

# EFFECTS OF HUMIDITY ON MECHANICAL PROPERTIES AND MICROSTRUCTURE OF 3D PRINTED PLA FILAMENT

This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)



## FATIMA HANIM BINTI HAMEZAH B051710197 980117-11-5466

FACULTY OF MANUFACTURING ENGINEERING 2021



# UNIVERSITI TEKNIKAL MALAYSIA MELAKA

#### BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

Tajuk: EFFECTS OF HUMIDITY ON MECHANICAL PROPERTIES AND MICROSTRUCTURE OF 3D PRINTED PLA FILAMENT

Sesi Pengajian: 2020/2021 Semester 2

#### Saya FATIMA HANIM BINTI HAMEZAH (980117-11-5466)

mengaku membenarkan Laporan Projek Sarjana Muda (PSM) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

- 1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
- 2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.

اونيوم سيتي تنكنيكل مليسيا ملاك

3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.

1	*C:10	tandakan	(1)	1
4	*8118	tandakan	(V	)

SULIT / E (Mengandungi makl Malaysiasebagaiman	umat yang berdarjah keselamatan atau kepentingan na yang termaktub dalam AKTA RAHSIA RASMI 1972
TERHAD (Mengandungi makl badan di mana peny	lumat TERHAD yang telah ditentukan oleh organisasi/ elidikan dijalankan)
/ TIDAK TERHAD	
	Disahkan oleh:
	Jane -
lamat Tetap:	Cop Rasmi: DR. RAHIMAH BINTI HJ. ABDUI. HAMID Senior Lecturer Faculty of Manufacturing Engineering

Universiti Teknikal Malaysia Melaka

Hang Tuah Jaya 76100 Durian Tunggal, Melaka

A-22, Kg Tepus, Bukit Besi, 23200 Dungun, Terengganu.

Tarikh: 2 September 2021 Tarikh: 2 September 2021

\*Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

## **DECLARATION**

I hereby, declared this report entitled "Effects of Humidity on Mechanical Properties and Microstructure of 3D Printed PLA Filament" is the result of my own research except as cited in references.

Signature

Author's Name : FATIMA HANIM BINTI HAMEZAH

Date : 2 September 2021

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:



### **ABSTRAK**

Filamen asid polilaktik (PLA) sensitif terhadap kelembapan kerana sifat higroskopiknya menyebabkan gelembung dan kualiti cetakan yang kurang baik. Ramai pengguna Fused Deposition Modelling (FDM) mengabaikan kawalan kelembapan dan cara penyimpanan filamen yang betul selepas digunakan. Banyak kajian memberi tumpuan kepada kesan parameter proses pada bahagian cetakan 3D; tetapi pengaruh kelembapan pada kualiti percetakan kurang diterokai. Kelembapan boleh mempengaruhi sifat-sifat mekanikal bahagian cetakan 3D kerana kewujudan air boleh mengubah ikatan rantai polimer justeru melemahkan kekuatan mekanikal. Objektif kajian ini adalah untuk mengkajit pengaruh kelembapan pada kekuatan tegangan dan lenturan serta perubahan mikrostruktur. Oleh itu, tiga tetapan bersyarat telah dihasilkan; menggunakan gulungan PLA baru sebagai rujukan, gulungan PLA yang disimpan dalam beg vakum bersama agen pengering, dan gulungan PLA yang disimpan dalam persekitaran terbuka, terdedah kepada kelembapan selama 24 - 150 jam. Filamen ini digunakan untuk menghasilkan spesimen yang mematuhi ASTM D638 -Jenis IV (spesimen tegangan) dan ASTM D790 (spesimen lenturan) untuk diuji pada Mesin Ujian Universal Shimadzu AGS-X (UTM) pada kelajuan 5mm/s, dan 50 mm/s. Spesimen tegangan yang telah patah kemudiannya disalut dan digunakan pada mesin Scaning Electron Microscope (SEM) untuk mengkaji mikrostrukturnya. Keputusan menunjukkan bahawa kelembapan mengurangkan kekuatan tegangan dan lenturan sambil meningkatkan keupayaan untuk patah. Kehadiran kelembapan memplastikkan polimer, memudahkannya untuk dibentuk dan mengurangkan ketegarannya kerana kelembapan mengubah struktur molekul polimer. Imej SEM menunjukkan bahawa kelembapan memperluaskan jurang interlayer, menggalakkan peningkatan dalam keupayaan untuk patah disebabkan oleh peningkatan ruang pengubahsuaian makromolekul. Namun, kami mendapati bahawa filamen terpakai yang disimpan dalam beg vakum bersama beberapa agen pengering menunjukkan kekuatan tegangan yang setara dengan spesimen rujukan. Kesimpulannya, kelembapan mempengaruhi sifat-sifat mekanikal dan harus dikawal untuk kualiti percetakan bahagian PLA yang baik. Sebagai cadangan, semua pengguna percetakan 3D dinasihatkan untuk menyimpan filamen PLA mereka dalam beg vakum bersama agen pengering untuk menggantikan kabinet pengeringan bagi mengawal kelembapan dalam percetakan.

#### **ABSTRACT**

Polylactic acid (PLA) filament is sensitive to humidity due to its hygroscopic properties, resulting in bubbles and poor printing quality of 3D printed parts. Even though it is significant to control the PLA filament's humidity and store it properly after use, many Fused Deposition Modelling (FDM) users ignore this factor. Many studies focus on the effect of process parameters on the 3D printed part; however, the influence of humidity on the printing quality is less explored. Moisture can influence the mechanical properties of 3D printed parts due to the water's existence, which could alter the polymer chain's bonding, resulting in a lower mechanical strength. The objectives of this study are to investigate the influence of humidity on the tensile strength, fracture strength, and the alteration of microstructure caused by moisture. In order to do so, three conditional settings were established; a new PLA roll acts as the reference, used PLA roll stored in the vacuum bag with desiccant, and used PLA roll stored in an open environment, exposed with the humidifier for 24 - 150 hours. These filaments are subsequently used to fabricate specimens that comply with ASTM D638 - Type IV (tensile specimen) and ASTM D790 (flexural specimen) to be tested in the Shimadzu AGS-X Universal Testing Machine (UTM) at the speed of 5mm/s, and 50 mm/s, respectively. The fractured tensile specimen is then sputtercoated and used in the Scanning Electron Microscope (SEM) machine to study its microstructure. The results show that the humidity decreases the tensile and flexural strength while increasing the fracture strain. The presence of moisture causes the polymer to plasticize, increasing its deformability and reducing its rigidity as the moisture alters its molecular structure. The SEM images show that humidity expands the interlayer gap, promoting enhancement in the fracture strain due to increased macromolecules deformation space. Nevertheless, we found that the used PLA filament stored in a vacuum bag with some desiccants shows an equivalent tensile strength with the reference specimen. To conclude, humidity influences the mechanical properties and should be controlled for a good printing quality of PLA parts. As a recommendation, all 3D printing users are advised to store their PLA filament in a vacuum bag with dehumidifying agents to substitute for the drying cabinet to control the humidity in their printing.

# **DEDICATION**

This humble work is dedicated to my beloved father, mother and siblings.



#### **ACKNOWLEDGEMENT**

Praises and thanks to Allah, the most gracious and the most merciful, for His countless of blessings throughout my journey to complete this final year project successfully.

I would like to express my deep and sincere gratitude to my supervisor, Dr. Rahimah Binti Hj. Abdul Hamid. Thank you so much in every point of this research for the precious time, advice, feedback, suggestions, and guidance. It was a great opportunity and pleasure to work and study under her guidance.

Besides, a special thank you goes to my beloved parents and siblings for their encouragement, support and prayers for the success of the project. I would also like to thank all my friends, especially my classmates and roommates for their infinite support.

Last but not least, my appreciation goes to those who have helped me directly and indirectly in completing this report. I am very thankful that lot of people have support and inspire me to carry out this project.

## **TABLE OF CONTENTS**

Abst	rak		i
Abst	ract		ii
Dedi	cation		iii
Ackr	nowledge	ement	iv
Table	e of Con	tents	v
List	of Tables	3	vii
List	of Figure	es	ix
List	of Abbre	viations	xi
List o	of Equati	ions	xii
	of Symbo		xiv
		1: INTRODUCTION	
1.1		ground	1
1.2		em Statement	3
1.3 1.4	Objec Scope	5 Mali 16 16 - 16 - 16 - 16 - 16 - 16 -	4
1.5	Signif	ficance of Study	5
1.6	Organ	nization of Report	5
CHA	APTER 2	2: LITERATURE REVIEW	
2.1	Overv	view of 3D Printing	7
2.2	Fused	Deposition Modelling (FDM)	8
	2.2.1	Process parameters	9
	2.2.2	Materials	10
2.3	Polyla	actic Acid (PLA)	12
	2.3.1	Production of PLA	13
	2.3.2	Properties of PLA	15
2.4	Humi	dity	18
	2.4.1	Measurement of relative humidity	18
		2.4.1.1 Wet and dry bulb hygrometer	18

	2.4.1.2 Hair hygrometer	
	2.4.1.3 Regnault's hygrometer	
	2.4.1.4 Electronic hygrometer	
	2.4.2 Measurement of absolute humidity	
	2.4.2.1 Mass spectrometry	
2.5	Humidity Effects on 3D Printing	
2.6	Tensile Test	
2.7	Flexural Test	
2.8	Scanning Electron Microscopy	
2.9	Sputter-coating	
2.8	Summary	
CHA	PTER 3: METHODOLOGY	
3.1	Process Flow of the Study	
3.2	Relationship between Objective and Methodology	
3.3	Flow Chart of Methodology	
3.4	Preparation of PLA Filaments	
3.5	CAD Model	
3.6	3D Printing	
	3.6.1 Setting parameters	
	3.6.2 Number of specimens	
3.7	Tensile Test	
3.8	Flexural Test	
3.9	Sputter-coating	
3.10	Scanning Electron Microscopy (SEM) Analysis	
CHA	PTER 4: RESULT AND DISCUSSION	
4.1	Tensile Strength Analysis	
4.2	Flexural Strength Analysis	
4.3	Stress-strain Analysis	
	4.3.1 Tensile-stress-strain Analysis	
	4.3.2 Flexural-stress-strain Analysis	
4.4	Microstructure Analysis	

## **CHAPTER 5: CONCLUSION AND RECOMMENDATION**

5.1	Conclusion	54
5.2	Recommendation	55
5.3	Sustainability Element	55
5.4	Lifelong Learning Element	56
5.5	Complexity Element	56

#### **REFERENCES**

### **APPENDICES**

A	Gantt Chart of FYP 1	64
В	Gantt Chart of FYP 2	65



## LIST OF TABLES

2.1	Physical and chemical properties of PLA	
2.2	Physical properties of PLA	17
3.1	Implications of methodology used in the study	30
3.2	Setting of process parameters for 3D printing	36
3.3	The type of PLA filaments and number of specimens printed for the study	37
4.1	The average maximum force and maximum stress results for tensile test	43
4.2	The average maximum force and maximum stress results for flexural test	46
4.3	The findings of ImageJ software for the length of the interlayer gap	53
	UNIVERSITI TEKNIKAL MALAYSIA MELAKA	

## LIST OF FIGURES

1.1	The cause-and-effect diagram of FDM process parameters	2
2.1	Process parameters of FDM	9
2.2	The results for the compared thermoplastic materials	10
2.3	PLA structure	12
2.4	Production of lactic acid from renewable source	12
2.5	Optical isomers of lactic acid	13
2.6	Polymerization steps of PLA	14
2.7	Wet and dry bulb hygrometer	19
2.8	Hair hygrometer	19
2.9	Regnault's hygrometer	20
2.10	Electronic hygrometer	20
2.11	Mass spectrometry	21
2.12	Tensile test setup	23
2.13	Flexural test setup	25
2.14	The microstructure of 3D printed PLA	26
2.15	Fracture surfaces of dog bones produced using ULTEM®9085 filaments	26
3.1	Flowchart of the study	29
3.2	Flow chart of methodology used to achieve stated objectives	31
3.3	PLA filaments	32
3.4	Dimension of tensile specimen based on the ASTM-D638 (Type IV) standard	33
3.5	Dimension of flexural specimen based on the ASTM-D790 standard	33
3.6	CAD model of ASTM-D638 (Type IV) standard	34
3.7	CAD model of ASTM-D790 standard	34
3.8	FlashForge's Adventurer3 3D Printer	35
3.9	3D printing setup	35
3.10	3D printed specimens	37

3.11	Tensile test setup	38
3.12	Flexural test setup	39
3.13	Example of a specimen dimension	39
3.14	The difference in SEM images for (a) before sputter-coating and (b) after	40
	sputter-coating	
3.15	SC7620 Mini Sputter Coater machine	41
4.1	Tensile maximum force (N) plot for all conditions	44
4.2	Flexural maximum force (N) plot for all conditions	48
4.3	Stress-strain curve for tensile test	50
4.4	Stress-strain curve for flexural test	51
4.5	The measured interlayer gap of the 3D printed objects using new PLA	52
	filament (reference)	
4.6	The measured interlayer gap of the 3D printed objects using used PLA	52
	filament stored in vacuum bag with addition of desiccant	
4.7	The measured interlayer gap of the 3D printed objects using PLA	53
	filament humidified for 150 hours	
	اونيوسيتي تيكنيكل مليسيا ملاك	
	UNIVERSITI TEKNIKAL MALAYSIA MELAKA	

#### LIST OF ABBREVIATIONS

3D - Three dimensional

ABS - Acrylonitrile butadiene styrene

AM - Additive manufacturing

ASTM - American society for testing and materials

CAD - Computer-aided design

CAM - Computer-aided manufacturing

C-O - Carbon-Oxygen

FDM - Fused Deposition Modelling

FTIR - Fourier transform infrared

HIPS - High-intensity polystyrene

ISO International Organization for Standardization

Mw Molecular weight

N-H Nitrogen-Hydrogen

PC - Polycarbonate

PDLA Poly (D-lactic acid)

PDLLA - Amorphous poly (D, L-lactic acid)

PE UNIVERSIT Percentage of error ALAYSIA MELAKA

PEEK - Polyether ether ketone

PEI - Polyetherimide

PET - Polyethylene terephthalate

PETG - Polyethylene terephthalate glycol

PLA - Polylactic acid

PLLA - Poly (L-lactic acid)

PPSF - Polyphenylsulfone

PSM - Projek Sarjana Muda

Pt - Platinum

ROP - Ring opening polymerization

RP - Rapid prototyping

SEM - Scanning electron microscope

SLA - Stereolithography

STL - Standard triangle language

Tg - Glass transition temperature

THF - Tetrahydrofuran

Tm - Melting temperature

TPU - Thermoplastic polyurethane

UFM - Universal tensile machine

UFS - Ultimate flexural strength

UTS - Ultimate tensile strength



# LIST OF EQUATIONS

3.1	Average	
4.1	Normal stress	49
42	Strain	10



## LIST OF SYMBOLS

% - Percent

° - Degree

°C - Degree Celsius

cm<sup>-1</sup> - Centimetre power <sup>-1</sup>

dl/g - Decilitre per gram

g/cm<sup>3</sup> - Gram per centimetre cube

 $g/m^3$  Gram per metre cube

g/ml - Gram per millilitre

g/mol - Gram per molecule

J/g - Joule per gram

K Kelvin

Kg Kilogram

kJ/mol Kilo joule per molecule

kN - Kilonewton

lbs Pounds

mg/l - Milligram per metre

mm U-NIVERSIT Millimetre (AL MALAYSIA MELAKA

mm/min - Millimetre per minute

mm/s - Millimetre per second

N - Newton

N/mm<sup>2</sup> - Newton per millimetre square

nm - Nano metre

∠H°m - Enthalpy

μm - Micron metre

# CHAPTER 1 INTRODUCTION

This chapter describes the introduction of this research work, including the background, problem statement, objective, scope, and significance of the study. A study on the effect of humidity on the mechanical properties and microstructure of 3D printed PLA filament is carried out in this report.

#### 1.1 Background

Additive Manufacturing (AM) or 3D printing is a modern manufacturing technology that has experienced massive growth over the last few years. 3D printing allows physical models and complex geometric structures to be produced with high precision and low cost. Due to the personalized and straightforward manufacturing of functional models, conceptual models, and prototypes, 3D printing has been hugely embraced by the military, automotive, medical, food industry, aircraft, and other allied industries (Mohamed, Masood & Bhowmik, 2015).

According to Zhang, Fan, and Liu (2020), Fused Deposition Modelling (FDM) is one of the mainly utilized AM processes because of its ease and availability of machinery with reasonable costs that employs thermoplastic materials as hot melt adhesive properties. Kwon *et al.* (2020) declared that FDM is among the most prevalent processes owing to its adaptability and low expense. Three-dimensional structures that integrate the primary material in layers via the heated nozzle of the FDM 3D printer are produced in the FDM process. The nozzle's horizontal and vertical directions are entirely funded by the software called computer-aided manufacturing (CAM).

Filaments from thermoplastics such as polycarbonate (PC), acrylonitrile butadiene styrene (ABS), and polylactic acid (PLA) are typically used as the raw material instead of metals in this technology because of their low melting point and its ability to melt continuously under heat and to be re-solidified after the heat is removed (Wang *et al.*, 2017). Consequently, this helps the FDM printer to produce a solid product after the semi-liquid extrusion of the raw material.

Different variables determine the properties of the filament's materials. Humidity is one of the key factors influencing the filament's properties, as shown in Figure 1. For example, if the polylactic acid (PLA) filament has been subjected to heavy moisture for an extended period, the filament has lost tensile strength at the time of printing which allows bubbles to emerge on the surface of the product (Valerga *et al.*, 2018). Additionally, PLA is susceptible to humidity, and changes in its properties are caused by exposure to high humidity. As humidity increases, it has been recorded that the elongation ratio, degradation rate, and average molecular weight decrease (Liu *et al.*, 2017).

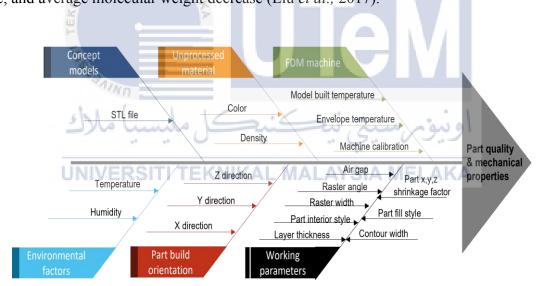


Figure 1.1: The cause-and-effect diagram of FDM process parameters (Mohamed, Masood & Bhowmik, 2015)

In this study, the effect of humidity on the mechanical properties, which includes tensile strength and flexural strength, and microstructure of the PLA printed parts exposed to various humidity environments was investigated.

#### 1.2 Problem Statements

Polyesters are widely recognized for absorbing humidity, and the presence of moisture causes severe degradation events. The degradation is mainly caused by the hydrolysis of macromolecular chains, which significantly reduces molecular weight (Titone *et al.*, 2021). According to Mallick *et al.* (2018), polylactic acid (PLA) or polylactide is a biologically degradable and bioactive polyester consisting of building blocks of lactic acid. The filaments of PLA are made of fermented plant starch such as maize, sugarcane, sugar beet pulp, and cassava, making them distinct from other thermoplastics. Lesaffre *et al.* (2017) suggested that PLA is sensitive to such environmental restrictions. Exposure to different natural weather factors such as sunlight, rainfall, and humidity, typically contributes to the aging of the substance, which will affect both the physico-chemical and fire-resistant properties of the material.

Filaments made of PLA possess hygroscopic characteristics that appear to absorb moisture from the air, which contributes to inconsistent and poor printing quality (Dwamena, 2020). Suppose the filaments are stored in a humid environment. In that case, the water vapor tends to be absorbed, and steam is generated during the heating process, resulting in an imperfect product surface at the end of the process. In addition, this will lead to a generation of bubbles on the surface of the printed part. Valerga et al. (2018) stated that the filament had reduced tensile strength if the PLA filament is exposed to high humidity for an extended period. Therefore, the filaments must be kept sealed in a vacuum so that the filaments are still in a dry condition to ensure good printing quality. A study on the effect of humidity on the mechanical properties and microstructure of the 3D printed PLA specimens still lacks, which encouraged this study to be carried out. On the other hand, dehumidifying agents like silica gel or desiccant might be able to control the moisture content in the PLA filament. However, it is unknown to what extent it works and how it influences the PLA specimen's mechanical properties compared to the new PLA filament. Therefore, in this study, an attempt to investigate the influence of using these items on the mechanical properties of the PLA specimen is also conducted.

#### 1.3 Objectives of Study

The objectives of this study are:

- (a) To analyze the tensile strength of the 3D specimens fabricated using the non-exposed and moisture-exposed PLA filaments.
- (b) To examine the flexural strength of the 3D parts produced from the non-exposed and moisture-exposed PLA filaments.
- (c) To investigate the microstructure of the fractured 3D printed tensile samples using the Scanning Electron Microscope (SEM).

#### 1.4 Scope of Study

The scope of this study are as follows:



(a) In this study, a 1.75 mm diameter of the PLA filament (light blue) was used for all conditions.

- (b) The humidity level was decided through three conditions as follows:
  - i. New PLA filament roll, which acts as the reference.
  - ii. Used PLA filament roll stored in the vacuum bag, with the desiccant.
  - iii. Used PLA filament roll stored in an open environment, exposed to a humidifier for variant of 24, 48, 72, 96, 120, and 150 hours.
- (c) The printing parameters for all conditions were similar, with three replications of each conditional setting.
- (d) The FDM machine, FlashForge's Adventurer3, was used to print the samples.

- (e) The analysis of the tensile strength (ASTM D638 Type IV) and flexural test (ASTM D790 of the FDM printed parts were carried out to investigate the influence of humidity on the quality of printing.
- (f) The fractured tensile samples which have the closest maximum force value to the average value were used for the analysis of the microstructure using the scanning electron microscope (SEM) due to the limitation of laboratory usage during the Covid-19 pandemic outbreak.
- (g) The samples used for SEM analysis were cut into 10 mm and then sputter-coated with 10nm thick of gold-palladium using SC 7620 Mini Sputter Coater machine.

#### 1.5 Significance of Study

This study helps to analyse and examine the effect of moisture on PLA mechanical properties and microstructure. If the humidity is found to be affecting the quality and properties of the printed parts, a clear message to emphasize the importance of controlling this factor could be highlighted. Thus, it can improve the printing quality of parts produced from PLA and reduce the PLA waste. The reduction of 3D printing waste can promote sustainable 3D printing since the number of 3D printer users is increasing due to its affordability. In addition, the mechanical properties of the PLA filament, which is stored with desiccant in a vacuum bag, are found to be equivalent to the new PLA filament. This finding shows that the use of dehumidifying agents is sufficient to keep the humidity away from the used PLA filament to substitute drying rack, which is too costly for hobbyists. Therefore, it is hoped that this study could prove the effect of humidity on the printing quality of parts and simultaneously create awareness for all 3D printer users.

#### 1.6 Organization of Report

In this study, there are five chapters consisting of introduction, literature review, methodology, result and discussion, conclusion and recommendation. Chapter 1 began with a study background, problem statement, objectives, and scope of the study. Chapter 2

comprised of previous study or research about the FDM process, PLA filaments, the influence of humidity towards the 3D printed parts, and the testing method of the mechanical properties. The methodology to achieve the stated objectives were explained in Chapter 3. Later, Chapter 4 analysed the tensile strength, flexural test and SEM analysis results. Lastly, the conclusion and recommendation about this study were discussed in Chapter 5.

