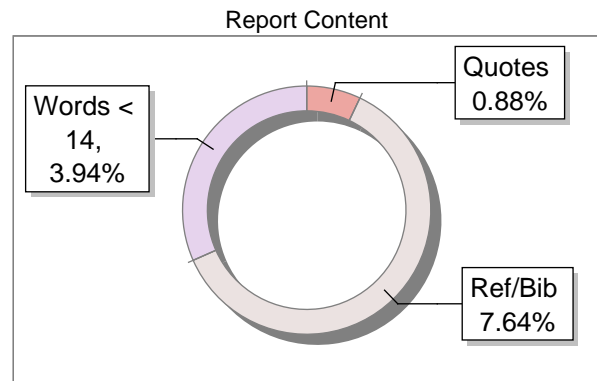
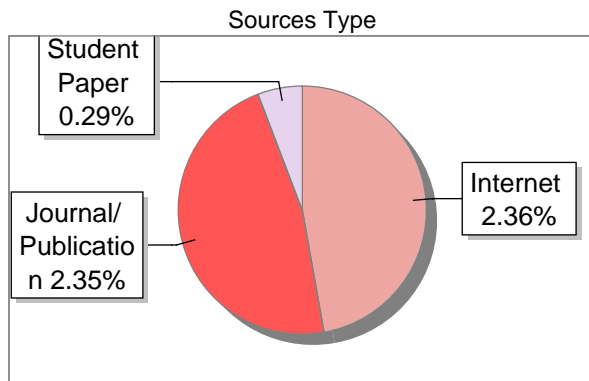
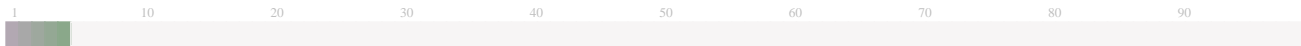


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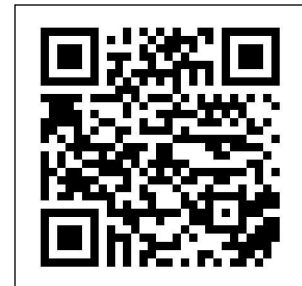
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JNANA SANGAMA, BELAGAVI - 590018



A Project Phase Report on

**“Development of
Metal–Organic Framework Reinforced Polylactic Acid
Composite Fabricated by Additive Manufacturing”**

⁸ Submitted in partial fulfilment of the requirements ¹³ for the award of the degree of

Bachelor of Engineering

In

¹⁵ **Department of Mechanical Engineering**

for the Academic Year: 2025-26

Submitted by

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Certificate

This is to certify that the project work entitled “*Studies on Processing and Characterization of MOF Reinforced PLA Composite Composite Fabricated by Additive Manufacturing*” has been carried out by *Bidyudipto Bandyopadhyay (INT22ME018)*, *Harshitha Priyadarshini AV (INT22ME027)* and *Shantanu Madavan (INT22ME055)*, bonafide students of **Nitte Meenakshi Institute of Technology**, in partial fulfillment of the requirements for the award of the degree of **Bachelor of Engineering in the Department of Mechanical Engineering**, under **Visvesvaraya Technological University**, Belagavi, during the academic year **2025–2026**.

The project report has been examined and approved as it meets the academic requirements specified under the autonomous scheme of **Nitte Meenakshi Institute of Technology** for the said degree.

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Place: Bengaluru

Date:20/05/2026

i

DECLARATION BY THE STUDENTS

We, the project group members, hereby declare that the project work entitled “**Development of Metal–Organic Framework Reinforced Polylactic Acid Composite Fabricated by Additive Manufacturing**” was carried out by us under the esteemed guidance of **Dr. J Sudheer Reddy**, Dean Academics, **Department of Mechanical Engineering, Nitte Meenakshi Institute of Technology, Bengaluru** in partial fulfilment of the requirements for the award of the degree of **Bachelor of Engineering in Mechanical Engineering** of **Visvesvaraya Technological University, Belagavi** during the academic year **2025-26**.

We further declare that the work embodied in this project report is original and **has not been submitted to any other University or Institution** for the award of any degree or diploma.

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Place: Bengaluru

Date: 21-05-2026

ii

Abstract

The increasing demand for high-performance materials in additive manufacturing has driven extensive research toward enhancing commonly used thermoplastics such as Polylactic Acid (PLA). PLA is widely employed in Fused Deposition Modeling (FDM) due to its biodegradability, ease of processing, and cost-effectiveness. However, its relatively low mechanical strength and limited thermal resistance restrict its application in load-bearing and high-performance engineering components.

This study focuses on the development and characterization of Metal–Organic Framework (MOF)-reinforced PLA composites aimed at improving the mechanical and functional properties of PLA for advanced additive manufacturing applications. A solvent-assisted processing technique was adopted to promote uniform dispersion of MOF particles within the PLA matrix. The processed material was subsequently subjected to controlled thermal treatment, followed by the fabrication of composite material suitable for FDM-based 3D printing.

Standardized test specimens were designed and manufactured with varying infill densities to evaluate the influence of internal geometry on mechanical performance. Experimental investigations were carried out using a 25 kN hydraulic Universal Testing Machine to conduct tensile and fatigue tests. The results indicated noticeable improvements in specimen quality

tensile and fatigue tests. The results indicated noticeable improvements in specimen quality, surface finish, tensile strength, and fatigue resistance when compared to pure PLA samples.

The enhancement in mechanical performance can be attributed to improved interfacial bonding between the PLA matrix and MOF particles, which facilitates efficient stress transfer and delays ¹⁰² crack propagation under cyclic loading conditions. In addition, the optimized dispersion of MOFs contributed to improved structural stability and consistency during the printing process. ¹²⁴ The findings of this study demonstrate the potential of MOF-reinforced PLA composites as a feasible and scalable solution for the development of high-performance components through additive manufacturing. This work contributes toward the advancement of material engineering in FDM technology, enabling broader applications in industries requiring improved durability, strength, and sustainability.

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CHAPTER 1

INTRODUCTION

Additive Manufacturing (AM) has emerged as an important manufacturing technology due to its ability to produce complex geometries directly from digital models with reduced material wastage and improved design flexibility. Among the various AM techniques, Fused Deposition Modeling (FDM) is widely used because of its simplicity, low cost, and ease of

operation. In FDM, thermoplastic materials are melted and deposited layer-by-layer to fabricate three-dimensional components.

Polylactic Acid (PLA) ⁶ is one of the most commonly used materials in FDM because of its biodegradability, ease of processing, and compatibility with additive manufacturing systems. However, PLA exhibits limitations such as low mechanical strength, brittleness, and poor thermal resistance, which restrict its application in high-performance engineering components.

To overcome these limitations, reinforcement materials such as fibers, ceramic fillers, and ¹⁰⁹ nanoparticles have been incorporated into PLA matrices. ⁴⁹ In recent years, Metal–Organic Frameworks (MOFs) have gained attention as potential reinforcement materials because of their high surface area, porous structure, and favorable interfacial characteristics. However, achieving proper dispersion of MOF particles within PLA while maintaining printability remains a major challenge.

This study focuses on the development of MOF-reinforced PLA composites for additive manufacturing applications. The work involves solvent-assisted composite preparation, pellet extrusion-based 3D printing, and fabrication of ASTM-standard specimens for tensile and fatigue testing ²⁵ to evaluate the feasibility and mechanical performance of the developed composite system.

1.1 Background

⁷ Additive Manufacturing (AM) has significantly transformed modern manufacturing by enabling the fabrication of complex components with reduced material wastage, shorter production time, and improved design flexibility. Among the various AM techniques, Fused Deposition Modeling (FDM) ⁶ is one of the most widely adopted processes because of its simplicity, affordability, and compatibility with thermoplastic materials.

Polylactic Acid (PLA) is extensively used in FDM applications due to its biodegradability, renewable origin, low warping tendency, and ease of processing. Despite these advantages, PLA exhibits limitations such as relatively low mechanical strength, brittleness, and limited thermal resistance, which restrict its application in structural and high-performance

engineering components.

To improve the performance of PLA, several reinforcement approaches involving fibers, ceramic fillers, and nanoparticles have been investigated. These reinforcements aim to improve the mechanical strength, stiffness, durability, and thermal stability of printed parts. However, challenges such as poor filler dispersion, weak interfacial bonding, and processing difficulties continue to affect the consistency and reliability of reinforced PLA composites.

Metal-Organic Frameworks (MOFs) are a class of porous crystalline materials formed through the coordination of metal ions and organic ligands. MOFs are known for their exceptionally high surface area, tunable pore structures, and good thermal and chemical stability. These unique properties have led to increasing interest in their application across fields such as gas storage, catalysis, filtration, sensing, energy storage, and polymer composites.

In recent years, the use of MOFs as reinforcement materials in polymer systems has gained attention because of their potential to improve interfacial interaction and stress transfer within the matrix material. However, the application of MOFs in Additive Manufacturing, particularly in FDM-based PLA composites, remains relatively unexplored. Challenges related to particle dispersion, agglomeration, printability, and material processability continue to limit their practical implementation in AM applications.

The present study focuses on the development of Fe-MOF reinforced PLA composites for additive manufacturing applications. The work investigates solvent-assisted composite preparation, pellet extrusion-based 3D printing, and fabrication of ASTM-standard specimens for mechanical evaluation of the developed composite system.

1.2 Motivation

The growing demand for lightweight, durable, and sustainable materials in additive manufacturing has created the need for improving the performance of commonly used thermoplastics such as Polylactic Acid (PLA). Although PLA is widely used in FDM applications because of its biodegradability, ease of processing, and cost-effectiveness, its

relatively low mechanical strength and limited thermal resistance restrict its use in structural and load-bearing applications.

Several reinforcement approaches involving fibers, ceramic fillers, and nanoparticles have been investigated ⁵⁰ to improve the performance of PLA composites. However, issues such as poor filler dispersion, agglomeration, weak interfacial bonding, and processing difficulties continue to affect the reliability and consistency of reinforced materials used in additive manufacturing.

Metal–Organic Frameworks (MOFs) ¹³⁶ have recently gained attention as potential reinforcement materials ⁴⁰ because of their high surface area, porous structure, and favorable interfacial characteristics. Despite their growing applications in material science, the use of MOFs in additive manufacturing-based polymer composites remains limited, particularly in PLA systems fabricated using extrusion-based processes.

This study was motivated by the need to explore the feasibility of incorporating MOFs into PLA for additive manufacturing ⁸⁰ applications while addressing challenges related to material processing, printability, and mechanical performance. The work also aims to evaluate the suitability of pellet extrusion-based additive manufacturing for fabricating reinforced PLA composite specimens for mechanical testing and further development.

1.3 Problem Statement

¹⁴ Polylactic Acid (PLA) is one of the most widely used thermoplastics ² in Fused Deposition Modeling (FDM) because of its biodegradability, ease of processing, and compatibility with additive manufacturing systems. However, the relatively low mechanical strength, brittleness, and limited thermal resistance of PLA restrict its application in structural and load-bearing engineering components.

Several reinforcement techniques involving fibers, ceramic fillers, and nanoparticles have been explored ⁵⁰ to improve the performance of PLA-based composites. Although these reinforcements can enhance material properties, challenges such as non-uniform filler dispersion, particle agglomeration, weak interfacial bonding, and poor extrusion behavior continue to affect the consistency and reliability of FDM-fabricated components.

Metal–Organic Frameworks (MOFs) possess unique properties ⁵⁹ such as high surface area, porous structure, and favorable interfacial characteristics, making them potential reinforcement

materials for polymer composites. However, the use of MOFs in FDM-based PLA composites remains relatively unexplored because of challenges associated with particle dispersion, material processability, and extrusion stability.

Problem Statement:

The primary problem addressed in this study is the difficulty in developing an Fe-MOF reinforced PLA composite that can be consistently processed and fabricated using extrusion-based FDM additive manufacturing while maintaining acceptable dispersion characteristics and mechanical performance.

1.4 OBJECTIVES

The primary objective of this study is to develop and evaluate an Fe-MOF reinforced PLA composite for extrusion-based Fused Deposition Modeling (FDM) applications.

The specific objectives of the work are as follows:

- To prepare Fe-MOF reinforced PLA composite material using a solvent-assisted processing technique.
- To study the effect of different solvent and precipitation systems on composite preparation and material behavior.
- To fabricate PLA–MOF composite specimens using pellet extrusion-based additive manufacturing.
- To manufacture ASTM-standard tensile and fatigue test specimens using varying infill densities.
- To evaluate the tensile and fatigue performance of the developed composite material.
- To study the influence of reinforcement incorporation and printing parameters on the mechanical behavior of FDM-fabricated components.
- To assess the feasibility of using MOF-reinforced PLA composites for additive manufacturing applications.

1.5 Scope of Work

The scope of the present work is focused on the development and evaluation of Fe-MOF reinforced PLA composites for extrusion-based additive manufacturing applications. The study

includes the preparation of composite material using solvent-assisted processing techniques and the investigation of suitable solvent and precipitation systems for improving material processability.

The work further involves the fabrication of composite specimens using pellet extrusion-based Fused Deposition Modeling (FDM) through a single-screw extrusion system. ASTM-standard tensile and fatigue specimens were manufactured using varying infill densities to study the influence of internal structural parameters on mechanical behavior.

Mechanical characterization of the fabricated specimens was carried out through tensile and fatigue testing using a Universal Testing Machine. The scope of the study is limited to material preparation, additive manufacturing, and mechanical performance evaluation of the developed composite system.

CHAPTER 2

LITERATURE REVIEW

2.1 Additive Manufacturing and Fused Deposition Modeling (FDM)

Additive Manufacturing (AM) is a manufacturing process in which components are fabricated layer-by-layer directly from digital models. Compared to conventional manufacturing methods, AM offers advantages such as reduced material wastage, improved design flexibility, and rapid prototyping capability. Because of these advantages, AM is widely used in industries such as aerospace, automotive, biomedical, and product development.

Fused Deposition Modeling (FDM) is one of the most commonly used additive manufacturing techniques due to its simplicity, affordability, and ease of operation. In FDM, thermoplastic material is heated and extruded through a nozzle to build components layer-by-layer. The quality and mechanical performance of FDM-printed parts are influenced by factors such as material properties, nozzle temperature, layer adhesion, and infill density.

The growing use of FDM in engineering applications has increased the demand for reinforced thermoplastic materials with improved mechanical and thermal properties.

2.2 POLYLACTIC ACID (PLA) IN ADDITIVE MANUFACTURING

Polylactic Acid (PLA) is one of the most widely used thermoplastic materials in Fused Deposition Modeling (FDM) because of its biodegradability, ease of processing, low warping tendency, and cost-effectiveness. PLA is derived from renewable resources such as corn starch and sugarcane, making it an environmentally sustainable material for additive manufacturing

applications.

Despite its advantages, PLA exhibits limitations such as low mechanical strength, brittleness, and poor thermal resistance, which restrict its use in structural and high-performance engineering applications. These limitations have led to increasing research on reinforced PLA composites for improving the performance of FDM-fabricated components.

2.3 Reinforcement Techniques for Pla Composites

Various reinforcement techniques have been investigated to improve the mechanical and thermal performance of PLA-based composites used in additive manufacturing. Reinforcement materials such as carbon fiber, glass fiber, ceramic fillers, metal oxides, and nanoparticles have

been incorporated into PLA matrices to improve strength, stiffness, durability, and dimensional stability.

These reinforcements improve load transfer within the material and help reduce crack propagation during mechanical loading. However, challenges such as particle agglomeration, poor interfacial bonding, non-uniform dispersion, and extrusion instability continue to affect the consistency and reliability of reinforced PLA composites fabricated using FDM.

2.4 Metal–Organic Frameworks (MOFs)

Metal–Organic Frameworks (MOFs) are a class of porous crystalline materials formed through the coordination of metal ions and organic ligands. MOFs are known for their high surface area, tunable pore structures, and good thermal and chemical stability, making them suitable for a wide range of engineering and material science applications.

In recent years, MOFs have gained attention as potential reinforcement materials for polymer composites because of their favorable interfacial characteristics and ability to improve stress transfer within the matrix material. Despite their growing applications in fields such as gas storage, catalysis, sensing, and filtration, the use of MOFs in additive manufacturing-based polymer composites remains relatively limited.

2.5 MOFs In Polymer Composites

The incorporation of Metal–Organic Frameworks (MOFs) into polymer matrices has

gained increasing attention because of their potential to improve mechanical performance, interfacial interaction, and structural stability of composite materials. ⁶² The porous structure and high surface area of MOFs promote better interaction between the reinforcement particles and the polymer matrix, which can contribute to improved stress transfer characteristics.

Recent studies on MOF–polymer composites have reported improvements in properties such as tensile strength, thermal stability, and material consistency. However, challenges such as particle agglomeration, poor dispersion, and processing difficulties continue to affect the development of reliable MOF-reinforced composites, particularly in extrusion-based additive manufacturing systems such as FDM.

2.6 Solvent-Assisted Composite Processing

Solvent-assisted processing is commonly used in polymer composite preparation to improve reinforcement dispersion and material mixing within the polymer matrix. In PLA-

based composite systems, solvents such as Dichloromethane (DCM) and chloroform are often used because of their ability to dissolve PLA effectively and facilitate reinforcement incorporation.

The use of solvent-assisted processing can improve particle distribution and interfacial interaction between ¹³⁷ the reinforcement material and the polymer matrix. However, challenges such as particle agglomeration, inconsistent precipitation behavior, solvent removal, and material solidification continue to affect the quality and processability of reinforced composites.

In MOF-reinforced polymer systems, solvent selection and precipitation conditions ¹⁸ play an important role in determining the dispersion characteristics and final morphology of the composite material prior to extrusion-based additive manufacturing.

2.7 Influence Of Printing Parameters

The mechanical performance and structural integrity of FDM-fabricated components are strongly influenced by printing parameters such as nozzle temperature, ² layer height, print speed, raster orientation, and infill density. These parameters affect layer adhesion,

dimensional accuracy, surface finish, and overall mechanical behavior of printed parts.

131 Among these parameters, infill density 3 plays a significant role in determining the tensile strength, stiffness, and fatigue resistance of FDM components. Higher infill densities generally improve internal load distribution and reduce void formation within the printed structure. Understanding the influence of printing parameters is therefore important for optimizing the performance of reinforced PLA composites in additive manufacturing applications.

2.8 Research Gaps Identified

123 Although extensive research has been carried out on PLA-based 97 composites for additive manufacturing applications, limitations related to mechanical strength, brittleness, and thermal resistance continue to restrict the use of PLA in structural engineering applications. Various reinforcement approaches have been explored; however, challenges such as poor particle dispersion, agglomeration, weak interfacial bonding, and extrusion instability still affect the reliability of reinforced PLA composites fabricated using FDM.

Metal–Organic Frameworks (MOFs) have shown promising characteristics as reinforcement materials 40 because of their high surface area and favorable interfacial properties. 72 However, limited research has been reported on the use of MOFs in PLA-based extrusion additive manufacturing systems, particularly with respect to material processability, solvent-assisted composite preparation, and pellet extrusion-based fabrication.

In addition, 96 comparatively fewer studies have investigated the combined influence of reinforcement incorporation and printing parameters such as infill density on the tensile and fatigue behavior of PLA–MOF composites. These gaps highlight the 82 need for further investigation into the development and evaluation of MOF-reinforced PLA composites for additive manufacturing applications. 23

CHAPTER 3

MATERIALS AND METHODOLOGY

This chapter presents the materials, chemicals, and experimental procedures adopted for the development of Fe-MOF reinforced PLA composites for extrusion-based additive manufacturing applications. The study primarily focuses on solvent-assisted composite preparation, pellet extrusion-based Fused Deposition Modeling (FDM), and fabrication of ASTM-standard specimens for mechanical evaluation.

The experimental work involved the preparation of PLA–MOF composite material using different solvent and precipitation systems to study their influence on material behavior, *Thermogravimetric Analysis and Dynamic Mechanical Analysis*. Based on the experimental observations, suitable

dispersion characteristics, and processability. Based on the experimental observations, suitable processing conditions were identified for composite preparation and additive manufacturing.

The developed composite material was further processed using a pellet extrusion-based additive manufacturing system to fabricate tensile and fatigue test specimens with varying infill densities. Mechanical characterization of the fabricated specimens was carried out through tensile and fatigue testing using a Universal Testing Machine (UTM).

3.1 Materials and chemicals

The materials and chemicals used in the present study were selected based on their suitability for solvent-assisted composite preparation and extrusion-based additive manufacturing. The experimental work primarily focused on the development of an Fe-MOF reinforced PLA composite using controlled dissolution and non-solvent precipitation techniques prior to pellet extrusion-based fabrication.

Polylactic Acid (PLA) was used as the base polymer matrix material because of its compatibility with additive manufacturing processes and ease of thermal processing. Fe-MOF particles were incorporated as reinforcement material to study their influence on the mechanical behavior and structural characteristics of the developed composite system. Dichloromethane (DCM) was used as the primary dissolving solvent for PLA because of its rapid dissolution capability, while hexane was used as the non-solvent precipitation agent during composite recovery and solidification.

The prepared composite material was further processed using a pellet extrusion-based additive manufacturing system for the fabrication.

3.1.1 Polylactic Acid (PLA)

Polylactic Acid (PLA) was used as the primary matrix material for composite preparation because of its biodegradability, ease of processing, and widespread use in extrusion-based Fused Deposition Modeling (FDM) applications. PLA is a thermoplastic polymer derived from renewable resources such as corn starch and sugarcane and is commonly preferred in additive manufacturing because of its low warping tendency, stable extrusion behavior, and compatibility with desktop and industrial 3D printing systems.

In the present work, commercially available PLA pellets were used for solvent-assisted composite preparation. The PLA material served as the continuous matrix phase responsible for binding and supporting the Fe-MOF reinforcement particles within the composite structure. The material was selected due to its suitability for pellet extrusion-based additive manufacturing and its ability to undergo dissolution and precipitation during solvent-assisted processing.

However, preliminary experimental observations indicated that the PLA material exhibited sensitivity to solvent processing and precipitation conditions. During initial trials, improper solidification behavior and thin film formation were observed under certain processing conditions, which affected composite recovery and processability. These observations influenced the selection of the final solvent-assisted preparation route adopted in the study.

3.1.2 Fe-MOF reinforcement material

Fe-based Metal–Organic Framework (Fe-MOF) particles were used as the reinforcement material in the PLA matrix. Fe-MOFs are porous crystalline materials formed through the coordination of iron metal ions and organic ligands, resulting in structures with high surface area and favorable interfacial characteristics.

The Fe-MOF reinforcement was incorporated in low weight percentages to study its influence on the processability, printability, and mechanical behavior of the developed PLA composite system. The reinforcement particles were introduced into the dissolved PLA solution during solvent-assisted processing to facilitate dispersion within the polymer matrix prior to precipitation and drying.

During experimental trials, it was observed that excessive particle accumulation could lead to localized agglomeration within the polymer solution, affecting material homogeneity and

composite consistency. Therefore, controlled reinforcement loading and continuous mixing were adopted during composite preparation to improve particle distribution within the PLA matrix.

3.1.3 Dichloromethane (DCM)

Dichloromethane (DCM) was used as the primary solvent for dissolving PLA during composite preparation. DCM was selected because of its rapid dissolution behavior and ability to effectively break down PLA pellets to form a processable polymer solution suitable for reinforcement incorporation.

Among the solvent systems evaluated during the experimental work, DCM demonstrated the most effective dissolution behavior and comparatively stable processing characteristics for PLA-based composite preparation. The use of DCM enabled easier incorporation and mixing of Fe-MOF particles within the polymer solution prior to non-solvent precipitation.

3.1.4 Hexane

Hexane was used as the non-solvent precipitation agent during composite preparation. The addition of hexane to the dissolved PLA composite solution promoted precipitation and recovery of the composite material by reducing the solubility of PLA within the solvent system.

Experimental observations showed that hexane provided comparatively better solidification behavior and formation of recoverable composite material when compared to other precipitation conditions investigated during preliminary trials. The precipitation process enabled the formation of solid composite feedstock suitable for drying and subsequent pellet extrusion-based additive manufacturing.

3.2 Experimental methodology overview

The experimental ²³ methodology adopted in the present study was developed to investigate the feasibility of producing an Fe-MOF reinforced PLA composite suitable for extrusion-based additive manufacturing applications. The methodology primarily focused on solvent-assisted composite preparation, non-solvent precipitation, pellet extrusion-based fabrication, and mechanical evaluation of the developed composite specimens.

The overall workflow of the study involved material selection, polymer dissolution, reinforcement incorporation, composite recovery through precipitation, drying, additive manufacturing, specimen fabrication, and mechanical testing. Each stage of the experimental

116 work was carried out systematically to maintain consistency in material preparation and specimen fabrication throughout the study.

The prepared PLA–MOF 117 composite material was fabricated using a pellet extrusion-based additive manufacturing system to produce ASTM-standard tensile and fatigue test specimens. Mechanical characterization was subsequently 21 carried out to evaluate the influence of reinforcement incorporation and printing parameters on the behavior of 24 the developed composite system.

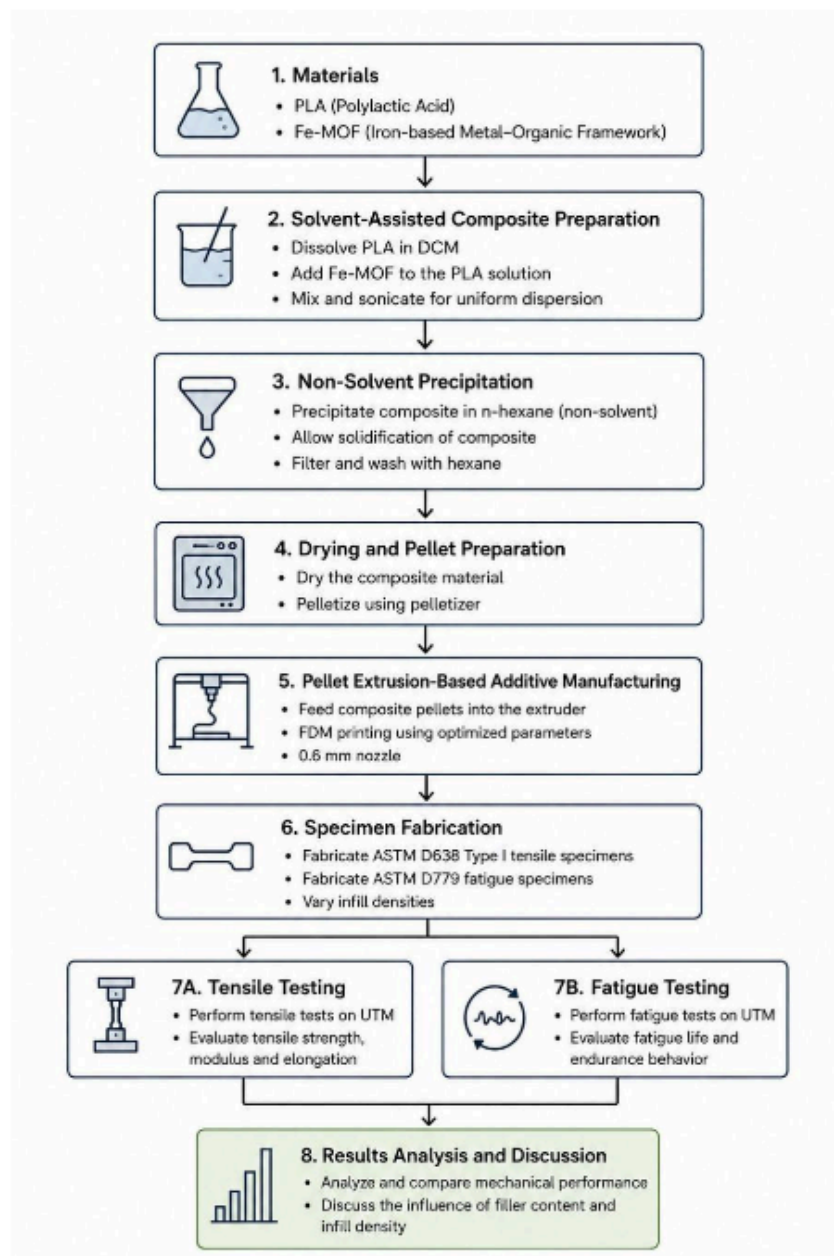


Fig 3.1: Methodology Flow Diagram

The detailed experimental procedure adopted during the study is described below:

1. PLA material collection:

PLA material was collected and selected as the primary polymer **matrix material for composite preparation because of its** compatibility with extrusion-based Fused Deposition Modeling (FDM) applications. The material served as the continuous matrix phase responsible for binding and supporting the reinforcement **particles within the developed composite** system.

2. Pelletization of PLA material:

The collected PLA **material was processed into pellet form** to obtain feedstock suitable for solvent-assisted composite preparation and pellet extrusion-based additive manufacturing. The use of recycled PLA material was also considered as part of the study to promote sustainable material utilization and evaluate the feasibility of reusing PLA waste in additive manufacturing applications.



Fig 3.2: Pelletized PLA

3. Drying of PLA pellets:

The prepared PLA pellets were dried prior to solvent processing to reduce moisture content present within the material. Proper drying was necessary to improve dissolution behavior, reduce processing inconsistencies, and minimize the possibility of moisture-related defects

during composite preparation and extrusion processing.

4. Dissolution of PLA:

The dried PLA pellets were gradually introduced into Dichloromethane (DCM) under controlled stirring conditions. DCM acted as the primary dissolving solvent and enabled the formation of a processable polymer solution suitable for reinforcement incorporation and composite preparation.

5. Addition of Fe-MOF reinforcement:

Fe-MOF particles were incorporated into the dissolved PLA solution in controlled low-weight percentages to prepare the reinforced composite system. The reinforcement material was introduced gradually to reduce excessive particle accumulation and improve interaction between the reinforcement particles and the polymer matrix.

6. Mixing and dispersion process:

Continuous stirring and mixing of the PLA–MOF solution was carried out to improve dispersion of Fe-MOF particles within the dissolved polymer system. This step was necessary to reduce localized agglomeration and promote more consistent distribution of reinforcement particles throughout the composite material prior to precipitation.





Fig 3.3: Controlled stirring in standard ATM



Fig 3.4: Ferrous MOF-PLA mixture

7. Non-solvent precipitation:

Hexane was added to the prepared PLA–MOF solution to initiate non-solvent precipitation of the composite material. The addition of hexane reduced the solubility of PLA within the solvent system and promoted recovery of the composite material in solid form suitable for further processing.



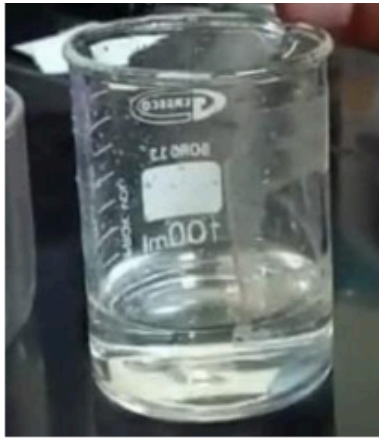


Fig 3.5: Non-solvent precipitation of PLA MOF

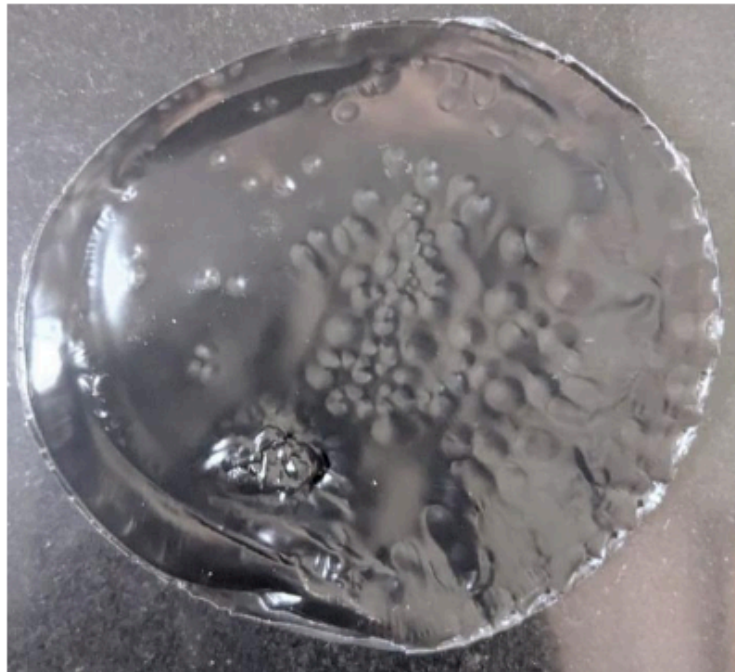


Fig 3.6: Result of Non-solvent Precipitation

8. Pelletization of PLA–MOF composite:

The recovered composite material was separated and processed into pellet form to obtain feedstock suitable for pellet extrusion-based additive manufacturing. Pelletization was carried out to improve material handling and ensure compatibility with the pellet extrusion printing system used in the study.

9. Additive manufacturing of test specimens:

The prepared PLA–MOF composite pellets were processed using a Picoeact G5 Ultra pellet extrusion additive manufacturing system equipped with a single-screw extrusion mechanism and a 0.6 mm nozzle. ASTM-standard tensile and fatigue specimens were fabricated using controlled printing parameters and varying infill densities.

10. Mechanical testing:

The fabricated specimens were subjected to tensile and fatigue testing using a Universal Testing Machine (UTM) to evaluate the mechanical behavior of the developed composite material. The testing process was carried out to study the influence of reinforcement incorporation and printing parameters on specimen performance.

11. Results and analysis:

The experimental results obtained from the mechanical testing process were analyzed to evaluate the printability, structural behavior, tensile performance, and fatigue characteristics of the developed PLA–MOF composite system. The observations obtained

during the study were further used to assess the feasibility of using Fe-MOF reinforced PLA composites for extrusion-based additive manufacturing applications.

3.3 Solvent-assisted composite preparation

The preparation of the Fe-MOF reinforced PLA composite was carried out using a solvent-assisted processing technique followed by non-solvent precipitation. The methodology was developed to improve the incorporation of Fe-MOF particles within the PLA matrix prior to pellet extrusion-based additive manufacturing. The overall process involved dissolution of PLA using Dichloromethane (DCM), reinforcement incorporation, controlled mixing, non-solvent precipitation using n-hexane, and recovery of the composite material.

3.3.1 PLA dissolution using DCM

PLA pellets were initially dried to reduce moisture content before solvent processing. The dried PLA pellets were gradually introduced into Dichloromethane (DCM) under controlled stirring conditions. DCM was selected as the primary solvent because of its rapid dissolution capability and stable interaction with PLA during preliminary experimental trials.

The dissolution process was carried out until the PLA pellets were sufficiently dissolved

to form a processable polymer solution suitable for reinforcement incorporation. Continuous stirring was maintained throughout the process to improve dissolution consistency and reduce localized accumulation of partially dissolved material.

Experimental observations indicated that DCM provided comparatively faster dissolution behavior and improved processability when compared to other solvent conditions evaluated during the initial stages of the study.

3.3.2 Fe-MOF incorporation and mixing

After obtaining the dissolved PLA solution, Fe-MOF particles were gradually introduced into the polymer solution in controlled low-weight percentages. The reinforcement particles were added slowly under continuous stirring conditions to reduce excessive particle accumulation and improve interaction between the reinforcement material and the dissolved PLA matrix.

Continuous mixing was maintained during the incorporation process to improve particle dispersion and reduce localized agglomeration within the solution. The reinforcement loading

was intentionally maintained at low concentrations to preserve processability and extrusion stability during subsequent additive manufacturing stages.

During preliminary experimental observations, higher localized particle concentration and insufficient mixing were found to affect material consistency and dispersion behavior. Therefore, controlled addition and continuous stirring were adopted throughout the composite preparation process.

3.3.3 Non-solvent precipitation using n-hexane

Following reinforcement incorporation and mixing, a non-solvent precipitation process was carried out using n-hexane. The prepared PLA-MOF solution was gradually introduced into n-hexane to initiate precipitation and recovery of the composite material.

The addition of n-hexane reduced the solubility of PLA within the solvent system and promoted solidification of the dissolved composite material. Experimental observations during preliminary processing trials indicated that n-hexane provided comparatively better recovery

preliminary processing trials indicated that n-hexane provided comparatively better recovery behavior and formation of recoverable composite material suitable for further processing.

The precipitation stage also played an important role in separating the composite material from the solvent system prior to drying and pellet extrusion processing.

3.3.4 Composite recovery and drying

The precipitated PLA–MOF composite material was separated and collected after completion of the precipitation process. The recovered material was subsequently dried to remove residual solvent content prior to extrusion processing.

Proper drying of the recovered composite material was necessary to improve handling characteristics, reduce solvent retention, and ensure stable extrusion behavior during pellet extrusion-based additive manufacturing. The dried composite material was then processed into pellet form for subsequent specimen fabrication.

3.4 Pellet extrusion additive manufacturing

The fabricated PLA–MOF composite material was processed using a pellet extrusion-based additive manufacturing system for the fabrication of ASTM-standard mechanical test specimens. Pellet extrusion additive manufacturing was adopted in the present study to enable

direct processing of the prepared composite feedstock without the additional requirement of filament fabrication.

Compared to conventional filament-based Fused Deposition Modeling (FDM), pellet extrusion systems provide improved flexibility for processing experimental composite materials and recycled polymer feedstock. The approach also reduces intermediate processing stages associated with filament production and enables direct utilization of composite pellets for specimen fabrication.

3.4.1 Pellet extrusion manufacturing setup

The additive manufacturing process was carried out using a Piocreat G5 Ultra pellet extrusion-based 3D printing system equipped with a single-screw extrusion mechanism. The

system was selected because of its compatibility with pellet-based polymer processing and its suitability for experimental composite material fabrication.

A 0.6 mm extrusion nozzle was used during the printing process to facilitate stable material flow and improve extrusion consistency of the prepared PLA–MOF composite material. The pellet extrusion system enabled direct feeding of the composite pellets into the extrusion unit, where the material was heated and deposited layer-by-layer during specimen fabrication.

The use of pellet extrusion additive manufacturing also supported the utilization of recycled PLA-based feedstock within the study, aligning with the sustainability-oriented objectives of the experimental work.



Fig 3.7: Piocreat G5 Ultra single screw extrusion printer

3.4.2 Printing parameters

The fabricated specimens were printed using controlled process parameters to maintain extrusion stability and specimen consistency during additive manufacturing. ²¹ Parameters such as extrusion temperature, print speed, layer deposition behavior, and infill density were monitored during the fabrication process.

Different infill densities ³⁶ were selected for specimen fabrication to study their influence on the mechanical behavior and structural characteristics of the printed PLA–MOF composite

specimens. The printing process was carried out under controlled conditions to reduce printing defects and maintain dimensional consistency across the fabricated samples.

3.4.3 ASTM specimen fabrication

ASTM-standard specimens were fabricated for tensile and fatigue testing of the developed PLA–MOF composite material. Tensile specimens were prepared according to ASTM D638 standards, while fatigue specimens were fabricated based on the required dimensions for cyclic loading evaluation.

A total of six specimens were fabricated during the study, consisting of tensile and fatigue test samples manufactured using the prepared composite material. The fabricated specimens were visually inspected after printing to evaluate surface finish, layer consistency, dimensional stability, and overall print quality prior to mechanical testing.



Fig 3.8: Piocreat G5 Ultra single screw extrusion printer printing ASTM sample

3.4.4 Fabrication observations

During the additive manufacturing process, the prepared PLA–MOF composite material demonstrated acceptable extrusion behavior and specimen formation under pellet extrusion conditions. However, preliminary observations indicated that reinforcement incorporation and

material processing conditions influenced extrusion consistency and surface finish of the fabricated components.

Localized particle accumulation and minor variations in material flow were observed during certain stages of the printing process, highlighting the influence of reinforcement dispersion and composite preparation on additive manufacturing performance. Despite these challenges, ASTM-standard specimens suitable for mechanical evaluation were successfully fabricated using the developed composite system.

3.5 Mechanical testing methodology

Mechanical testing of the fabricated PLA–MOF composite specimens was carried out to evaluate the tensile and fatigue behavior of the developed material system under controlled loading conditions. ASTM-standard specimens fabricated through pellet extrusion-based additive manufacturing were used for the experimental evaluation.

The testing methodology was designed to study the influence of infill density and Fe-MOF reinforcement on the mechanical performance of the printed composite specimens. Tensile testing and fatigue testing were conducted using a Universal Testing Machine (UTM), and the corresponding loading parameters were selected based on specimen geometry, calculated stress values, and structural limitations of the fabricated specimens.

3.5.1 Tensile testing methodology

Tensile testing was conducted to evaluate the tensile strength and deformation behavior of the fabricated PLA–MOF composite specimens under bilateral loading conditions. ASTM D638 (at 75% scale) standard tensile specimens were fabricated using varying infill densities to study the influence of internal structural configuration on tensile performance.

The fabricated tensile specimens were mounted on the Universal Testing Machine and subjected to gradually increasing axial tensile loading until specimen failure occurred. During the testing process, stress–strain data was continuously recorded to evaluate tensile response, load-bearing capability, and deformation characteristics of the developed composite material.

Three tensile specimens with infill densities of 25%, 50%, and 75% were selected for the study. The cross-sectional area of the specimens was maintained at approximately 30 mm² for calculation consistency.

The tensile load values were determined using the standard stress relation:

$$\sigma = \frac{F}{A}$$

Where:

σ = Ultimate tensile stress (MPa)

F = Failure load (N)

A = Cross-sectional area (mm²)

Rearranging the equation:

$$F = \sigma \times A$$

Specimen ID	Infill Density (%)	Cross-sectional Area (mm ²)
MOF-T25	25	30
MOF-T50	50	30
MOF-T75	75	30

Table 3.1: Tensile testing parameters

The selected infill densities enabled evaluation of the relationship between internal structural variation and tensile behavior of the fabricated PLA–MOF composite specimens.

3.5.2 Fatigue testing methodology

Fatigue testing was conducted to evaluate the cyclic loading behavior and endurance characteristics of the fabricated PLA–MOF composite specimens under repeated loading conditions. ASTM-standard fatigue specimens fabricated through pellet extrusion-based additive manufacturing were subjected to cyclic loading using the Universal Testing Machine.

The ³³ fatigue loading conditions were selected based on the estimated load-bearing capability of the fabricated specimens and the corresponding infill density configurations. Controlled cyclic loading parameters were adopted to avoid immediate catastrophic failure and enable observation of specimen response under fluctuating stress conditions.

Fatigue specimens with infill densities of 10%, 20%, and 30% were selected for evaluation. The applied loading conditions included maximum load, minimum load, mean load, load amplitude, maximum stress, and loading frequency.

The mean load and load amplitude used during fatigue testing were determined using the following relations:

$$\text{Mean Load} = \frac{F_{max} + F_{min}}{2}$$

$$\text{Load Amplitude} = \frac{F_{max} - F_{min}}{2}$$

Where:

F_{max} = Maximum applied load

F_{min} = Minimum applied load

Infill Density (%)	Fmax (N)	Fmin (N)	Mean Load (N)	Load Amplitude (N)	Frequency (Hz)
10	540	54	297	243	2
20	600	60	330	270	2
30	630	63	346.5	283.5	2

Table 3.2: Fatigue loading parameters

The fatigue testing methodology was designed to evaluate the cyclic response and

structural endurance characteristics of the fabricated PLA–MOF composite specimens under controlled fluctuating loading conditions.

3.5.3 Test evaluation parameters

The mechanical testing process was conducted to evaluate tensile strength, deformation behavior, cyclic loading response, and structural stability of the fabricated PLA–MOF composite specimens. During testing, observations related to specimen deformation, layer adhesion behavior, structural integrity, and overall print consistency were recorded for further analysis in the results and discussion chapter.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter presents the experimental results obtained from the tensile and fatigue testing of the fabricated PLA–MOF composite specimens produced through pellet extrusion-based additive manufacturing. The influence of infill density, reinforcement incorporation, and printing behavior on the mechanical performance of the developed composite system is discussed based on the obtained test data and experimental observations.

The chapter also discusses the processing behavior of the developed composite material during additive manufacturing, including extrusion consistency, specimen fabrication quality, and factors affecting the overall performance of the printed specimens.



Fig 4.1: ASTM sample in UTM machine

4.1 Tensile test results and discussion

Tensile testing was conducted to evaluate the mechanical behavior of the fabricated PLA–MOF composite specimens under uniaxial loading conditions. ASTM-standard tensile specimens fabricated with infill densities of 25%, 50%, and 75% were tested using a Universal Testing Machine to study the influence of internal structural configuration on tensile performance.

The stress–strain behavior obtained from the tensile tests was used to compare the load-bearing characteristics and deformation response of the fabricated specimens. The tensile test graphs corresponding to different infill density configurations are presented below.

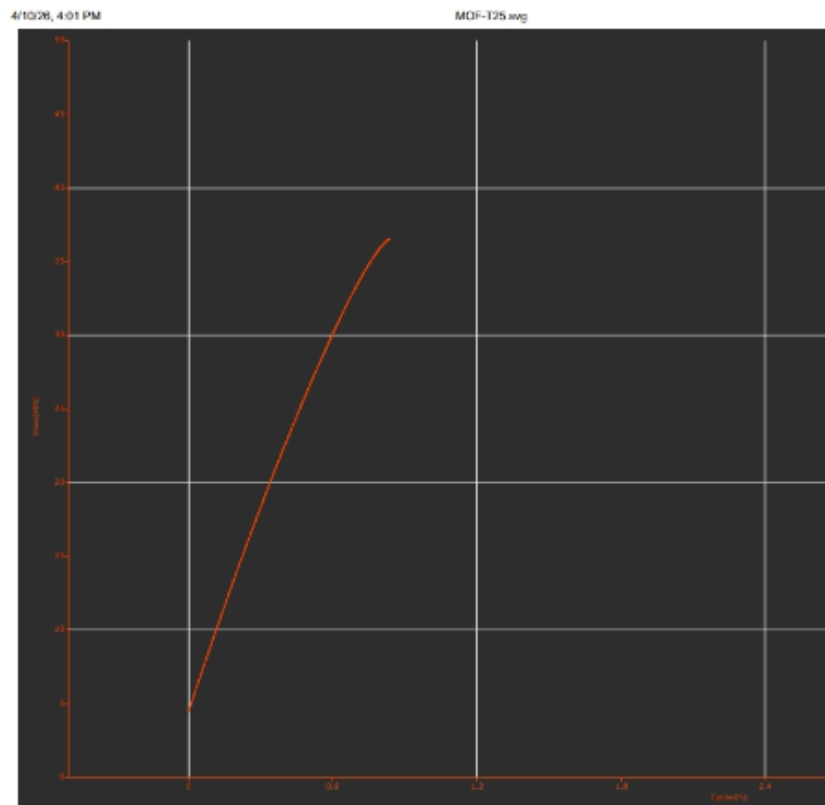
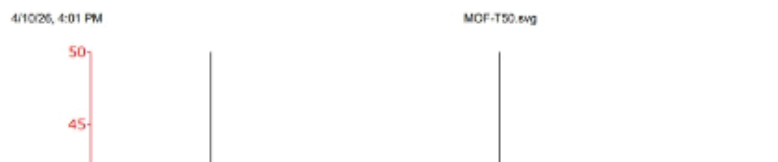


Fig 4.2: 25% infill tensile test stress-strain graph



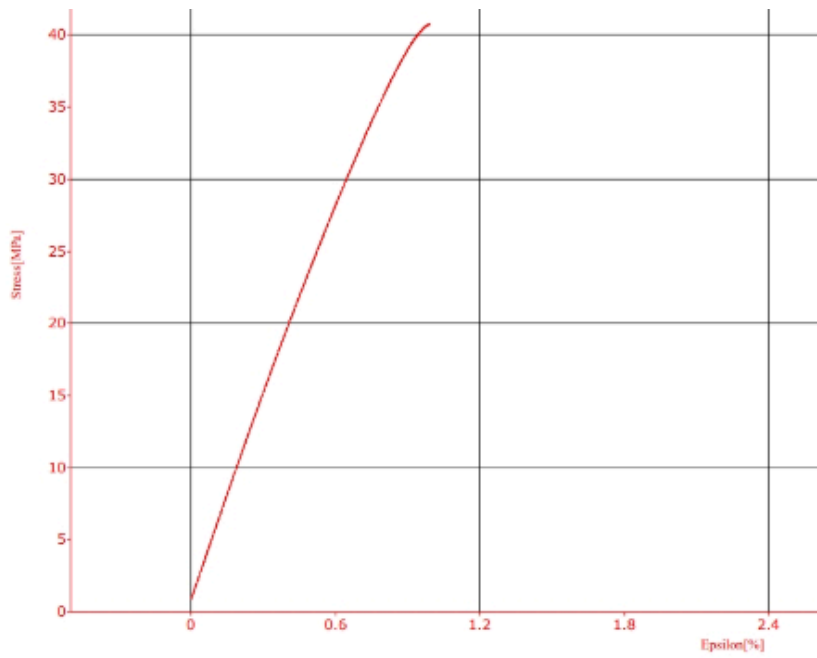


Fig 4.3: 50% infill tensile test stress-strain graph

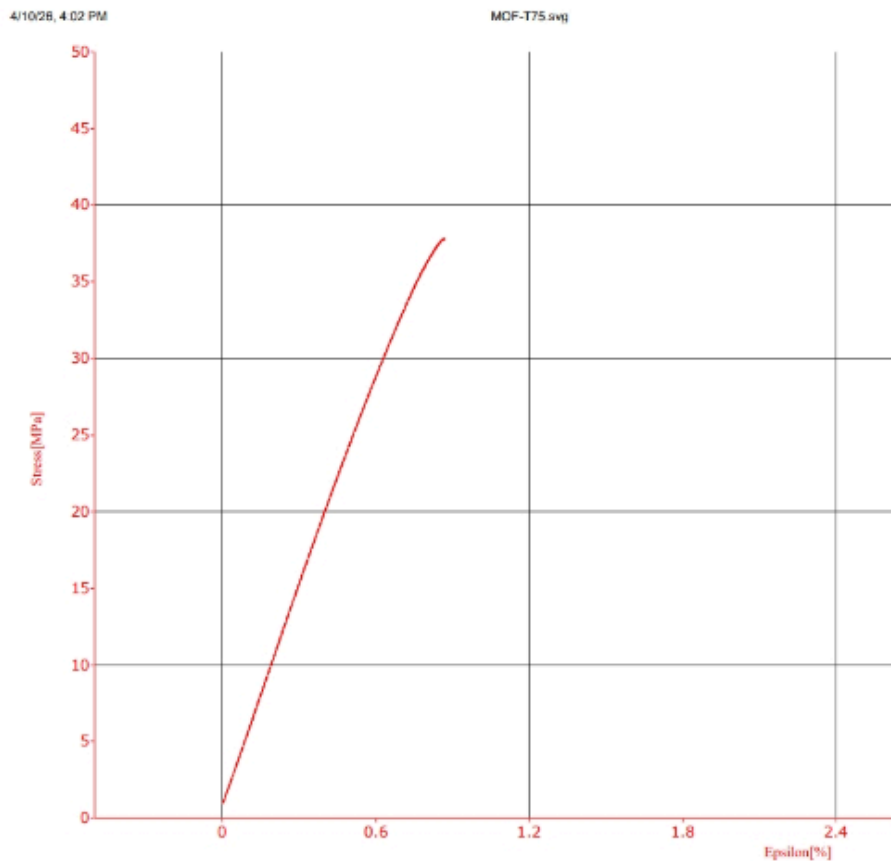


Fig 4.4: 75% infill tensile test stress-strain graph

Specimen ID	Infill Density (%)	Ultimate Tensile Stress (MPa)	Failure Load (N)	Failure Load (kN)	Approximate Failure Strain (%)	General Observation
MOF-T25	25	36.5	1095	1.095	~0.82	Lower load-bearing capability with earlier deformation response
MOF-T50	50	40.8	1224	1.224	~0.95	Highest tensile strength and improved structural continuity

Specimen ID	Infill Density (%)	Ultimate Tensile Stress (MPa)	Failure Load (N)	Failure Load (kN)	Approximate Failure Strain (%)	General Observation
MOF-T75	75	37.7	1131	1.131	~0.88	Stable tensile response with improved internal density

Table 4.1: Tensile test observations

91 The tensile test results indicate that infill density significantly influenced the tensile behavior of the fabricated PLA–MOF composite specimens. The specimen fabricated with 50% infill density demonstrated the highest tensile strength among the tested configurations.

This behavior may be attributed to improved load distribution and reduced internal void concentration within the printed structure.

The 25% infill specimen exhibited comparatively lower tensile strength because of reduced internal material continuity and increased void presence within the specimen structure. Although the 75% infill specimen demonstrated improved structural density, the tensile response did not increase proportionally, indicating the influence of internal layer interaction and material deposition behavior during additive manufacturing.

The obtained stress–strain curves also indicated typical brittle thermoplastic deformation characteristics, with limited plastic deformation prior to specimen failure. Overall, the tensile testing results demonstrated that infill density and internal structural configuration played a significant role in determining the tensile performance of the fabricated PLA–MOF composite specimens.

4.2 Fatigue test results and discussion

Fatigue testing was conducted to evaluate the cyclic loading behavior and endurance characteristics of the fabricated PLA–MOF composite specimens under repeated loading conditions. ASTM-standard fatigue specimens fabricated with infill densities of 10%, 20%, and 30% were subjected to cyclic loading using a Universal Testing Machine.

The fatigue response of the specimens was evaluated using cyclic stress behavior, load stability, and the number of cycles sustained prior to failure. The fatigue graphs corresponding to different infill density configurations are presented below.



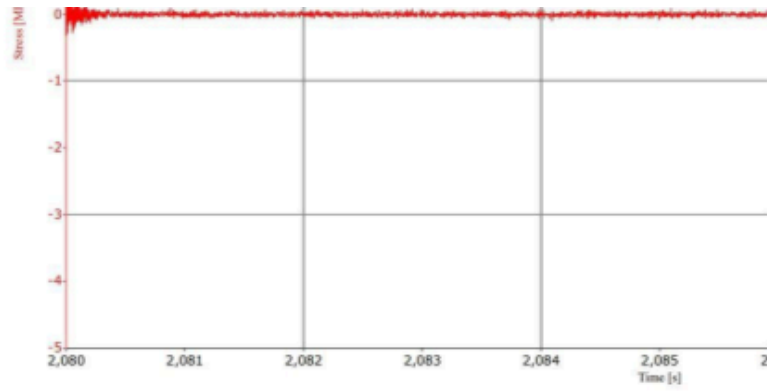


Fig 4.5: 10% infill fatigue test force-time graph

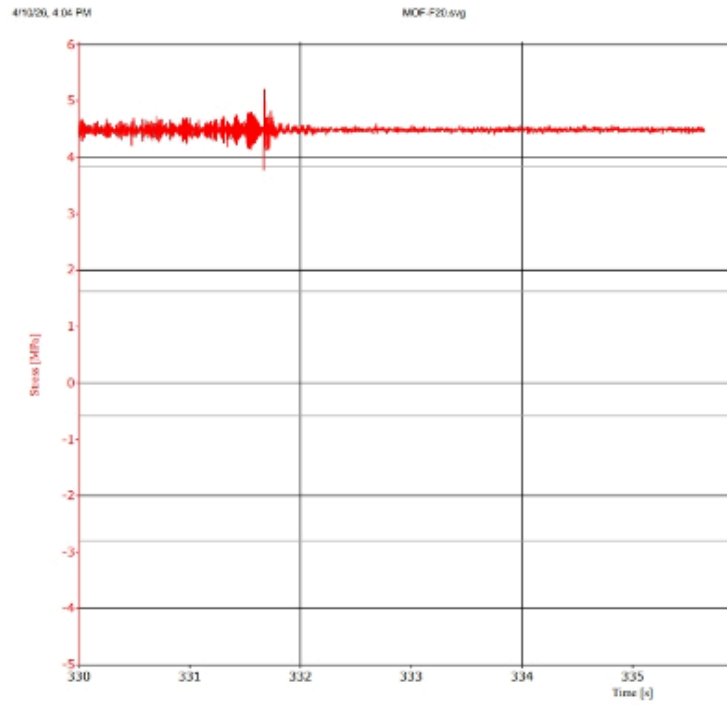
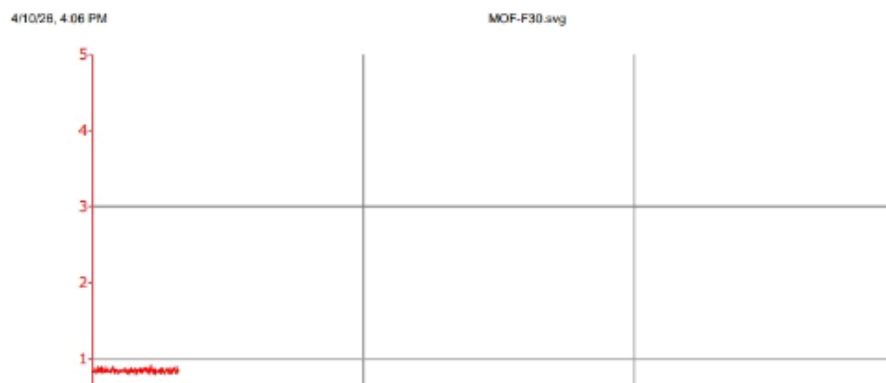


Fig 4.6: 20% infill fatigue test force-time graph



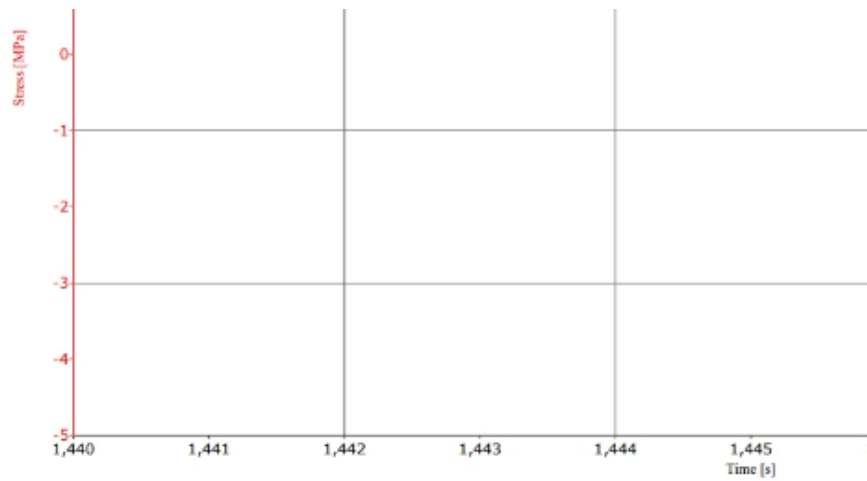


Fig 4.7: 30% infill fatigue test force-time graph

Specimen ID	Infill Density (%)	Fmax (N)	Fmin (N)	Mean Load (N)	Load Amplitude (N)	Frequency (Hz)	Approximate Number of Cycles	General Observation
MOF-F10	10	540	54	297	243	2	~4170	Lower cyclic stability with earlier fatigue response
MOF-F20	20	600	60	330	270	2	~670	Moderate cyclic stability and improved endurance behavior
MOF-F30	30	630	63	346.5	283.5	2	~2890	Improved cyclic response and better structural consistency

Table 4.2: Fatigue test observations

⁸⁴ The fatigue testing results demonstrated that infill density significantly influenced the cyclic loading behavior of the fabricated PLA–MOF composite specimens. ⁴⁷ Specimens with higher infill density exhibited improved internal structural continuity, which contributed to better load distribution during repeated loading conditions.

The specimen fabricated with lower infill density showed comparatively earlier instability during cyclic loading because of increased internal void concentration and reduced structural support within the printed geometry. In contrast, specimens with increased infill density demonstrated comparatively improved cyclic stability and endurance characteristics.

The obtained fatigue graphs also indicated fluctuations in stress response during cyclic loading, which may be attributed to internal layer interaction, reinforcement distribution, and localized stress concentration within the printed structure. The fatigue behavior observed during testing highlights the influence of additive manufacturing parameters and internal structural configuration on the endurance characteristics of PLA–MOF composite specimens.

4.3 Effect of infill density on mechanical performance

The mechanical performance of the fabricated PLA–MOF composite specimens was significantly influenced by infill density during both tensile and fatigue testing. Infill density affected the internal structural continuity, material distribution, and void concentration within the printed specimens, thereby influencing their load-bearing behavior and cyclic response.

During tensile testing, the 50% infill specimen demonstrated the highest tensile strength among the tested configurations, indicating improved load distribution and internal structural stability. The 25% infill specimen exhibited comparatively lower tensile performance because of reduced material continuity and increased internal void presence.

Fatigue testing also showed that specimens with higher infill densities demonstrated comparatively improved cyclic stability and endurance behavior under repeated loading conditions. The results indicate that optimization of infill density plays an important role in improving the mechanical behavior of PLA–MOF composite specimens fabricated using extrusion-based additive manufacturing.

4.4 Processing and fabrication observations

The solvent-assisted composite preparation process demonstrated that Dichloromethane (DCM) provided effective dissolution of PLA and enabled preparation of a processable polymer solution suitable for reinforcement incorporation. The use of hexane during non-solvent precipitation enabled successful recovery of the composite material in solid form for further processing.

During reinforcement incorporation, localized ⁹⁵particle agglomeration was observed during certain stages of mixing, indicating the importance of controlled stirring and reinforcement distribution during composite preparation.

Pellet extrusion-based additive manufacturing using the Piocreat G5 Ultra system demonstrated the feasibility of directly processing the prepared PLA–MOF composite feedstock without intermediate filament fabrication. The fabricated specimens exhibited acceptable extrusion behavior, layer formation, and dimensional consistency suitable for mechanical evaluation.

CHAPTER 5

65 CONCLUSION AND FUTURE SCOPE

This chapter presents the major conclusions drawn from the present study along with the limitations of the work and recommendations for future research related to PLA–MOF composite development for additive manufacturing applications.

5.1 Conclusion

The present study investigated the development and evaluation of Fe-MOF reinforced PLA composites for extrusion-based additive manufacturing applications. A solvent-assisted composite preparation methodology involving PLA dissolution using Dichloromethane (DCM) and non-solvent precipitation using hexane was successfully implemented for composite fabrication.

The prepared PLA–MOF composite material was processed using pellet extrusion-based additive manufacturing through a Piocreat G5 Ultra system to fabricate ASTM-standard tensile and fatigue specimens. Mechanical testing was carried out to evaluate the tensile and cyclic loading behavior of the fabricated specimens with varying infill densities.

The experimental results demonstrated that infill density significantly influenced the mechanical behavior of the printed composite specimens. The fabricated PLA–MOF composite specimens exhibited acceptable printability, extrusion behavior, and structural consistency during additive manufacturing.

The study demonstrates the feasibility of developing and processing Fe-MOF reinforced PLA composites using solvent-assisted processing and pellet extrusion-based additive manufacturing techniques for mechanical evaluation and further research applications.

5.2 Limitations of the present work

The present study was primarily focused on the development and preliminary mechanical evaluation of Fe-MOF reinforced PLA composites for extrusion-based additive manufacturing applications. Although successful fabrication and testing of composite specimens were

achieved, ⁷⁰ certain limitations were identified during the course of the work.

¹² Advanced material characterization techniques such as Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), ⁵⁶ Fourier Transform Infrared Spectroscopy (FTIR), and

thermal ¹³⁰ analysis were not carried out ⁵⁶ within the scope of the present study. In addition, challenges related to reinforcement dispersion, localized particle agglomeration, and extrusion consistency were observed during composite preparation and fabrication.

The study was also limited to a specific reinforcement concentration range, selected infill density configurations, and a limited number of fabricated specimens for mechanical evaluation. Further process optimization and material characterization are therefore required for a more comprehensive understanding of the developed composite system.

5.3 Future scope

²² The present study establishes the feasibility of developing Fe-MOF reinforced PLA composites for extrusion-based additive manufacturing applications; however, several areas remain available for further investigation and optimization.

Future work can focus on ¹² advanced material characterization techniques such as Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), ⁵⁶ Fourier Transform Infrared Spectroscopy (FTIR), Differential Scanning Calorimetry (DSC), and Thermogravimetric Analysis (TGA) to better understand reinforcement dispersion, interfacial bonding, thermal stability, and structural behavior of the developed composite system.

⁹⁴ Further studies can also investigate the influence of different Fe-MOF concentrations on mechanical performance, extrusion behavior, and printability. Optimization of reinforcement loading and particle dispersion techniques may help reduce agglomeration and improve structural consistency of the fabricated specimens.

Additional research may be carried out using alternative polymer matrices such as Nylon, PETG, ABS, or Polyamide for improved mechanical and thermal performance. Comparative studies between filament extrusion and pellet extrusion-based additive manufacturing systems may also provide further understanding of process compatibility and material behavior.

The implementation of alternative additive manufacturing ⁷⁰techniques such as Selective Laser Sintering (SLS) can also be explored for improved reinforcement distribution and enhanced structural uniformity of MOF-based composite systems.

Future investigations may additionally focus on:

- Optimization of printing parameters
- Thermal and impact testing
- Long-term fatigue evaluation
- Moisture absorption studies
- Wear and tribological analysis
- Development of lightweight structural components for engineering applications.

The use of recycled PLA feedstock in combination with reinforcement materials may also be further explored to support sustainable and environmentally conscious additive manufacturing practices aligned with circular manufacturing and material reuse objectives.

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