies (Specific aim 2).
FIGURE 3.6. Visual picture of a representative specimen fabricated at volumetric flow rate of 3.19 mm$^3$/s: (a) front view and (b) back view

FIGURE 3.7. Visual picture of a representative specimen fabricated at volumetric flow rate of 2.2 mm$^3$/s: (a) front view and (b) back view
FIGURE 3.8. Visual picture of a representative specimen fabricated at volumetric flow rate of 2.97 mm$^3$/s: (a) front view and (b) back view

FIGURE 3.9. Visual picture of a representative specimen fabricated at optimized parameter: (a) front view and (b) back view

Since defect free specimens were successfully fabricated by 3D printing, therefore, process parameters such as infill density, layer height and shell perimeters were investigated and their impact on mechanical properties evaluated. Various infill density levels ranging from 10% to 100% (while keeping
other parameters constant and layer height and number of shell perimeter at their default value at 0.1 mm and 2 respectively) were evaluated to obtain the optimal infill density. Infill density was first selected because it has been found to be the most sensitive to change and have the most significant impact on the mechanical performance of 3D printed parts using FDM [6]. Typical stress-strain curve obtained (figure 3.10) and ultimate tensile strength calculated based on the highest stress point in which necking was observed because at that point the part already lost its structural integrity and cannot be used. The results (figure 3.11 and table 3.3) showed 10% (14.286 ± 0.43 MPa) leads to the lowest while 100% (16.217 ± 0.433 MPa) leads to the highest ultimate tensile strength with a positive trend from 10% to 100%. The overall mechanical properties trend increased with increase in infill density which is in agreement with reported literature on PLA filament that indicates at higher infill density the amount of material inside the print is higher which lead to stronger parts [72]. Figure 3.11 and table 3.3 shows the summary of the results. However, to find out which parameter will be the preferred infill density, statistical analysis was carried out using the Fisher LSD method (figure 3.12). The result showed that three infill densities of (100%, 90%, 70%) and two infill densities of (50% and 70%) respectively were not statistically significant, however five infill density of (10%, 30%, 50%, 90% and 100%) were statistically significant. In order to find the preferred infill density, the 70%, 90% and 100% data was analyzed, and the 70% infill density was discarded as it showed statistically insignificance between the 50% infill density data. The 90% was selected because it showed significant deviance from the 50% data (statistically insignificant from 70% data) and properties similar (no statistically difference) to 100% infill density. Therefore 90% infill density was selected as the preferred condition rather than 100% as lesser material would be used using 90% infill density with similar properties being obtained.
FIGURE 3.10. Stress-strain curves for different infill density values

FIGURE 3.11. Ultimate tensile strength for different infill density values
TABLE 3.3. Summary of statistical analysis for different infill density values

<table>
<thead>
<tr>
<th>Infill Density (%)</th>
<th>Number of Samples</th>
<th>Mean (MPa)</th>
<th>SE</th>
<th>StDev</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.00</td>
<td>5</td>
<td>14.286</td>
<td>0.193</td>
<td>0.43</td>
</tr>
<tr>
<td>30.00</td>
<td>5</td>
<td>14.773</td>
<td>0.0492</td>
<td>0.11</td>
</tr>
<tr>
<td>50.00</td>
<td>5</td>
<td>15.53</td>
<td>0.0735</td>
<td>0.164</td>
</tr>
<tr>
<td>70.00</td>
<td>5</td>
<td>15.902</td>
<td>0.06395</td>
<td>0.143</td>
</tr>
<tr>
<td>90.00</td>
<td>5</td>
<td>16.086</td>
<td>0.11</td>
<td>0.247</td>
</tr>
<tr>
<td>100.00</td>
<td>5</td>
<td>16.217</td>
<td>0.194</td>
<td>0.433</td>
</tr>
</tbody>
</table>

FIGURE 3.12. Statistical analysis on ultimate tensile strength of PCL test specimen with different infill density values

With infill density remaining constant at 90% and number of shell perimeter remaining constant at default value of 2, further variations of another printing parameters (layer thickness) were evaluated from 0.1 mm to 0.3 mm. Stress-strain curves at varying layer thickness were obtained (figure 3.13) and ultimate tensile strengths determined (figure 3.14). Specimen printed at 0.1 mm layer height had the highest tensile strength (16.0806 ± 0.247 MPa) while specimen printed at 0.3 mm resulted in the lowest tensile strength (12.889 ± 0.164 MPa, table 3.4). The overall mechanical properties trend decreased with increase in layer height (figure 3.14 and table 3.4) most probably, because the higher layer height results
in a lower amount of interface between PCL filament which is consistent with previous studies on PLA that showed lower layer height resulted in higher tensile strength [69]. In order to determine the preferred layer height, the data ranging from 0.1 mm to 0.30 mm were analyzed using the Fisher LSD method and result suggested that 0.1 mm layer height was significant compared to the others, therefore, layer height of 0.1 mm was selected as the preferred value (figure 3.15).
FIGURE 3.13. Stress-strain curve for different layer height values

FIGURE 3.14. Ultimate tensile strength for different layer height values
TABLE 3.4. Summary of statistical analysis for different layer heights values

<table>
<thead>
<tr>
<th>Layer Heights (mm)</th>
<th>Number of Samples</th>
<th>Mean (MPa)</th>
<th>SE</th>
<th>StDev</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>5</td>
<td>16.086</td>
<td>0.11</td>
<td>0.247</td>
</tr>
<tr>
<td>0.15</td>
<td>5</td>
<td>12.784</td>
<td>0.0842</td>
<td>0.188</td>
</tr>
<tr>
<td>0.2</td>
<td>5</td>
<td>13.055</td>
<td>0.0303</td>
<td>0.0677</td>
</tr>
<tr>
<td>0.25</td>
<td>5</td>
<td>13.05</td>
<td>0.0279</td>
<td>0.0624</td>
</tr>
<tr>
<td>0.3</td>
<td>5</td>
<td>12.889</td>
<td>0.0731</td>
<td>0.164</td>
</tr>
</tbody>
</table>

FIGURE 3.15. Statistical analysis on ultimate tensile strength of PCL test specimen with different layer height values

While preferred infill density and layer height were determined, the next parameter which is number of shell perimeters such as 1, 2 and 3 were investigated. Stress-strain curves shown in Figure 3.16 and ultimate tensile strength (figure 3.17) were evaluated. The result showed two number of shell perimeters produced the strongest test specimen with tensile strength of 16.086 ± 0.247 MPa. Statistical analysis using the Fisher LSD method (table 3.5 and figure 3.18) showed that UTS from shell 1 (14.410 ± 0.267 MPa) and shell 3 (14.882 ± 0.213 MPa) were lower than the UTS obtained from shell 2 (16.086 ± 0.247 MPa). Therefore, two number of shell perimeters was selected as the preferred value.
FIGURE 3.16. Stress-strain curves for different shell perimeter values

FIGURE 3.17. Ultimate tensile strength for different shell perimeter values
### TABLE 3.5. Summary of statistical analysis for different number of shell perimeter values

<table>
<thead>
<tr>
<th>No. of Shell Perimeters</th>
<th>Number of Samples</th>
<th>Mean (MPa)</th>
<th>SE</th>
<th>StDev</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>14.41</td>
<td>0.12</td>
<td>0.267</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>16.086</td>
<td>0.11</td>
<td>0.247</td>
</tr>
<tr>
<td>3</td>
<td>5</td>
<td>14.882</td>
<td>0.095</td>
<td>0.213</td>
</tr>
</tbody>
</table>

FIGURE 3.18. Statistical analysis on ultimate tensile strength of PCL test specimen with different shell perimeter values

3.5 Summary and Conclusions

The effect of printing conditions on PCL test specimens (made using Printrbot simple metal FDM 3D printer) was characterized using tensile tests. The data showed that nozzle temperature of 165 °C and volumetric flow rate of 2.97 mm³/s as conditions that resulted in defect free 3D printed PCL specimens. Other print parameters such as bed temperature and print speed in the chosen range investigated showed no effect on print quality. With above printing condition remaining constant (nozzle temperature: 165 °C, volumetric flow rate: 2.97 mm³/s, bed temperature: 21 °C and print speed: 55 mm/s) various process parameters such as infill density, layer height, and shell perimeters were investigated to evaluate their
effect on mechanical properties. Results showed infill density of 90%, layer height of 0.1 mm and shell perimeter of 2 produced the highest UTS. The results indicated that the print parameters (infill density, layer height, and shell perimeters) have an impact on part quality and will need optimization as new materials are being developed for FDM 3D printing.
CHAPTER 4: CONCLUSIONS AND FUTURE STUDY

FDM technology offers several advantages for part fabrication as opposed to traditional fabrication methods. This technology also shows great potential to be used in biomedical applications using biocompatible materials. In this study, we have demonstrated how print and process parameters affect the final part quality and property. The data showed that nozzle temperature of 165 °C and volumetric flow rate of 2.97 mm³/s as conditions that resulted in defect free 3D printed PCL specimens. Other print parameters such as bed temperature and print speed in the chosen range investigated showed no effect on print quality. With above printing condition remaining constant (nozzle temperature: 165 °C, volumetric flow rate: 2.97 mm³/s, bed temperature: 21°C and print speed: 55 mm/s) various process parameters such as infill density, layer height, and shell perimeters were investigated to evaluate their effect on mechanical properties. Results showed infill density of 90%, layer height of 0.1 mm and shell perimeter of 2 produced the highest UTS. The results indicated that the print parameters (infill density, layer height, and shell perimeters) have an impact on part quality and will need optimization as new materials are being developed for FDM 3D printing.

Although, results provided insight on how properties of PCL varied with different process parameters further improvement and variability of properties could be obtained by fabricating PCL based composites for 3D printing. Biodegradable and biocompatible materials such as Magnesium (Mg) and Polymers can perform their functions and gradually degrade. This feature has reduced the risk of long-term complications in patients. However, both magnesium and polymers have their limitations. Polymers such as PCL have low tensile strength starting from 10 MPa which make them susceptible to failure while magnesium alloys have tensile strength starting from 160 MPa [75] with high degradation rate. Although to control the degradation of magnesium, polymer coatings have been studied but no reports were found on a viable Mg based system that could be 3D printed. To the best of our knowledge, there
have been no studies regarding designing and developing hybrid PCL filament for rapid prototyping with other biodegradable metals such as magnesium. This could offer the potential to vary the tensile strength of PCL while maintaining the viability to 3D print. Therefore, the fabrication of PCL/Mg filament and evaluation of the effect of different process parameters on part properties merits scientific investigation.
REFERENCES
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*3D PRINTING POLYCARBONATE FILAMENT*


