Chapter 02. Literature review

2.1 Preamble

This chapter reviews the use of CFF as reinforcement in composites. A systematic literature review of existing literature is carried out to present the appropriateness of CFF as reinforcement in composites. A summary of various methods used for manufacturing CFF reinforced composite is also presented to understand the research gap.

2.2 Introduction to Fibers

Fibers used in the composite are mainly of two types: natural and synthetic. Natural fibers include fibers derived from animals and plants. Plant fibers have short growth cycles and they include fibers derived from seeds, bast, leaves, and pericarp of fruits. Animal fibers are usually the outer protective layer of their body which include hair, feathers, skin, and exoskeletons like shells. The major benefit of natural fibers is their high strength-to-weight ratio (Karimah et al., 2021). The other favorable parameters are low density and high stiffness. Also, the manufacturing cost is very low. Figure 2-1 presents the classification of fibers.

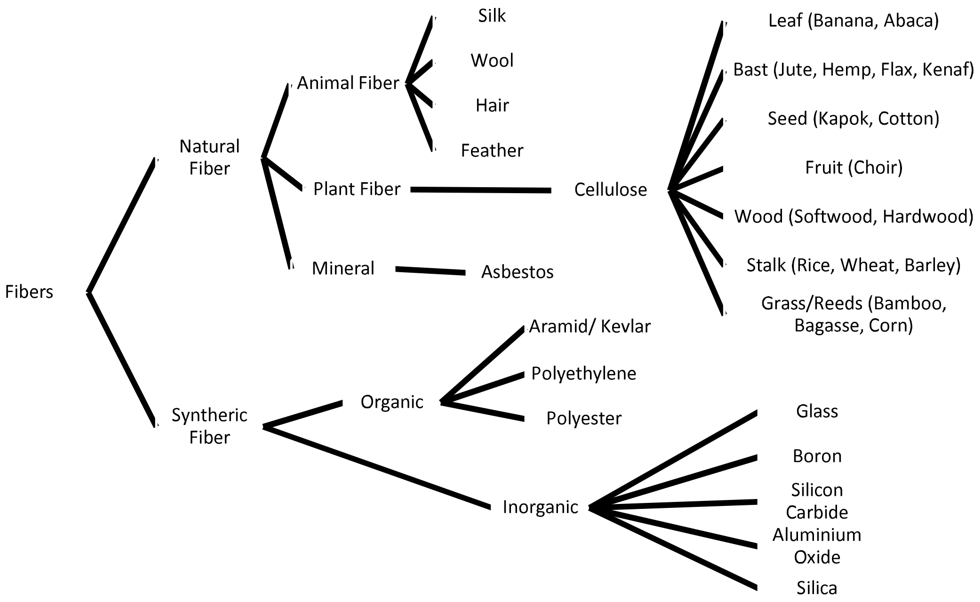


Figure 2-1 Classification of Fibers.

The majority of the plant-based fibers are cellulose based while the animal fibers are keratin based. The annual consumption of poultry has increased significantly in recent decades from

450 Metric kilotonnes to 900 Metric kilotonnes (Hannah Ritchie and Roser, 2021) at an average increase of 3.5% annually. Annual consumption is shown in Figure 2-2.

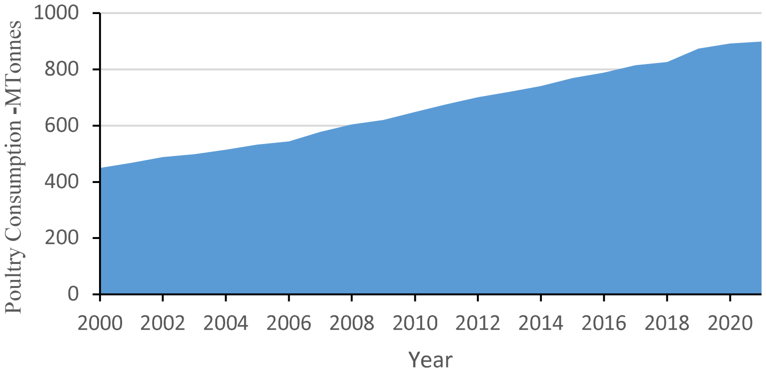


Figure 2-2 Annual poultry consumption worldwide(Hannah Ritchie and Roser, 2021).

The feathers amount to an average of about 6-7 % of the total weight of the chicken (Solcova et al., 2021) forming about 54-63 metric kilotons of chicken feathers and increasing to 1.7 metric kilotons annually. Currently, disposal of waste disposal is a major concern for municipalities and industries (Che et al., 2013). Existing methods of hydrolysis, incineration, animal feed, and energy production (C. M. Williams, 2013) are not enough for increasing chicken feather waste disposal. This will lead to large areas of landfills being occupied with chicken feather waste disposals. Also, the existing methods utilize huge amounts of resources including time, energy and equipment.

Converting chicken feathers into sustainable and environmentally compatible solutions is one of the key research areas. CFFs are environmentally compatible, cytotoxic, lightweight, low density and available at almost no cost. It is required to characterize morphologically, physically, chemically, mechanically, thermally and electrically for finding alternate methods to manage and consume chicken feathers.

Chicken feathers are presently managed by incineration, composting in the presence of keratinolytic fungi (Ardyati et al., 2019), methane production by anaerobic digestion (Forgács et al., 2011), protein additive in chicken meals(Shih, 1993), making non-woven fabrics (Evazynajad et al., 2002), hydrolysis (Solcova et al., 2021), etc. this methods are not sufficient to manage them completely (Muduli et al., 2019). Compared to other methods, utilizing chicken feathers by hydrolysis has less environmental impact but scaling the process is an issue(Adhikari et al., 2018; Solcova et al., 2021; Staroń et al., 2014). It is reported about manufacturing biodiesel from used chicken fat (Kirubakaran and Selvan, 2018), generates peptone resources for bacterial growth (Taskin and Kurbanoglu, 2011), as a soil amendment for better cultivation growth (Islam et al., 2021), as bio-fertilizers (Bhari et al., 2021). Reviews are presented for the use of Chicken feathers as reinforcement in composites (Adil et al., 2023; Khan et al., 2022).

Biocomposites, a recently developing area in the field of materials are explored to use natural fibers as reinforcement (Ho et al., 2012). Researchers have reported the use of various natural fibers as reinforcement with epoxy resins as matrix materials(Maldas, Kokta, and Daneault, 1989; Schneider et al., 1995; G. I. Williams and Wool, 2000; Saravana Bavan and Mohan Kumar, 2010; Gope, Singh, and Rao, 2015;). The composites are reported to have been utilized in civil engineering, and biomedical applications (Sadiku et al., 2019; Ibrahim et al., 2019; Kupolati et al., 2019). Keratin, a derivative of avian feathers has also been reviewed for appropriateness for various applications (W. Schmidt and Barone, 2004). Chicken feather has been reviewed as reinforcement in composite manufacturing(Bansal et al., 2017; Khumalo et al., 2019; W. F. Schmidt and Jayasundera, 2004; Tesfaye, Sithole, and Ramjugernath, 2017).

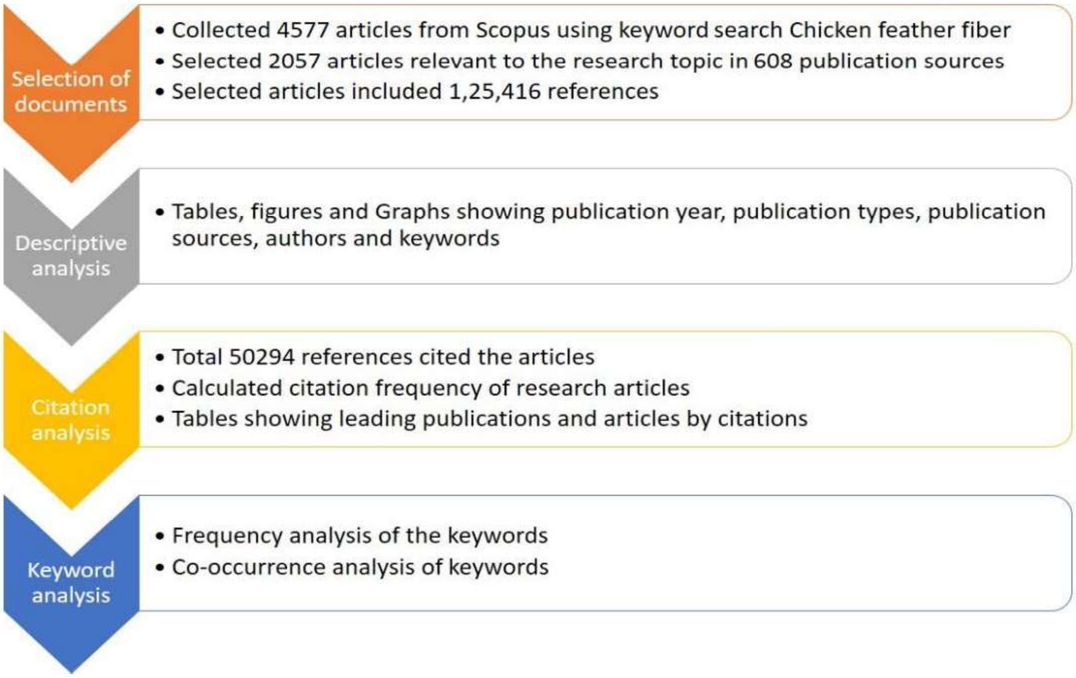
For a better understanding of utilizing chicken feathers, exploratory research of the existing research is carried out. The exploratory research is carried out in two steps

1. Research that provides answers to who, where, when and what. (Bibliometric studies)
2. Research to summarize the content and find paths for future research (state of art literature review)

2.3 Bibliometric studies

Bibliometric studies can be used to analyze and quantify the growth of a particular research area (J. Tao et al., 2020). Research performance can be evaluated using bibliometric studies (Wallin, 2005). Bibliographic databases provide useful indicators for finding the relevance of the field under study(Hood and Wilson, 2003). The bibliometric study is useful to analyze huge amounts of scientific data in a meaningful way. The visualization of similarities used in bibliometric analysis provides an easy method of understanding the analyzed data (Van Eck and Waltman, 2010, 2017). The systematic approach followed in the study is the one proposed by Tranfield (Tranfield et al., 2003).

The Scopus database was searched with the keywords ‘chicken feather fiber’ and associated keywords. The search resulted in 4577 articles. Filtering for relevant keywords in engineering, a database of 2057 articles was considered for evaluation. The keywords are presented in Table 2-1. A descriptive analysis based on publication year type, source, authors and keywords was carried out. The analysis is represented in the form of tables, figures, and graphs for better understanding. The research methodology adopted for the bibliometric studies is presented in Figure 2-3.



- Collected 4577 articles from Scopus using keyword search Chicken feather fiber
- Selected 2057 articles relevant to the research topic in 608 publication sources
- Selected articles included 1,25,416 references

- Tables, figures and Graphs showing publication year, publication types, publication sources, authors and keywords

- Total 50294 references cited the articles
- Calculated citation frequency of research articles
- Tables showing leading publications and articles by citations

- Frequency analysis of the keywords
- Co-occurrence analysis of keywords

Figure 2-3 Research methodology for Bibliometric studies.

The entire Scopus database was searched for the keywords, considering the following categories:

Table 2-1 Categories for keywords searched in the Scopus database.

Category	**Keyword**	
Material	Chicken, Chickens Poultry, Feather Natural Fibers, Chicken Feather, Chicken Feathers, Poultry Feathers, Chicken Feather fiber, Keratin, Keratins, Protein,	
Tests	**Mechanical**	Mechanical Properties Tensile Strength, Tensile Testing Bending Strength Impact Strength Dynamic Mechanical Analysis Elastic Moduli Compressive Strength

	Thermal	Thermal Properties, Thermodynamic Stability, Thermodynamic Properties, Glass Transition Temperature, Thermal Conductivity Dielectric Losses
	Physical	Thermogravimetric Analysis, Scanning Electron Microscopy Fourier Transform Infrared Spectroscopy, Differential Scanning Calorimetry
	Behaviour	Water Absorption, Particle Size, Porosity, Crystallinity, Adsorption
Composite Applications	Composite Materials Reinforcement, Composites, Feathers, Reinforced Plastics, Biopolymers, bio-composites, Biocompatibility, Biodegradable Polymers, Fillers, Polymer Matrix Composites, Fiber Reinforced Plastics, Polymer, Hybrid Biocomposite, Polymer Composite	
Processes	Carbonization, Hydrolysis, Pyrolysis, Extraction, Degradation Grafting (chemical), Compression Molding, Enzyme Activity Isotherm,	
Application	Wastewater Treatment, Waste Management, Tissue Engineering, Pollutant Removal, Textile Industry, Medical Applications, Water Pollutant, Water Pollutants, Chemical, Thermal Insulation, Environmental Impact	

The search parameters resulted in records from 1976 to 2022. Figure 2-4 indicates an increasing trend in publication count post-2013. From 2013 to 2021 a significant increase in the number of publications is observed. This indicates the increasing importance of CFF research. The best fit trendline indicates the number of published CFF articles, as exponential.

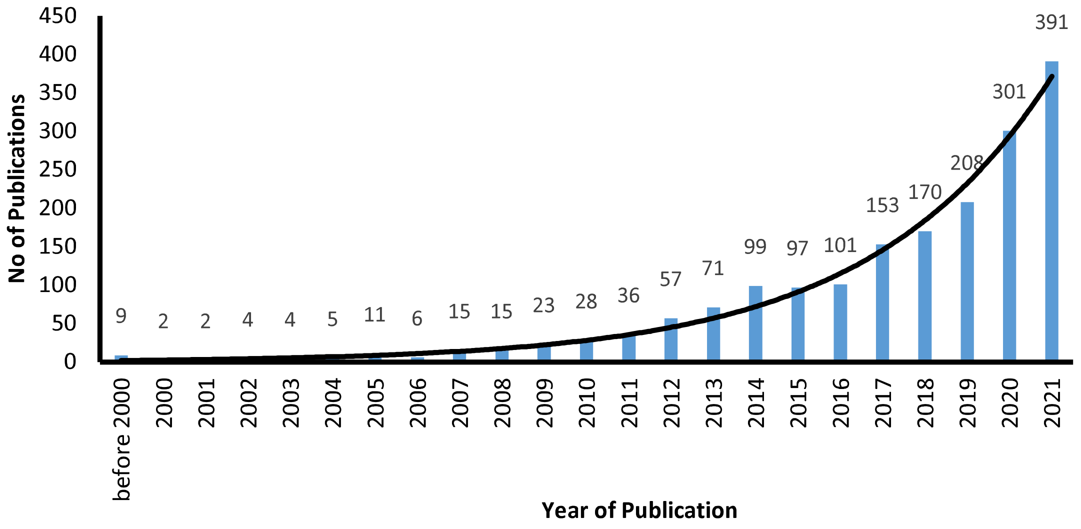


Figure 2-4 Annual number of publications.

The forecast model is prepared with a confidence level of 80% and exponential smoothing. The smoothing coefficients are Alpha (0.75), Beta (0.75), Gamma (0), and the mean absolute scaled error (MASE) (3.93), symmetric mean absolute percentage error (SMAPE) (0.15), mean absolute error (MAE) (23.87), and root mean square error (RMSE) (30.06). The forecasting model was prepared for the period from 2021 to 2030. The forecast model indicates a rising interest and opportunity in CFF research. Figure 2-5 depicts the forecast model for the publication numbers till the year 2030.

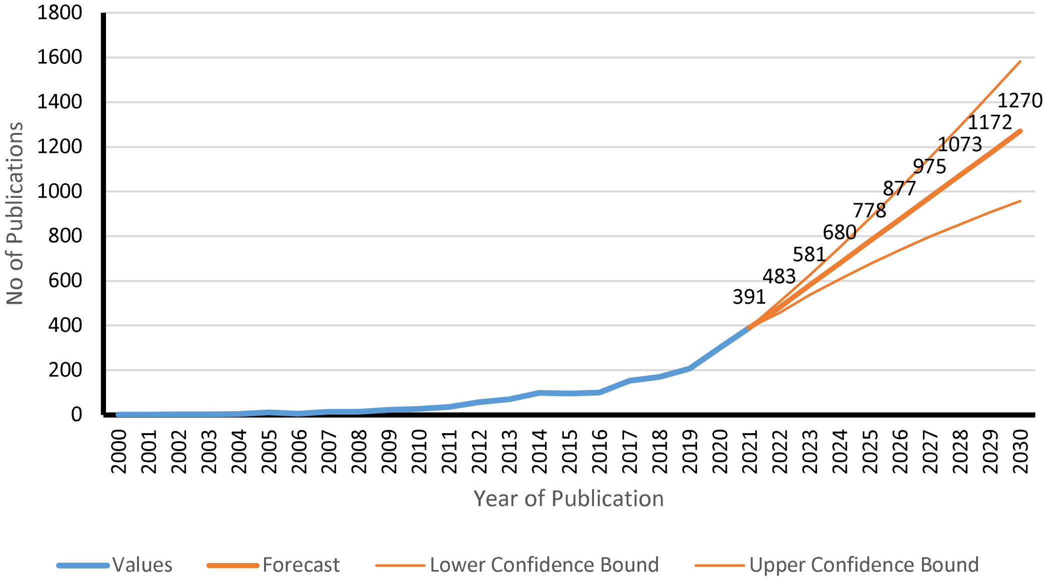


Figure 2-5 Forecast model for the number of publications for the decade 2021-2030.

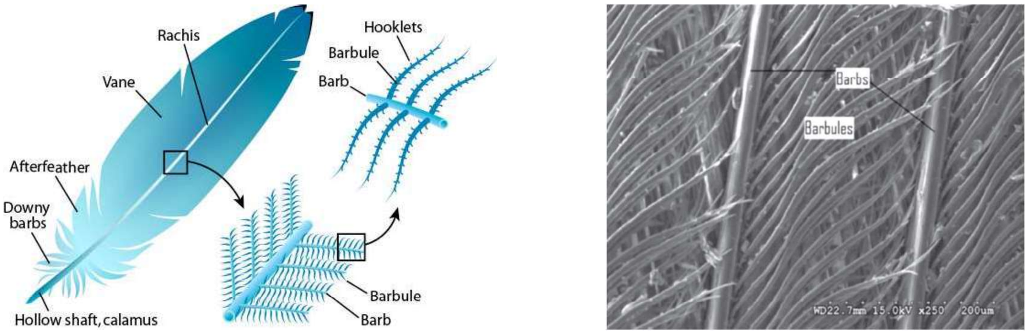
The majority of the research was published in the form of research articles, reviews and conference papers. 76% of the publications were in the form of research articles. The majority of the research papers were reported in the Journal of Polymers and the Journal of Applied Polymer Science. The publications indicate that the directions investigators have pursued are towards material science (about 21.4%) and second in the field of composites technology (18.3%). Chinese and Indian researchers have been the major contributors, to investigating solutions for alternate use or characterization of the chicken feather. Yang Y and Wang Y of China are the investigators who have produced maximum publications. The research carried out by Imran Ali and his co-researchers on using chicken feathers for the removal of organic pollutants from wastewater is one of the most cited publications and is presented in the Journal of Environmental Management (Ali et al., 2012).

2.4 State of Art Literature Review

After a bibliometric review of the literature, the contents have been reviewed. A state of art literature survey of the CFF and their composites is presented.

2.4.1 Fiber characterization

A feather consists of two parts: the central shaft (rachis) and the vanes. Rachis consists of a hollow tube like structure with honeycomb foam called a medulla. The end of the central shaft which is connected to the skin of the chicken has a denser honeycomb structure called the calamus. Vanes are formed by interlinked hooks called barbs, which are further divided into barbules as shown in Figure 2-6-a (Wang et al., 2016). Similar features of chicken feathers are observed under a microscope by NIKON Eclipse LV100ND as shown in Figure 3-4. The hooks at the end of barbs and barbules entangle with each other, hence enhancing the bulking properties as shown in Figure 2-6-b (Reddy and Yang, 2010; W. F. Schmidt, 1998).



a. b.

Figure 2-6 a. Feather morphology b. SEM of Chicken feathers (Reddy and Yang, 2007).

The internal structure of chicken feather is hollow (Hong and Wool, 2005). With an increase in phenolic concentration, temperature, and pH the adsorption properties of feathers improve (Banat and Al-Asheh, 1999). Chicken feathers are one of the very few natural fibers which exhibit hygroscopic, hydrophilic and hydrophobic nature (Khosa et al., 2013; Misra et al., 2001; Saucedo-Rivalcoba et al., 2011). Chicken feathers consist of (~60%) hydrophobic amino acids and fats (Barone and Gregoire, 2006; Saravanan and Dhurai, 2012) while the remaining (~40%) amino groups are hydrophilic (Arai et al., 1986; Gokce et al., 2017). The dual nature is responsible for the enhanced adsorption and biosorption properties of chicken feathers.

Chicken feathers contain 90% proteins (keratins). The feather contains aspartic acid (~5%), glutamic acid (~7%), arginine (~5%), proline (~12%), glycine (~11%), alanines (~8%), cysteine (~7%), valine (~9%), leucines (~11%), and serine (~4%) (Arai et al., 1986; Martínez-Hernández et al., 2005; Tesfaye, Sithole, Ramjugernath, and Chunilall, 2017).

The tensile strength of chicken feathers is reported as 23.9 g/tex, moisture regained 12%, breaking extension 1 to 6%, and density is 0.8 to 1.12 g/cc.(Reddy and Yang, 2007; Saravanan and Prakash, 2020). The mean tenacity of the chicken feather was observed at 16.93 cN/tex, while the mean elongation at maximum load was 0.48mm (Tesfaye et al., 2018). The Young's modulus of keratin from birds is mean around 1.78 GPa. The flexural stiffness of the whole rachis is dependent on the cross-sectional morphology (Bonser and Purslow, 1995; Cameron, Wess, and Bonser, 2003). The tensile modulus of CFF was 3.59±1.09 GPa, and the average tensile strength was 203±74 MPa. Failure follows the Weibull distribution with a scale parameter value of 178.7 MPa and a shape parameter value of 2.32 (Zhan and Wool, 2011).

The chicken feather undergoes three different stages when subjected to heat. The first stage is observed between 43-145 °C where it starts losing moisture. The second stage is observed between 230-275 °C, where the entire moisture is removed and the feather starts blackening (W. F. Schmidt and Line, 1996). The third stage is observed between 280 °C to 340 °C. In the third stage, the total decomposition of the chicken feather occurs. The decomposition of the crystalline structure of the chicken feather is observed in the second stage, i.e., between 230-275 °C. This is indicative that the drying temperature for CFF should be above 100 °C and below 125 °C. The processing temperature of CFF composites should be maintained below 230 °C (Martínez-Hernández et al., 2005; W. F. Schmidt and Line, 1996; Tesfaye et al., 2018).

2.4.2 Chicken Feather Fiber composite

Literature reviews have been carried out to characterize chicken feathers and prove their suitability as reinforcement in composites (Alonso et al., 2013; Bansal and Singh, 2016; Jagadeeshgouda et al., 2014). The focus of the literature review was on the matrix material, the effect of fiber length on composite characteristics and methods of manufacturing.

2.4.2.1 Matrix material

Research has been reported to mention the optimal mix of CFF with various construction materials such as cement, plastering mortar, asphalt, and clay. Availability and low density have been the primary reason for utilizing CFF (Acda, 2010b; Adetola et al., 2014; Cao et al., 2013; Carrillo et al., 2013; Dalhat et al., 2020; Hamoush and El-Hawary, 1994; Mendoza et al., 2021; Sharma, 2016; Tesfaye, Sithole, Ramjugernath, Xu, et al., 2017; Xu et al., 2017; Zaimouglu et al., 2016).

There are research articles describing the use of processed Soya bean oil (AESO, MESO, etc.) resin as matrix material and CFF as reinforcement. They concluded that a reduction of the coefficient of thermal expansion, dielectric constant, and density is observed with the addition of CFF. While the storage modulus, fracture toughness, fracture energy, and electrical resistivity test results indicate an increase. These properties are comparable to commercially used PCB base materials (Dweib et al., 2004; Gogoi et al., 2019, 2022; Hong and Wool, 2005; Zhan and Wool, 2013a).

Various thermosetting polymers such as polyester, phenylester, Formaldehyde, Styrene-Butadiene copolymer, Epoxy resin, etc. have also been investigated as matrix materials with CFF. In the research, the tensile, flexural and impact strength increases to the peak value of 8-10 % weight fraction before dropping gradually (Álvarez-del-Castillo et al., 2022; Borazan and Gokdai, 2017; Choudary et al., 2018; Gokce et al., 2017; NagarajaGanesh and Sugumaran, 2012; Subramani et al., 2014; Uzun et al., 2011). Chicken feathers have been mixed with thermoplastics such as HDPE, LDPE, Polyvinyl Chloride, Thermoplastic polyurethane (TPU), Styrene Butadiene copolymer (SBS) upto 20 wt.% loadings. The addition of coupling agents or plasticizers such as PEgMAH increases the interfacial bonding between matrix and fibers and increases the tensile strength and thermal stability(Barone and Gregoire, 2006; Barone and Schmidt, 2005; Borazan and Gokdai, 2017; Choudary et al., 2018; Huda and Yang, 2008; Janowska et al., 2013; S. C. Mishra and Nayak, 2010; Moechnig et al., 2003; NagarajaGanesh and Sugumaran, 2012; Salehuddin et al., 2014; Srivatsav et al., 2018; Subramani et al., 2014; Uzun et al., 2011).

CFF and Polylactic acid (PLA) presents a good environment-friendly composite alternative. The elastic moduli and stiffness of CFF/PLA were higher than PLA for loading upto 5 wt.%. Compressive strength, hardness and flexural strength of the CFF/PLA composites were greater than PLA while the bending and impact strength was lower. The storage modulus also improves for CFF/PLA composites. There is a slight increase in the melting temperature of composites while the crystallinity decreases with an increase in CFF content (Akderya et al., 2020; Amaya-Amaya et al., 2021; Aranberri et al., 2017; Cheng et al., 2009; Li et al., 2010; Na Ayutthaya and Wootthikanokkhan, 2013; Özmen and Baba, 2017; Sanchez-Olivares et al., 2017).

2.4.2.2 Effect of fiber length.

CFF composites with short fiber have lower compressive modulus compared to those with long fiber. Composites with 5% long chicken fiber content are reported to have maximum impact strength about 23% more as compared to short fiber content. With an increase in fiber content, the impact strength deteriorates significantly for long fiber as compared to short fiber composites (Baba and Özmen, 2017).

The low aspect ratio and the intrinsic structure of CFF are responsible for its weak mechanical strength if cut into short fibers, while long fibers give higher strength. Short fiber is easy to process and requires lower manufacturing costs, so it is used widely in composite manufacturing. (Zhan and Wool, 2016)

2.4.2.3 Methods of Manufacturing

Composites are manufactured using various methods. The first reported use of chicken feathers as reinforcement is with cement as matrix material (Hamoush and El-Hawary, 1994). Solution moulding was used in this research. Figure 2-7 shows methods utilized for manufacturing CFF reinforced composites as reported in various research papers.

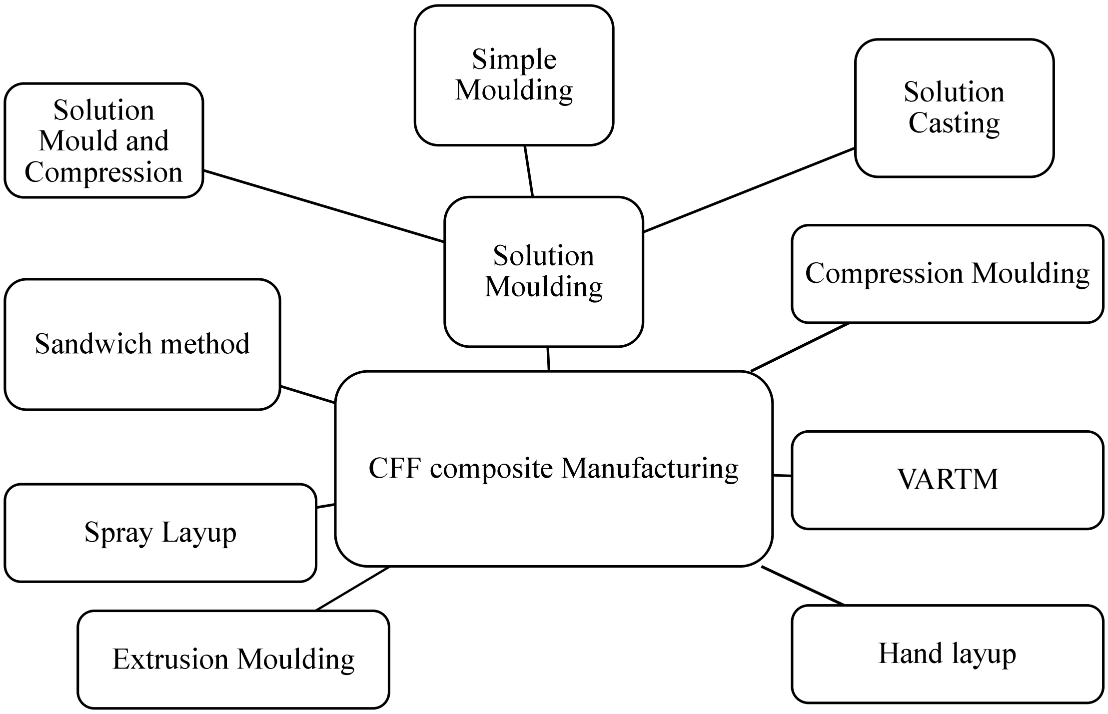


Figure 2-7 Various methods utilized for manufacturing CFF reinforced composites.

Various methods employed for manufacturing CFF reinforced composites are discussed in the subsequent sections.

- *Hand layup*

Hand layup is one of the oldest methods to manufacture composites. In this method, first, the mould is prepared by applying mould release spray and laying a thin sheet on it. The first layer of polymer mixed with the required additives is placed on the mould. CFF is then placed on this polymer layer to cover the entire mould. The layer is pressed with light pressure using a hand roller to ensure the complete soaking of the entire chicken feather in the polymer and the removal of any air trapped in between the layers. The process is repeated multiple times depending on the thickness of the component required (Kumar et al., 2020; Manral, Gariya, Bansal, Singh, and Negi, 2019; Mouzakis, 2012; Sah et al., 2020; Stalin et al., 2015). The schematic of the process is shown in Figure 2-8.

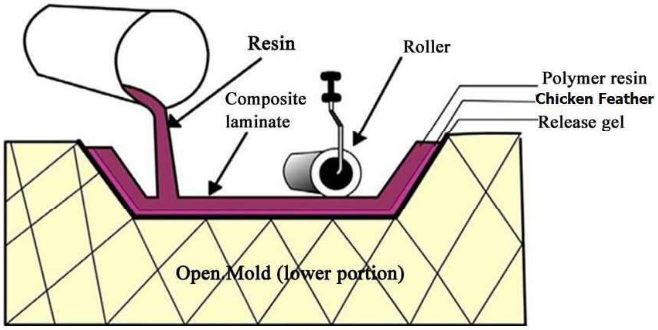


Figure 2-8 CFF composite preparation by Hand layup technique (Manral, Gariya, Bansal, Singh, and Rawat, 2019).

- *Compression moulding*

In compression moulding, chicken feathers, thermosets and thermoplastics are mixed and put inside a symmetrical mould. the mixture undergoes compression in a press at high pressure. The adhesion between the chicken feather and the matrix material is because of the plastic deformation of the matrix material under pressure (Álvarez-del-Castillo et al., 2022; Ghani et al., 2013; Moechnig et al., 2003; Oladele et al., 2014; Saravanan and Prakash, 2020; Winandy et al., 2007; Zhan and Wool, 2013b).

- *Extrusion Moulding*

The mixture of CFF and the matrix material is mixed and passed through a hot screw and barrel system and the mixture is pushed through a nozzle into a mould to form the component or pass it through a water bath to form filaments (Amieva et al., 2015; Leonor Mendez-Hernandez et al., 2018; Lucio et al., 2017).

- *Spray layup*

A mixture of resin and chopped CFF is sprayed using a hand gun on the mould surface in spray layup technique 10,11. This method is utilized for manufacturing large composite components (Reddy et al., 2014). The working principle is shown in Figure 2-9.

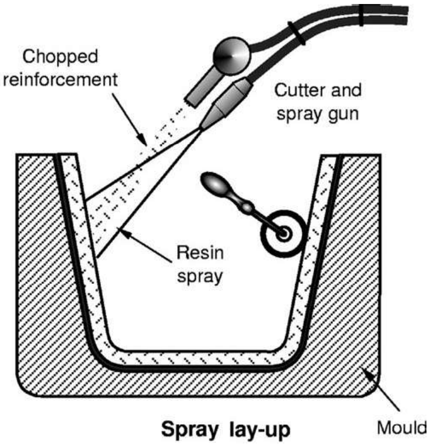


Figure 2-9 Spray layup technique for manufacturing composites (Swift and Booker, 2013).

- *Sandwich method*

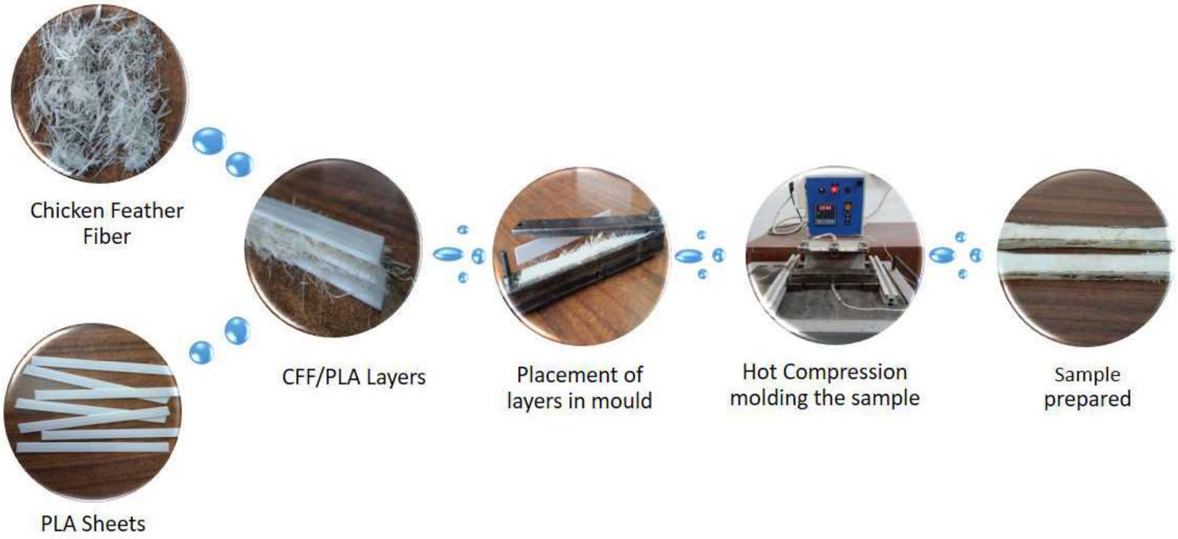


Figure 2-10 Process of preparing CFF/PLA samples using the Sandwich method (Adil et al., 2023).

In this method, the chicken feather is sandwiched between sheets of thermosets or thermoplastics. The sheets can be prepared using 3D printers also. The sandwich materials are then hot pressed at a temperature below the melting temperature of the matrix material and held under pressure for a predetermined time, depending on the thickness and layers of sheets as shown in Figure 2-10 (Adil et al., 2023). The method can be utilized for manufacturing open shapes which can be 3D printed.

- *Vacuum Assisted Resin Transfer Molding Process (VARTM)*

The resin is drawn into a vaccum sealed bag containing CFF. The resin is distributed evenly because of the partial vaccum. The process is shown in Figure 2-11. The mechanical properties of VARTM CFF reinforced composites are better, as the presence of void is reduced drastically. The process is used to manufacture large structural components (Dweib et al., 2004; Hong and Wool, 2005; Lucio et al., 2017; Senoz et al., 2013; Tamakuwala, 2021).

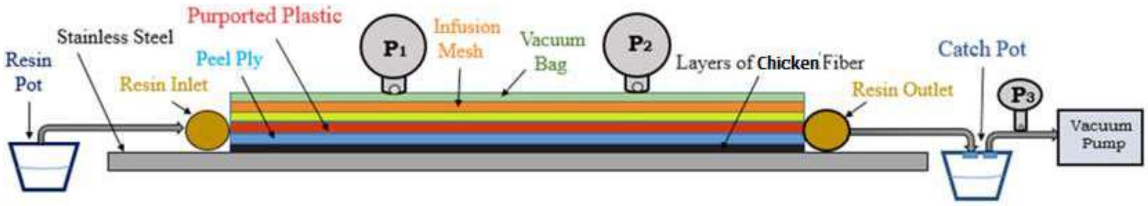


Figure 2-11 Line diagram of Vacuum Assisted Resin Transfer Molding Process (VARTM) (Gajjar et al., 2020).

- *Solution moulding*

The matrix material and the chopped CFF are filled in a container and thoroughly mixed till a homogenous solution is obtained. The solution is then poured into a mould to obtain the required shape. For uniform cross-section components like pipes, the process is carried out with constant cooling to obtain the casting. For sheet like components, the process is followed by compression to obtain uniformity in thickness (Barone and Schmidt, 2005; Borazan and Gokdai, 2017; Dalhat et al., 2020; Das et al., 2018; Gogoi et al., 2019; Gokce et al., 2017; Janowska et al., 2013; Jimenez-Cervantes Amieva et al., 2015; Srivatsav et al., 2018).

- *Fiber laying during 3D printing*

In this method, after each layer of printing, fibers are laid on the printed part, and the next layer is printed on it. The process is repeated to get the desired shape and the number of layers is decided based on the thickness to be achieved. This process can be helpful to generate complex shapes (Amaya-Amaya et al., 2021).

Few matrix materials have been used frequently in CFF reinforced composites. The most widely used manufacturing techniques for various matrix materials are mentioned in Table 2-2. Research papers, it is indicated that resin-based matrix have been utilized most frequently.

Table 2-2 Widely used manufacturing techniques for various matrix materials in CFF reinforced composites.

Matrix Material	**Method of Manufacturing**
Soybean oil/AESO	VARTM / Solution Moulding
Epoxy based resins	Hand layup / Solution moulding

Polyester based resins	Hand layup / Solution moulding/ Hot press moulding/ Compression moulding
Thermosets like HDPE/ LDPE/ PLA/ TPU	Compounding and compression moulding/ extrusion and injection moulding/ spray layup
Construction materials like Cement, mortar, clay asphalt	Hand layup / Solution moulding

The major limitations of these composite manufacturing methods are:

- Complex shapes are difficult to manufacture.
- The tooling cost is high, if the no of parts manufactured is very less.
- Adhesion failure between layers due to manual mismatch.
- Avoiding bubbles, air entrapment and unwetted interface requires utmost care.
- In these methods, bulk solidification/vitrification occurs, which requires a long duration of curing.
- In the case of manual methods, the homogeneity/ consistency of layer height is dependent on the stability of the person's hand, which sometimes causes the mechanical characterization unpredictable.

2.4.3 3D printing of composites

3D printing is a technology, which is now being explored to overcome the limitations of manufacturing composites with existing techniques. Three dimensional (3D) printing is a technology to manufacture material structures through a layer-by-layer approach (Hwang et al., 2015). Additive manufacturing (AM) is defined as the process in which an object is produced by joining several layers with specific thicknesses using different bonding technologies (Wahlström & Sahlström, 2016; Yang et al., 2017). Additive manufacturing(AM) /3D printing process is based on extrusion, the material being "selectively dispensed through a nozzle or orifice"(ISO / DIS 17296-1, 2015).

The parts manufactured by AM used to be primarily employed for prototyping and rapid tooling; however, their areas of application as finished components have broadened every year as the process more frequently meet the stringent requirements to deliver end-use products (Turner et al., 2014; Turner and Gold, 2015). The most common and well developed system for additive manufacturing is commonly known as fused deposition modeling (FDM). In this process, a heated thermoplastic is extruded through a heated die and selectively deposited into a stable, flat surface in layers. The deposited material bonds with the previous layer via glass transition bonding due to the heat input of the new materials (Turner et al., 2014; Turner and Gold, 2015). The layers are designed and converted into G-code directly from data via pre-processing software packages such as Simplify3D, Cura, etc. (Messimer et al., 2018). 3D printing is also called desktop fabrication where a structure is synthesized from its 3D CAD model. The 3D model is stored in a STL format and after that forwarded to a 3D printer. It can use a wide range of materials such as Polylactic Acid (PLA), Acrylonitrile Butadiene Styrene (ABS), and other composites as well. 3D printing is a rapidly developing and cost optimized form of rapid prototyping. The 3D printer prints the CAD design layer forming a real object. The 3D printing process is derived from inkjet desktop printers in which material is deposited by jets of the printing material, layer by layer as derived from CAD 3D data.

The term “3D printing” or “Additive manufacturing” refers to the process of building products by adding many very thin layers of material layer on top of layer. Additive manufacturing can trace its roots back to the 19th century, particularly in the fields of topography and photo sculpture (Matias and Rao, 2015). However, in a “Brief History of Additive Manufacturing and the 2009 Roadmap” by Beaman et al, they cite that in 1972 Ciraud released the first technology that truly represented today’s definition of additive manufacturing. Ciraud’s process is described as taking meltable materials and using a beam of energy to melt the material, thereby building a product by melting the layer on top of another layer (Matias and Rao, 2015).

The beginning of 3D printing is related to studies of photography, sculpting, and Landscape design, which took place in America. Much of the technology was not being developed until the mid-1980s. During this period, 3D printing was known as “Rapid Prototyping”. Chuck Hull, of 3D Systems Corporation, manufactured the first usable 3D printer (Patil et al., 2017). Later in the 80’s, Selective Laser Sintering (SLS) technology was synthesized by Dr. Deckard at the University of Texas during the commencement of the project being done by the Defense Advanced Research Projects Agency. In the 1990s, the technology was further improvised with the advancement of a method that uses UV light to solidify photopolymer, a highly viscous liquid material. In the 20th century, 3D printers were very expensive and were used to print several products. Most of the printers were owned by scientists and electronics groups for research and display. However, advancements in the area of 3D printing have allowed for the design of products to no longer be limited by complex shapes or colours (Durgun and Ertan, 2014).

Different types of additive manufacturing methods available are which use different principles for creating layers and building components (Gokhare et al., 2017; Kuznetsov et al., 2018).

- Stereolithography (SLA)
- Digital Light Processing (DLP)
- Selective Laser Sintering (SLS)
- Selective Laser Melting (SLM)
- Electronic Beam Melting (EBM)
- Laminated Object Manufacturing (LOM)
- Binder Jetting (BJ)
- Material Jetting (MJ)
- Fused deposition Modeling (FDM)

2.4.3.1 Fused Deposition Method (FDM)

The Fused Deposition method is the second most widely accepted method for additive manufacturing after Stereolithography. This method is majorly applied for manufacturing components using thermoplastics. Extrusion of a thermoplastic monofilament through a heated nozzle has presently become the most prevalent 3D printing technique (Kun, 2016).

It was developed by Scott Crump in the 1980s. In this process, a plastic or wax material is extruded through a nozzle that traces the part's cross sectional geometry layer by layer. Many times FDM is used synonymously with Filament fusion fabrication (FFF). The schematic of a typical FDM printer is shown in Figure 2-12.

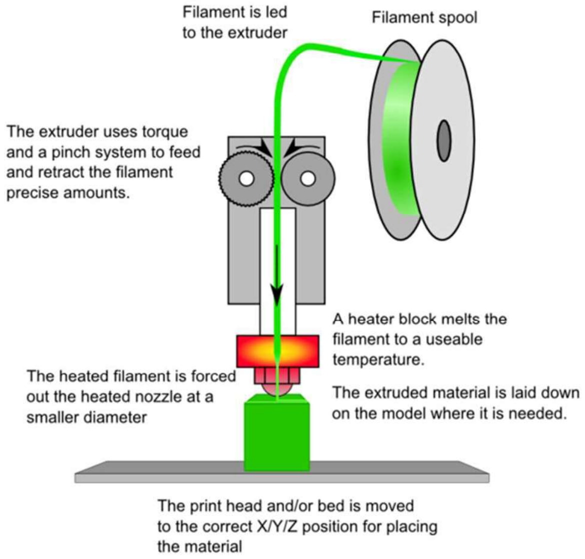


Figure 2-12 FDM/FFF Printing (Gokhare et al., 2017).

As shown in Figure 2-12, in FFF a plastic filament is unwound from a coil and supplies material to an extrusion nozzle. The nozzle is heated to melt the plastic and has a mechanism that allows the flow of the melted plastic to be turned on and off. The nozzle is mounted to an X-Y plotter type mechanism which traces out the part contours.

As the nozzle is moved over the table in the required geometry, it deposits a thin bead of extruded plastic to form each layer. The plastic hardens immediately after being squirted from the nozzle and bonds to the layer below. The object is built on a mechanical stage that moves vertically downward layer by layer as the part is formed. The entire system is contained within a chamber which is held at a temperature just below the melting point of the plastic. Support structures are automatically generated for overhanging geometries and are later removed by breaking them away from the object (Ramya and Vanapalli, 2016).

In “Basic and Advanced Materials for FDM.” (L. Novakova-Marcincinova and J. Novak-Marcincin, 2012), it was concluded that ABS and PLA thermoplastic are widely utilized in 3D printers and have enough availability in the market. In the majority of applications nowadays PLA is the first choice. PLA is favored for its biodegradability, absence of unpleasant odors when heated and for its overall environmental compatibility in all aspects of its life cycle. Additionally, PLA emits ten times fewer potentially dangerous ultra-fine particles (Tisserat et al., 2013) than ABS. The extruder designs found in commercial and hobbyist printers are mostly suitable for materials in filament form. While printing with filament is not a problem per se, the printing of materials that may not be readily available in filament form or not commercially viable remains untapped. e.g. Biopolymers and material blends (Whyman et al., 2018).

Generally, FDM operates by using an extruded thermoplastic filament and extruding it through a hot print head. This process is termed Fused Filament Fabrication(FFF). The limitation of FFF is that the raw material should be in filament form only (Popescu et al., 2018). The filament is usually made up of thermoplastics. For the ease of making the filaments, additives such as plasticizers are used. Composite filaments are manufactured with different blends of polymers and reinforcement materials. In the majority of the applications carbon fibers derived from natural resources (animal and plants) have been used.

Hwang carried out the thermo-mechanical characterization of metal polymer composite filaments (Hwang et al., 2015). They carried out a parametric study for the same in the FDM process establishing a procedure for carrying out the experimental study of similar type.

Ivey et al compared samples manufactured by 3D printing using PLA and PLA/Carbon fiber blends. They concluded that the tensile properties of the PLA and PLA/CF filaments showed increased elastic modulus because of the addition of carbon fibers (Ivey et al., 2017).

Gregory Mark (Mark, 2016) patented his work on embedding fiber reinforcement in moulded articles, to highlight that it is possible to embed fiber reinforcements in filaments and the same can be used to print complex shapes such as footwear.

Robles et al, (Domínguez-Robles et al., 2019) experimented by blending, biomaterials such as PLA and Lignin for 3D printing applications. The aim was to manufacture complex shapes using 3D printing for Healthcare applications. They concluded that, because of the versatility of the 3D printing process, using this blend would help hospitals to print wound dressings for patients on demand.

Agarwal et al., (Agarwal et al., 2018) from their experiments concluded that FFF using composite filament provides better design freedom and material properties compared to conventional FRP techniques. They concluded basis on their composite manufacturing of Epoxy Fiberglass fiber reinforced polymers with composite filament fabrication based Nylon-Fiberglass fiber reinforced polymers. The effect of process parameters on mechanical properties such as tensile strength, elastic modulus, and fatigue life were compared for the different processes.

Anuar carried out the thermal characterization of PLA- Kenaf fiber biocomposite and concluded that the addition of fiber enhances the thermal properties of components as well as the interfacial bonding is increased. The composite was prepared by using a twin extruder and injection moulding machine. The setup was similar to a twin extruder 3D printing machine (Anuar and Zuraida, 2011).

Mechanical characterization and their relationship with process parameters for biocomposites manufactured with PLA and fibers such as carbon fiber have been reported by Jiang et al (Jiang and Smith, 2017), Prashantha et al (Prashantha and Roger, 2017) and Touri et al (Touri et al., 2019) in their research papers.

The use of other 3D printing techniques for the bioprinting of tissues and organs has been reported in areas of biomedical Engineering (Bose et al., 2018; Guvendiren et al., 2016; Jammalamadaka and Tappa, 2018; Jose et al., 2016).

2.5 Filament Extruder

Rauwendaal described the process of extrusion of polymers. Polymer granules are to be heated at a temperature below their melting point before forcing it through a die or a nozzle in a shape as desired (Rauwendaal and Rauwendaal, 2018). For the extrusion of any material, the glass transition and the melting temperature are important to be known. Glass transition temperature is important when the physical properties of polymers are to be modified (Vrabič Brodnjak et al., 2017). Whelan et al in their handbook provided the background for the extrusion process of polymers (Whelan and Dunning, 1988). Krzysztof et al attempted to make a mathematical model for polymer extrusion (Krzysztof et al., 2019). Figure 2-13 shows the schematic of a generic single screw plastic extruder.

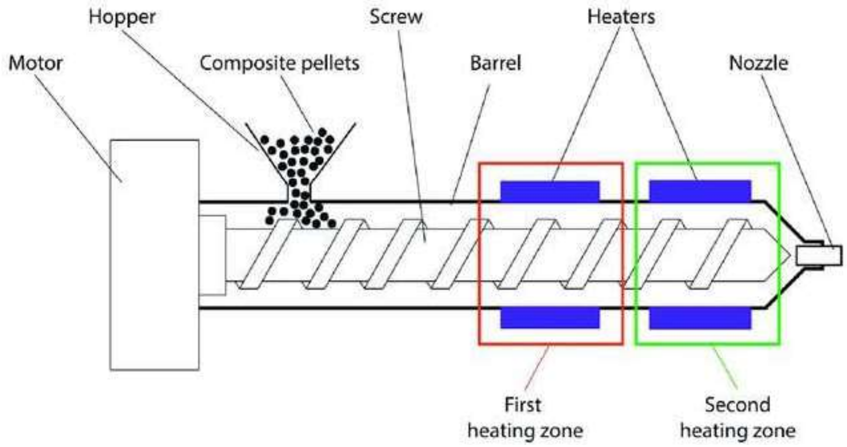


Figure 2-13 Schematic of a generic single screw plastic extruder (Podsiadły et al., 2021).

The material is forced through a die of the required dimension and cross-section to produce components with uniform cross-section. The material is fed into the hopper. After passing through the heated barrel, the material melts. The screw helps in the movement of the material ahead in the barrel. It also helps to create pressure to push the material out of the shaping die. A single extruder provides comparatively less shear between the barrel's inner wall and pallets and gives a very high throughput.

All single-screw extruders have a few common features. The main components to be designed or selected are an extruder screw, barrel, heaters for heating and melting the material, the motor-drive system for rotating the screw, and a control system for the heaters and motor speed. Single screw extruders can have a single-stage or multi-stage compression. Various zones of multi-stage extruder screws are presented in Figure 2-14.

Figure 2-14 Various zones of multistage extruder screws.

In the present design, multi-stage compression has been achieved by varying the root diameter of the screw. Multi-stage screws have three zones: the feed zone, the compression zone, and the metering zone (Womer et al., 2017). Figure 2-14 shows the various zones of multi-stage extruder screws. In the feed, section granules are preheated and compacted to feed forward. The compression zone however is the zone where granules start melting and compacting further. In the metering zone, the material will be homogenized and pushed through the die in the required quantity. The pressure required to pump out of the material from the die generate in this zone. In the case of polymers, the pressure required is proportional to the Melt Flow Index (MFI) of the material. For each zone, separate heater bands propose to have precise control of temperature in each zone. Sections of the screw design enhance the mixing of the matrix and fiber material. There are many aspects to be considered in extruder design that will influence the flow, level of mixing, and crystallization of materials. Figure 2-15 presents the typical temperature and pressure profile inside the extruder barrel as presented in various research (Yacu, 2020)

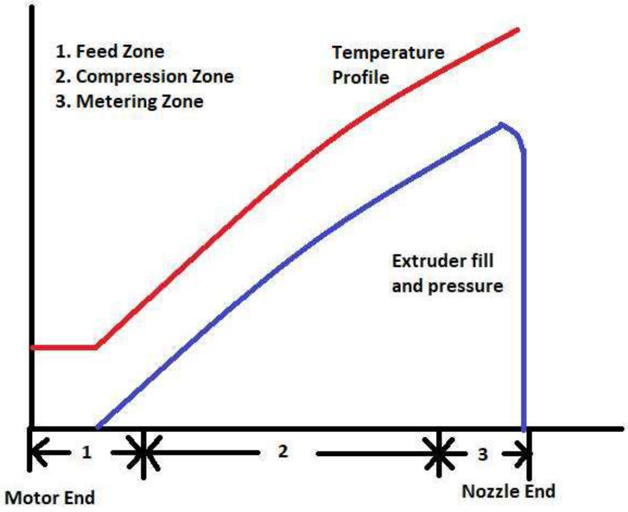


Figure 2-15 Typical temperature and pressure profile inside extruder barrel for single screw extruder.

In the feed zone, the clearance between the barrel and the screw root is maximum and constant. This results in constant pressure levels inside the zone. The channel depth is maximum to accept the pellets in a solid state. Constant pressure ensures the forward flow of material (Béreaux et al., 2009).

In the transition zone, the clearance between the barrel and the screw root decreases significantly, resulting in a pressure increase the clearance between the barrel and the channel depth is decreased because the pellets need to be melted and hence the pellets need to be near the heater bands. The shear force offered by the barrel aids in the melting of the pellets. (Gaspar-Cunha and Covas, 2001)

In the metering zone, the clearance between the barrel and the screw root is minimum. Hence, the pressure in this zone is maximum. Here, the melted granules and fiber material will face a change in the flow direction. For successful extrusion, a forward flow is desired to dominate the pressure. To tackle the backpressure, it is required to provide the screw with a torque that will result in forwarding flow. The pressure developed in the metering zone is the regulatory parameter for selecting a motor with torque dominating it.

The barrel is a simple hollow cylinder in which the screw rotates. The barrel acts as a barrier in such a way that the pellets are pushed forward. The barrel also helps in generating shear heating in the transition zone to soften the pellets. The barrel is generally heated, often with control of individual segments to precisely control the melting process of the polymer. The barrel conducts the heat from the heater band and supplies it to the required zones. The clearance between the barrel and the screw is kept in such a way that the material is easily forwarded to the feed zone. The barrel serves to maintain the flow pressure (Béreaux et al., 2007, 2009; Campbell et al., 2013; Chung and Chung, 2019; Krzysztof et al., 2019; Manideep et al., 2019; Y. Tao et al., 2019; Wilczyński, 1988, 1989).

For extrusion, external means of heating are required. The shear heating offered at the barrel and the screw interface is enough to soften the material but not enough to melt and mix the ingredient materials. Also, the temperature in each of the zone is to be kept different. This is achieved by covering the barrel with heater bands. Heater bands offer a conductive mode of heating via the barrel. Heater band selection is an important component of extruder design. There are majorly three materials available Mica, Ceramic and Mineral to be used as a heating element. Different materials provide different power densities. Based on the material to be extruded appropriate power densities i.e., watts of conductive heat per square meter are to be calculated. This calculation is the basis of heat band material selection.

The temperature profile in each of the zones is dependent on the type of material to be processed, its MFI, and its weight proportion. The temperature in the feed zone should be less than the lowest of the glass transition temperature of either material. In the transition zone, the temperature should be higher than the glass transition temperature and lower than the melting temperature. The temperature in the meter zone should be higher than the lowest melting temperature (Gaspar-Cunha and Covas, 2001).

The nozzle provides a converging path to the flow of the melted composite mixture and shapes it into a filament according to the desired dimension. The texture, expansion, and defects in the filament are dependent on the design of the nozzle. To obtain a filament with a uniform composition without porosity, the nozzle dimension should be appropriate. The nozzle dimension is selected based on L_n / D_n ratio. In the proposed design, the ratio is considered as 3. The ratio should be adequately determined as increasing the length can cause pressure build-up and a decrease would cause radial expansion of the filament (Ganjyal and Hanna, 2004).

The commonly used nomenclature for an extruder screw geometry is shown in Figure 2-16.

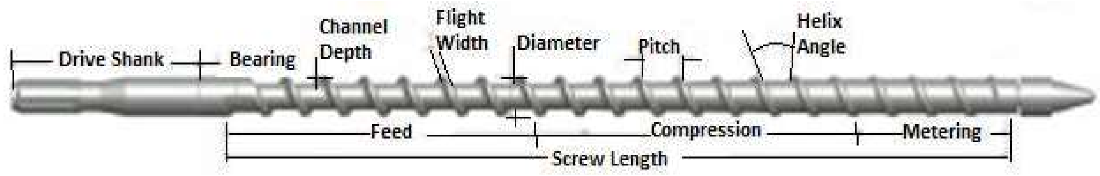


Figure 2-16 Nomenclature of extruder screw geometry (Y. Tao et al., 2019).

An industrial extruder utilizes the L/D ratio in the range of 23:1 to 30:1 (Womer, 2000). In the design proposed the L/D ratio is assumed to be 23:1 while the diameter of the screw (D) is considered as 30 mm. These assumptions are based on the availability of the components locally. The material to be selected for the screw and barrel should have very high dimensional stability at elevated temperatures. this is required to avoid expansion of the screw or barrel, which usually results in stalling. The material for screws in the present research is selected as nitrided steel. Nitrided steel tends to have better dimensional stability compared to alloy steels, even at elevated temperatures of 500 ̊C (Whelan and Dunning, 1988).

Alongwith material for the screw and the barrel, other parts of the nozzle assembly, heater bands and hopper are required to be designed and manufactured. The input parameters to be controlled and designed to get the desired outputs are (Wankhade and Bahaley, 2018; Womer et al., 2017)

a. Screw design,
    - Screw diameter
    - Root diameter
    - Screw geometry
    - Screw length
    - Screw pitch
    - Length of each zone cross section of the pitch,

b. Barrel design
    - Barrel length
    - Barrel Inner Diameter
    - Barrel thickness
    - Barrel and screw clearance (minimum and maximum)

c. Nozzle assembly
    - Nozzle diameter
    - Nozzle and barrel connection

d. Heater band
    - Maximum temperature achievable
    - Heat capacity of heater bands
    - The power density of heat bands

e. Hopper design
    - Minimum breath of hopper mouth for free flowing of bulk solid

These output parameters influenced by various input parameters are

a. The volumetric flow rate of extruded filament
b. Dispersion and distribution of fiber in the matrix material (Mixing efficacy)
c. Filament diameter
d. Extrusion pressure

Among all the variables the nozzle diameter affects the output the most (R. Mishra et al., 2022). The calculations for the filament extruder were presented in the research paper by Tao et al (Manideep et al., 2019; Murugan, 2016; Y. Tao et al., 2019)

2.6 Literature review summary

After a detailed review of the existing literature, it can be summarized as follows:

- CFF presents a good alternative as reinforcement in composites. 2 -5% weight loading of CFF is the most appropriate proportion for reinforcement.
- Composites prepared with short fiber lengths give better mechanical properties compared to those prepared with powder form.
- Poly-Lactic Acid has been researched widely as a matrix material for composites prepared with CFF as reinforcement and it concluded as a good alternative.

- Poly-lactic acid is not a petroleum based plastic, which makes it more suitable for the development of environmentally sustainable products.
- The use of various optimization techniques and mathematical simulations to obtain predictive models of CFF composites in various forms such as plates, filaments, rods, etc., are the major areas for future work.
- Various techniques have been used to manufacture CFF reinforced composites. Hand Layup and Solution Moulding methods have been used most widely. Additive manufacturing techniques are in the early stage for manufacturing CFF reinforced composites. FDM technique is the most widely used technique in additive manufacturing. The major limitation of FDM is the manufacturing of filaments with various materials.
- Single screw extruders offer a good alternative for manufacturing filaments used for additive manufacturing. The nozzle design is the most important factor to be considered in the design and manufacture of filament extruders.
- Filaments with reinforcement in the sizes at the micro level and nano level i.e. powder form are available but the fibers of short length and long length are in the developmental stage.
- The development of extruders for short fiber or long fiber reinforced composite filaments is in the infancy stage and requires further research.

With the above consideration, the present research work has been undertaken. The objective of the research is to manufacture CFF reinforced PLA biocomposite filament with an extruder developed exclusively to use short CFF. Also to study the filament for their mechanical, thermal and morphological characteristics. Our findings will direct future research in utilizing short fiber CFF reinforced PLA biocomposites in the additive manufacturing process.