



Vardaan Chauhan

**OPTIMIZING DESIGN AND PROCESS PARAMETERS
FOR RECYCLED THERMOPLASTIC NATURAL
FIBER COMPOSITES IN AUTOMOTIVE
APPLICATIONS**



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Abstract

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Factors such as governmental legislation, environmental concerns, and curiosity amongst researchers are fueling the need to find novel applications for the utilization of recycled thermoplastic waste. One of the exciting fields for such plastics is fiber-reinforced polymer composites. Even though polymer composite material has been in use for decades, the use of recycled thermoplastic waste in composites is still in its infancy. Indeed, most of the research on recycled thermoplastic waste composites focuses on recycling and the properties of the material. Meanwhile, there is an apparent lack of knowledge in their field of application.

The dissertation investigates the application of recycled thermoplastic waste extracted from automotive waste back into the automotive sector as a natural fiber reinforced polymer composite (NFPC). The optimal natural fiber type and content were initially studied. A simple and low-cost compression mold for manufacturing a demonstration NFPC automotive part was designed, and the design considerations and challenges were documented. Finally, the processing parameters for manufacturing the demonstration NFPC automotive part were optimized and reported.

The result shows that the NFPC automotive part produced using the newly designed compression mold exhibited decent part quality and properties, especially the rigidity of the part, which was significantly increased by the addition of fibers to the polymer matrix. The challenges of using such a simple and low-cost compression mold were mainly related to the heating of the mold, improper filling of material in the mold, and uneven level of horizontal surfaces of the lower mold. It was also observed that temperature was the most crucial processing parameter, and by using optimal processing parameters, the productivity of the process increased by 50%.

Keywords: mold design, natural fiber, recycled thermoplastic, automotive application, process parameters

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Vardaan Chauhan
April 2022
Lappeenranta, Finland

*To someone who is no longer with me anymore but now only
lives in my memories...*

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Abstract

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List of publications

This article-based dissertation is based on the following peer-reviewed articles. The rights have been granted by the publishers to include the articles in the dissertation. The reprint of each article is included at the end of the dissertation.

- I. **Chauhan, V.,** Kärki, T., and Varis, J. (2019). Review of natural fiber-reinforced engineering plastics composites, their applications in the transportation sector and processing techniques. *Journal of Thermoplastic Composite Materials*, pp. 1-41.
- II. **Chauhan, V.,** Kärki, T., and Varis, J. (2021). Effect of Fiber Content and Silane treatment on the Mechanical Properties of Recycled Acrylonitrile-Butadiene-Styrene Fiber Composites. *Chemistry*, 3(4), pp. 1258-1270.
- III. **Chauhan, V.,** Kärki, T., and Varis, J. (2021). Design of Tooling System and Identifying Crucial Processing Parameters for NFPC Manufacturing in Automotive Applications. *Journal of Composites Science*, 5(7), pp. 1-17.
- IV. **Chauhan, V.,** Kärki, T., and Varis, J. (2021). Optimization of Compression Molding Process Parameters for NFPC Manufacturing Using Taguchi Design of Experiment and Moldflow Analysis. *Processes*, 9(10), pp. 1-10.

Author's contribution

Publication I: Review of natural fiber-reinforced engineering plastics composites, their applications in the transportation sector and processing techniques.

Vardaan Chauhan gathered the data, conducted the study, analyzed the results, prepared the initial draft, and edited the material after comments from reviewers. Timo Kärki and Juha Varis acted as the supervisors for the study and commented on the draft of the article.

Publication II: Effect of Fiber Content and Silane treatment on the Mechanical Properties of Recycled Acrylonitrile-Butadiene-Styrene Fiber Composites.

Vardaan Chauhan processed the material, produced samples for testing, tested the samples, performed the analysis of the samples, prepared the initial draft, and edited the material after comments from the reviewers. Timo Kärki and Juha Varis acted as the supervisors for the study and commented on the draft of the article.

Publication III: Design of Tooling System and Identifying Crucial Processing Parameters for NFPC Manufacturing in Automotive Applications.

Vardaan Chauhan designed the compression mold, produced samples, tested the samples, performed the analysis of the samples, prepared the initial draft, and edited the material

after comments from the reviewers. Timo Kärki and Juha Varis acted as the supervisors for the study and commented on the draft of the article.

Publication IV: Optimization of Compression Molding Process Parameters for NFPC Manufacturing Using Taguchi Design of Experiment and Moldflow Analysis.

Vardaan Chauhan generated the idea and methodology for the study, performed the simulation, analyzed the results, prepared the initial draft, and edited the material after comments from the reviewers. Timo Kärki and Juha Varis acted as the supervisors for the study and commented on the draft of the article.

Supplementary studies

The ideas from some of the publications above are presented at international conferences as conference papers and presentations. The list of supplementary studies includes

- Chauhan, V., Varis, J., and Kärki, T. The potential of reusing technical plastics. In *25th International Conference on Production Research Manufacturing Innovation: Cyber Physical Manufacturing*, Chicago, Illinois, August 9–14, 2019.
- Chauhan, V. Kärki, T., and Varis, J. Process simulation of compression molding process and effect of fiber content on recycled polymer natural fiber composites using Moldflow analysis. In *26th International Conference on Production Research*, Taiwan, July 18–21, 2021 (Online event).

Nomenclature

In the present work, variables and constants are denoted using *italics*, vectors are denoted using **bold regular style**, and abbreviations are denoted using regular style.

Latin symbols

n	Number of tests
N	Total number of data points
Y	Value of warpage

Abbreviations

ABS	Acrylonitrile butadiene styrene
ANOVA	Analysis of variance
CO ₂	Carbon dioxide
DOE	Design of experiment
DOF	Degree of freedom
ELV	End-of-life vehicle
EV	Electric vehicle
MSD	Mean-square deviation
NFPC	Natural fiber polymer composite
OA	Orthogonal array
OEM	Original part manufacturer
P%	Percentage contribution
PC	Polycarbonate
PP	Polypropylene
PU	Polyurethane
PVC	Polyvinyl chloride
R-ABSmix	Mixed recycled polymer waste
RSM	Response surface methodology
S/N	Signal-to-noise
SS	Sum of squares
USD	United States dollar

V Variance

Units

bar Bar

m³ Cubic meter

°C Degree Celsius

GPa Gigapascal

kg Kilogram

kJ Kilojoule

kN Kilonewton

m Meter

min Minutes

mm Millimeter

MPa Megapascal

pcs Pieces

rpm Revolution per minutes

sec Seconds

m² Square meter

1 Introduction

Global polymer production has been gradually increasing since the 1950s, with only a 0.3% drop in 2020, which was estimated to be because of the pandemic's impact on the industry (Tiseo 2021a). This increase in polymer production has directly corresponded to the increase in the global polymer market size. It is forecasted that the global polymer market size will be valued at over 750 billion USD by 2028, at a compound annual growth rate of 3.8% from 2021 to 2028 (Tiseo 2021b). The growth of the polymer market is propelled by increasing consumption, as well as the construction, automotive, and electrical and electronics industries, combined with the growing world population and rapid urbanization and industrialization in economies such as India and China. The economic slowdown due to the pandemic has negatively impacted the demand for polymers in industries such as automotive and electrical, and electronics. However, at the same time, increased demand for polymers in medical devices and supplies such as gloves, syringes, medical trays, and testing equipment has been observed. Despite this, packaging, construction, consumer goods, automotive and electrical, and electronics contribute to over 80% of the global polymer market share (Grand View Research 2021b).

In the automotive industry, polymers are used in components such as bumpers, dashboards, body panels, interior trims, seating, lighting, upholstery, electrical components, and fuel systems (Patil, Patel et al. 2017, Khemka 2019). Thus, polymers in an automobile account for up to 10–15% of the weight of an average car (Fortune Business Insights 2020, Grand View Research 2021a, Patil, Patel et al. 2017). The main reason for using polymers in the automotive industry is to reduce the weight of the vehicles, which directly contributes to better fuel efficiency and reduced carbon emissions. Furthermore, stringent government regulations regarding emissions and the fuel economy of the vehicles have compelled automotive original part manufacturers (OEMs) to increase the use of lightweight materials such as polymers (Fortune Business Insights 2020, Markets and Markets 2021). It has been estimated that a 10% reduction in the weight of a vehicle can improve the fuel economy by 5–7% (Grand View Research 2021a, Markets and Markets 2021, Mordor Intelligence 2021). Additionally, the emergence of electric vehicles (EVs) due to environmental regulations, government incentives, and increased investments by OEMs has also propelled the further use of polymers in the automotive industry (Fortune Business Insights 2020, Markets and Markets 2021, Khemka 2019). This combined with a global recovery in the automotive sector from the pandemic downturn and an increasing focus on lightweight vehicles will further increase polymer consumption in the automotive sector.

Thermoplastics such as polypropylene (PP), polyurethane (PU), acrylonitrile butadiene styrene (ABS), polyvinyl chloride (PVC), and polycarbonate (PC) are the most common types of polymers used in the automotive sector (Grand View Research 2021a, Fortune Business Insights 2020, Markets and Markets 2021). These thermoplastics offer excellent mechanical and thermal properties, wear and chemical resistance, good aesthetics, and are easy to process, thereby making them a more suitable choice for the automotive sector

(Mordor Intelligence 2021). Figure 1 shows the types of polymers used in various components of a car.



Figure 1: Plastic applications and amounts in automotive parts (mod. Khemka 2019, Patil, Patel et al. 2017).

About 150–200 kg of waste can be recovered from end-of-life vehicles (ELVs) and possibly even more from EVs (EuRIC 2020). The potential of ELV waste is enormous for applications such as waste-to-energy, producing pyrolysis oil, recycled polymers for secondary use, and making composites. Composites of recycled polymer are reinforced with filler materials that can be either synthetic or natural. Natural fibers are generally divided into three subgroups namely, plant fibers, animal fibers, and mineral fibers. The low cost and ease of availability of natural fibers make them a perfect choice to pair up with recycled polymers due to the latter also being either cheap or no cost. Additionally, composites of synthetic fibers are expensive, non-eco-friendly, and require vast amounts of energy in production. For example, the production of a natural fiber mat such as flax fiber requires 82% less energy than a glass fiber mat (Anandjiwala, Blouw 2007, Holbery, Houston 2006). The concept of natural fibers in the automotive sector is not new. For decades car manufacturers have been using natural fibers as a filler material for polymer composites. Car manufacturers such as Mercedes, BMW, Audi, and Volkswagen have introduced natural fiber polymer composites (NFPCs) in both interior and exterior applications of their vehicles (Ramli, Mazlan et al. 2018, Sanjay, Arpitha et al. 2016).

1.1 Background and motivation

The use of recycled polymers as a replacement for virgin polymers in automotive applications is more desirable and feasible nowadays due to stringent environmental and governmental regulations and public awareness of environmental issues. For example, the European Union's ELV directive, 2000/53/EC mandates that 85% of the total weight of the vehicle should be recyclable by 2005 and 95% by 2015. The directive also mandates that vehicles should contain 95% recyclable material with 85% recoverable by mechanical recycling and the remaining 10% via energy recovery or thermal recycling (Directive 2020). Further, legislation passed by the European Commission and European Automobile Manufacturers Association has been for new passenger cars to reduce CO₂ emissions to 95 g/km by 2020 (European Environment Agency 2018). The post-pandemic recovery in the automotive sector could see further increases in the consumption of polymers combined with already growing medical and packaging sector consumption. Such scenarios have further environmental concerns if the polymer waste from these sectors is not handled properly. This compels us to seek innovative ways to utilize recycled polymers. Thus, NFPCs offer a great pathway to utilize recycled polymer material effectively and economically better than using it for pyrolysis and energy production since NFPC manufacturing requires less energy and produces fewer emissions. These NFPCs can be quickly processed through compression molding, extrusion, and injection molding. For decades these processes have been widely used in the manufacturing of virgin polymers components as well as composite components of these polymers.

1.2 Aim of the study

The main aim of the study was to explore the application of recycled thermoplastics waste extracted from the automotive sector back into automotive applications. For this purpose, a simple and low-cost compression mold for the manufacturing of a demonstration NFPC part was developed. The design consideration and challenges faced during the development of the new compression mold were documented to aid future designers in keen on exploring this research area. Additionally, the procedures, behavior, and implications of using recycled thermoplastic as the matrix and natural fiber as the filler in composite materials for automotive applications were explored. Finally, the study aimed to determine how NFPC components for automotive applications can be produced effectively and optimally.

1.3 Hypothesis and research question

The hypothesis explored in this study is that polymers extracted from automotive waste can be recycled back into the automotive sector in the form of NFPC by means of designing a new compression mold and optimizing the processing parameters of the compression molding process used to produce NFPC components.

The research questions answered in this study are:

1. What material compositions can be used to produce NFPC materials for automotive applications?
2. What are the properties of the NFPC materials made from recycled polymers from automotive waste?
3. What are the considerations and challenges faced during the design and development of a mold for manufacturing NFPC parts?
4. How can the NFPC manufacturing process be optimized and what are the optimal process parameters for NFPC manufacturing?
5. How do optimal process parameters influence the productivity of the process?

1.4 Scope and limitations of the study

The study focuses on using any thermoplastic waste extracted from the automotive sector as the matrix for an NFPC. This implies that maintaining the quality of the extracted polymer material will always remain a challenge. Unlike virgin polymers, whose quality characteristics can be easily controlled and maintained through the entire processing stage from production to utilization in component manufacturing, the same cannot be said for recycled polymers. For recycled polymers, there may always be impurities in the form of dust or other chemicals, which could differ from one extracted part to another and could severely affect the quality of the final NFPC product.

Furthermore, the composition of recycled polymers is also an essential issue in the study. For the study, the material composition was selected based on the availability of the automotive parts extracted from vehicles and their material composition. This implies that using different compositions of thermoplastic materials such as polymer types or different blends found in existing automotive parts can result in different outcomes. Therefore, the study is limited to the percentage of polymer composition used to process NFPC materials and later for the automotive part. Additionally, using specific additives in NFPC materials to improve the interfacial adhesion and plasticity of the material can also be a limiting factor. Additionally, the study focused on using wood fibers from European spruce trees as a filler material for the NFPC.

Finally, compression molding was selected as the primary process for producing tensile and impact samples for mechanical testing in Article II. Similarly, when producing the final automotive components from the NFPC material compression molding process, a specifically designed compression mold was employed in Article III.

1.5 Outline

Chapter 2 describes the existing state of the art of NFPC in automotive applications and their manufacturing.

Chapter 3 explains the methodologies implemented for all the articles related to the study.

Chapter 4 introduces and discusses the results of the study.

Chapter 5 presents the conclusions of the study.

1.6 Framework

Figure 2 presents the entire framework of the study.

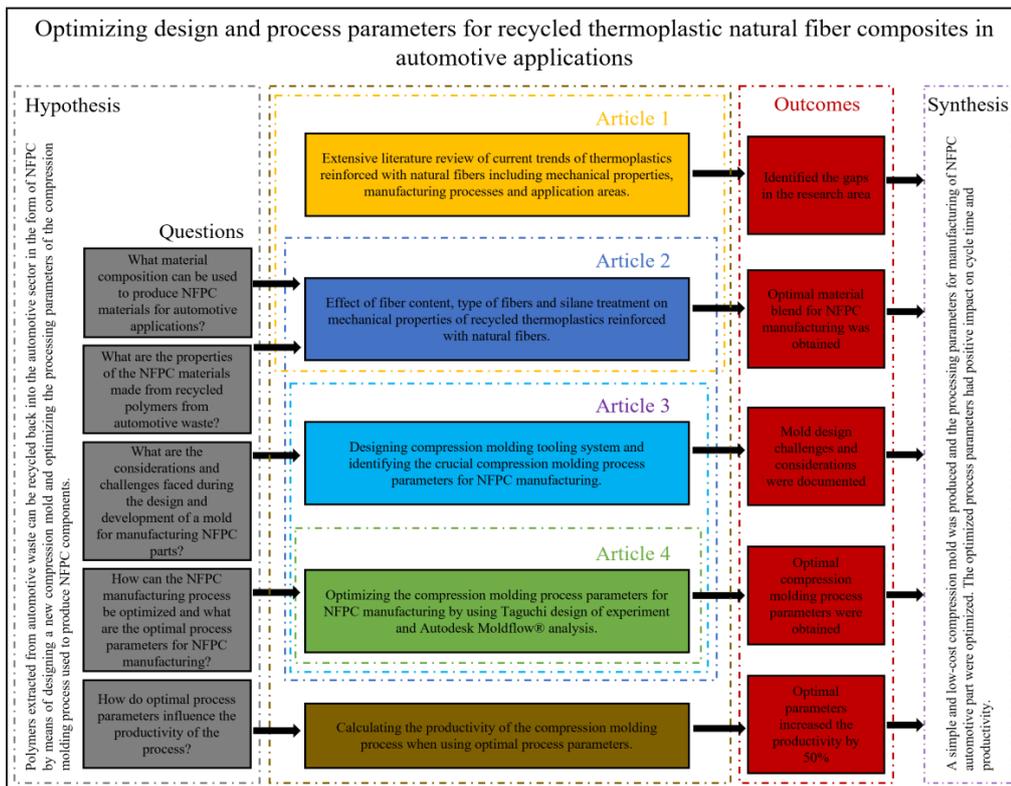


Figure 2: Framework of the thesis work.

2 State of the art

2.1 End-of-life vehicle waste

Annually, the automotive industry contributes 40 million ELVs worldwide (Sakai, Yoshida et al. 2014). In Europe, every year about 6–7 million ELVs are treated according to the European Union’s ELV directive, and this generates over 8 million tonnes of ELV waste. ELV polymer waste amounts to about 1 million tonnes of the total ELV waste (EuRIC 2020). Due to the stringent legislation in the European Union, ELV waste in the form of automotive shredder residue going into landfills is limited to 5% of the total vehicle weight. Currently, under the European Union’s ELV directive, 2000/53/EC, the ELV waste must be either recovered or disposed of in landfills. The recovery of ELV waste is either done via energy recovery or recycling as shown in Figure 3. The recycling of ELV waste is further classified as product recycling or material recycling. Product recycling means recovering parts and subassemblies which are in good condition that can be reused immediately after removal from the ELV waste with or without any restoration. Meanwhile, material recycling is defined as processing ELV waste that cannot be further used in product recycling. In Europe, this is the most preferred form of waste treatment; however, it is also quite complex and expensive. Material recycling is beneficial in reducing waste disposal in landfills, preserving non-renewable resources, and reducing energy consumption, since the production of recycled material consumes less amount of energy than the production of virgin material (Merkisz-Guranowska 2018).

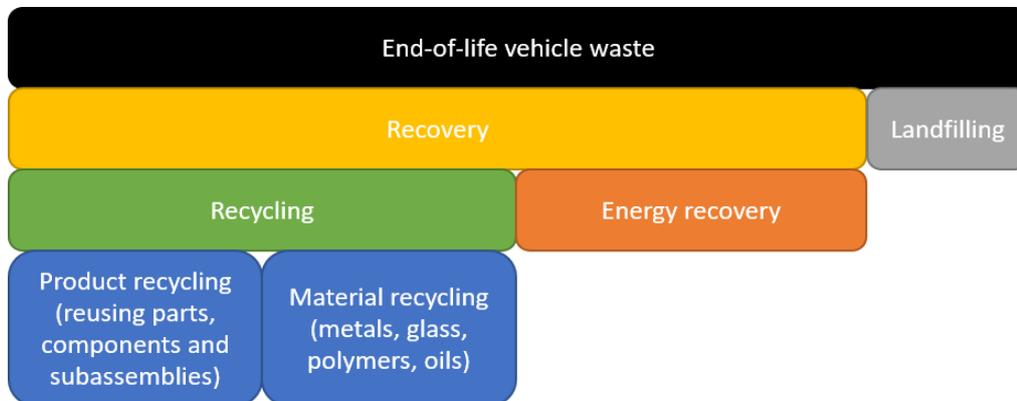


Figure 3: Treatment options of end-of-life vehicle (mod. Merkisz-Guranowska 2018).

Many mechanical, chemical and thermal recycling processes for recovering ELV polymer waste have been developed. ELV polymer waste is separated by each plastic grade using separation methods such as density separation or laser and infra-red systems (EuRIC 2020). In the case of ELV polymer waste with different blends of polymers, these are usually recovered as mixed plastic waste. Later, the recovered ELV polymer waste is

processed by shredding and extrusion to produce recycled polymer pellets for reuse in industrial applications (EuRIC 2020). The ELV polymer waste is mainly comprised of thermoplastics polymers, which are easy to process since they can easily be remolded by heating and then reshaped after solidification, unlike thermosets. Therefore, the applications of recycled thermoplastics extracted from ELV waste can be found in automotive, construction, packaging, electrical and electronic, agriculture, and houseware (PlasticsEurope 2020).

2.2 NFPC in automotive applications

Polymer matrices reinforced with natural fibers have already been in extensive use for many decades in various applications such as automotive, construction, aerospace, sports equipment, boats, furniture, and packaging (Holbery, Houston 2006, Thomas 2019, Shinoj, Visvanathan et al. 2010). For over two decades substantial research work has been done on the properties, processing, and applications of NFPC (Naik, Mishra 2005, Bledzki, A. K., Reihmane et al. 1996, Alam, Ahmed et al. 2008, Jayamani, Hamdan et al. 2016). The automotive sector has extensively employed NFPC material as one of the construction materials for vehicle parts by replacing many polymers and metal-based components and features with NFPCs. In Europe, the automotive sector accounts for 28.2% of the total market share for NFPC (Reports and Data 2020). Car manufacturers such as Audi, BMW, Mercedes, General Motors, and Ford have used NFPC in their car models for both interior and exterior applications, as shown in Table 1.

Table 1: NFPC application in automotive (Zhao, Zhou 2014).

Manufacturer	Model	Natural Fiber	Polymer	Component
Daimler	E class	Jute	Epoxy	Door Panel
BMW	7 series	Sisal	Acrylic	Door Panel
General Motors	Chevrolet Impala	Flax	PP	Trim panel and back shelf
Audi	A2	Flax/sisal	PU	Door trim panel
Ford	Mondeo	Kenaf	PP	Door panel

However, in the past few years, the research focus has shifted to the use of recycled polymers reinforced with natural fibers. Numerous studies focusing on recycled polymer composites have been published (Lu, Swan et al. 2012, Farahani, Ahmad et al. 2012, Inácio, Nonato et al. 2018, Mengeloğlu, Karakuş 2008, Adhikary, Pang et al. 2008, Alam,

Ahmed et al. 2008). In a study, Cholake, Rajarao et al. (2017) worked on utilizing automotive waste plastics alongside natural fiber and developed a novel approach to manufacturing composite panels. These composite panels made from automotive waste plastics and agricultural macadamia shell waste exhibit better flame retardancy and thermal properties than 100% plastics panels (Cholake, Rajarao et al. 2017). In another study, mechanical and thermo-chemical properties of composites made from waste automotive bumpers and sugarcane skin flour were analyzed (Wu, Zhao et al. 2019). Meanwhile, in a study by Kiziltas and Gardner 2012, the researchers observed that by reinforcing recycled nylon (6,6) with 20% microcrystalline cellulose, the tensile strength was increased by 110%, and the thermal expansion decreased, thereby making the composite material more suitable for under-the-hood automotive applications (Kiziltas, Gardner 2012).

2.3 NFPC manufacturing methods

Based on the literature, common manufacturing methods employed to process NFPC material include compression molding (Adekunle, Åkesson et al. 2010, Idicula, Sreekumar et al. 2009, Cisneros-López, Anzaldo et al. 2017, Bledzki, Andrzej K., Faruk 2004), injection molding (Nyström, Joffe et al. 2007, Shibata, Cao et al. 2006, Bledzki, Andrzej K., Faruk 2004) extrusion (Mulinari, Voorwald et al. 2009, Grande, Torres 2005), and resin transfer molding (Dansiri, Yanumet et al. 2002, Rouison, Sain et al. 2004, Idicula, Sreekumar et al. 2009). Common NFPC processing methods and their respective advantages and disadvantages are shown in Figure 4.

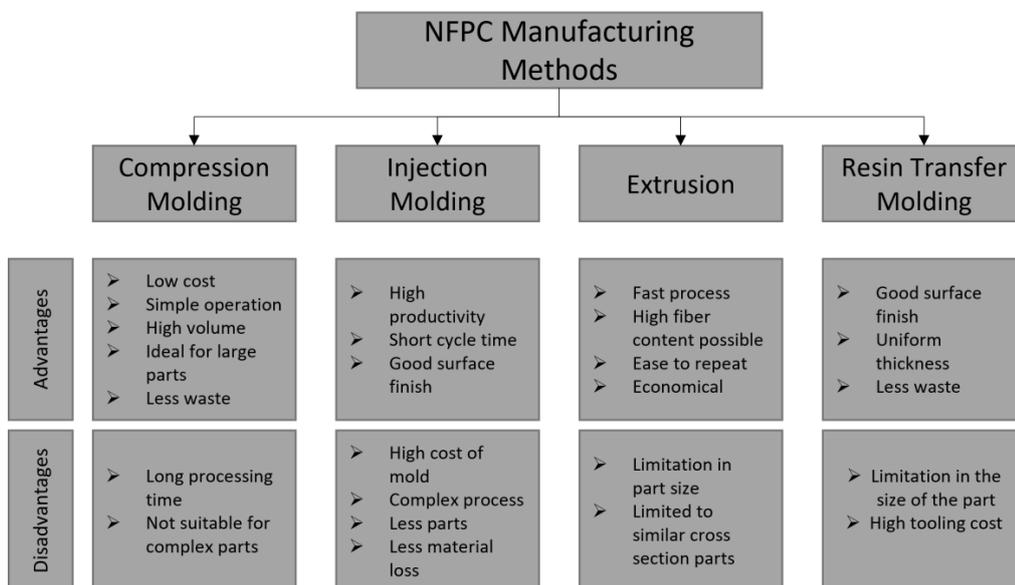


Figure 4: Common NFPC manufacturing processes (mod. Masri, Mohd et al. 2021, Lotfi, Li et al. 2021).

Compression molding is a low-cost, simple, high-volume process, and it is also suitable for large parts, which makes it a perfect process for NFPC manufacturing for automotive applications. Compression molding is a closed molding process in which the composite material is placed in the cavity of the mold as shown in Figure 5, and is formed by the application of heat and force (Jaafar, Siregar et al. 2019). The shape of the cavity of the mold is predetermined according to the final shape of the desired component. The composite material is either heated beforehand using an oven through radiation or can be heated inside the mold through conduction using heating elements prebuilt into the lower mold. Once the material is heated to the required temperature, the upper mold is pushed down until it fits the lower mold perfectly. Due to the application of force by the upper mold and heat, the composite material starts to move into the space between the lower and upper mold until it is filled, and thus the desired shape is obtained (Farris 2016).

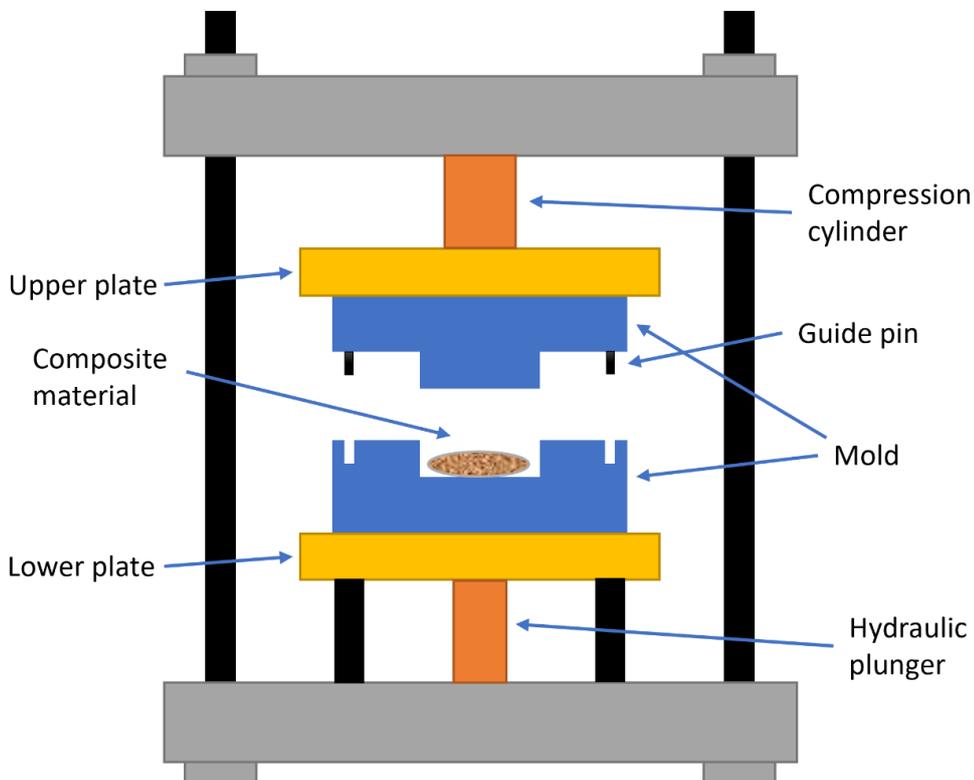


Figure 5: Compression molding setup.

2.4 Compression molding mold design considerations

The designing of the mold is a crucial part of the process considering the production of new NFPC components using compression molding. The mold design considerations can vary depending on the processing technique, raw material, processing parameters, and final application of the molded part. A proper mold design results in the production of quality molded parts by ensuring the uniform filling of the mold and should be able to aid in heat dissipation to reduce warpage and shrinkage (Fischer 2013). Some common considerations taken while designing mold are:

1. Sidewalls of both upper and lower mold must be at a taper angle to ensure easy removal of material from the mold after the molding process
2. An ejector pin can aid in the removal of the molded part from the mold and therefore provisions must be made during the design phase to add an ejector pin to the lower mold.
3. It is essential to provide clearance between the upper and lower mold for excessive material to flow out of the mold during the compression phase. Additionally, this clearing will also aid in the venting of gases produced due to the heating of the material in the mold.
4. The shape of the molded part has a direct consequence on the manufacturing time of molds and later the cycle time of the molding process. For example, a mold for a simple part can be easily made and will also have a relatively lower cycle time. However, a mold for a complex part will require longer manufacturing time, special tools, can require further subassemblies, and thus will also have a high cycle time.
5. Sharp corners and sudden variation in the cross-section of the regions should be avoided since they can lead to cracks or warpage. Additionally, it can affect the flow of the material in the mold during the compression phase.
6. If the molded part requires any unique surface feature such as raised or sunken designs, it must be added to the mold during the manufacturing stage.
7. The cycle time of the molding process is affected by the weight, surface area, and thickness of the molded part. In case of significantly varying thickness of the molded part, cooling must be carefully controlled otherwise excessive shrinkage can occur in the thicker sections (Mesquita, Barbosa et al. 2017, Riley 2012).

2.5 Processing parameters for compression molding

The processing parameters of the compression molding process also play a vital role in NFPC manufacturing. The selection of appropriate processing parameters is crucial to producing quality molded parts and achieving good mechanical properties (Cisneros-López, Anzaldo et al. 2017, Adekunle, Åkesson et al. 2010). The temperature, time, and

compression force are the three most dominating factors that influence the final performance of the NFPC material, as shown in Figure 6. The temperature is one of the most crucial parameters in compression molding. It is usually based on the melting temperature of the polymer material and the degradation temperature of the natural fibers. Furthermore, a high temperature can severely affect the natural fibers and reduce their properties (Jamiluddin, Siregar et al. 2018, Jamiluddin, Siregar et al. 2016). Additionally, a uniform temperature can improve the filling process and reduce warpage. The compression force is another crucial parameter since the use of a moderate-high compression force can improve the interfacial adhesion between the fiber and polymer matrix. Any extreme force can also result in fiber breakage, thus decreasing the properties of the NFPC (Rubio-López, Olmedo et al. 2015, Medina, Schledjewski et al. 2009). Finally, an insufficient holding/compression time can also cause defects in the molded part since an adequate time is required for the heated composite material to take on the shape of the mold and solidify. A summary of processing parameters used in the compression molding of NFPC in various literature is shown in Figure 7. In the figure, the temperature is represented on the x-axis, while the y-axis represents the pressure and the holding time is denoted by the size of the spheres. Additionally, most of the literature refers to the force as the pressure in their studies with units such as bar or MPa.

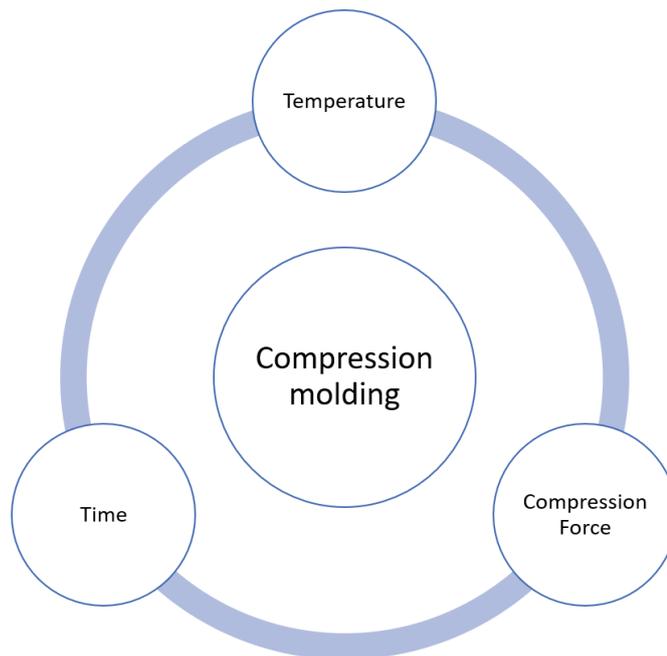


Figure 6: Crucial processing parameters in the compression molding process (mod. Yallew, Kassegn et al. 2020, Ho, Wang et al. 2012).

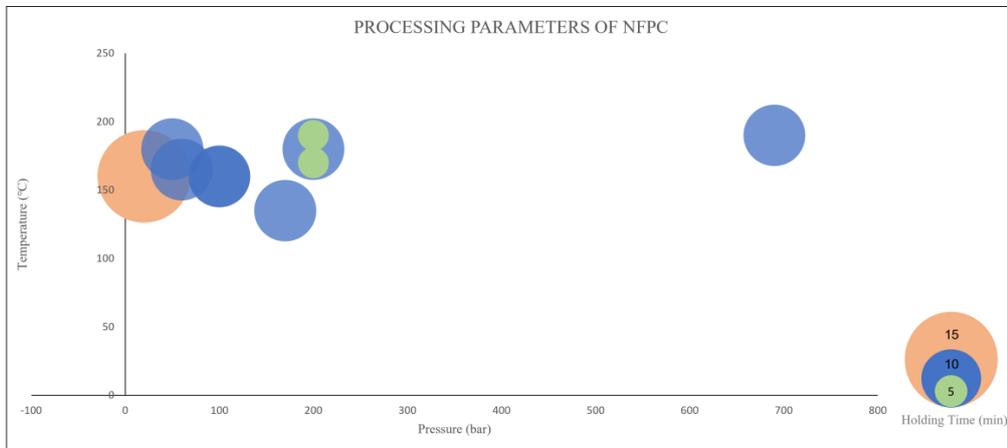


Figure 7: Processing parameters used in previous studies of compression molding for the NFPC manufacturing (mod. Yu, Ren et al. 2010, Jaafar, Siregar et al. 2018, Liu, Drzal et al. 2007, Zampaloni, Pourboghraat et al. 2007, Zhong, Li et al. 2011, Khondker, Ishiaku et al. 2005, Prasad, Agarwal et al. 2016, Shibata, Cao et al. 2005, Bledzki, Andrzej K., Faruk 2004).

2.6 Optimizing processing parameters

Furthermore, it is also important to select optimal processing parameters for NFPC manufacturing since they are vital in reducing the cycle time and can therefore result in greater economic benefits. In the past, several researchers have focused on optimizing the processing parameters for NFPC manufactured using compression molding (El-Shekeil, Sapuan et al. 2013, Govindaraju, Jagannathan et al. 2014, Kandar, Akil 2016, Selamat, Kasim et al. 2020, Zuhudi, Minhat et al. 2017, Van-Pham, Nguyen et al. 2018, Tharazi, Muhamad et al. 2017, Sumesh, Kanthavel 2020). As stated earlier, temperature, time, and compression force are the most crucial compression molding process parameters for NFPC manufacturing. Therefore, most of the studies have focused on optimizing these parameters. In order to optimize the processing parameters, it is essential to define the quality characteristics for which the processing parameters are optimized. These quality characteristics usually vary depending on the purpose of the study and include warpage, shrinkage, tensile strength, impact strength, flexural strength, or cycle time. Some common optimization techniques include the Taguchi Design of Experiment (DOE) method (El-Shekeil, Sapuan et al. 2013, Selamat, Kasim et al. 2020, Sumesh, Kanthavel 2020), and response surface methodology (RSM) using Box-Behnken designs (Kandar, Akil 2016, Govindaraju, Jagannathan et al. 2014). However, Taguchi DOE is widely used in optimizing compression molding process parameters. The Taguchi DOE method uses an orthogonal array (OA), which reduces the number of experimental trials to study the parameters. The experimental output from OA can be the warpage, shrinkage, tensile strength, impact strength, flexural strength, or cycle time depending on the aim of the study. Then this experimental output is transformed into a signal-to-noise (S/N) ratio. Afterward, either smaller-the-better quality characteristics are employed for the warpage,

shrinkage, and cycle time, or larger-the-better quality characteristics are used to measure the mechanical properties. In the end, an analysis of variance (ANOVA) is performed to measure the significance of each processing parameter on the value of quality characteristics (Kamaruddin, Khan et al. 2010, Oktem, Erzurumlu et al. 2007). The key literature studies related to optimizing compression molding processing parameters for NFPC manufacturing are shown in Table 2. The table explains the processing parameters, quality characteristics, and optimization methods used in the existing literature related to the optimization of compression molding parameters for NFPC materials.

Table 2: Key state of the art for optimizing compression molding parameters for NFPC materials.

References		Selamat, Kasim et al. 2020	Kandari, Akil 2016	Govindaraju, Jagannathan et al. 2014	El-Shekeil, Sapan et al. 2013	Zahedi, Minhat et al. 2017	Van-Pham, Nguyen et al. 2018	Tharazi, Muhamad et al. 2017	Suresh, Kanthavel 2020	Varadaraju, Srinivasan 2019	El-Shekeil, Sapan et al. 2014	Ramakrishnan, Moigne et al. 2019
	Composite blends	PALF/PP	Flax/PLA	Wool/PP	Kenaf/PU	Flax/PP	Banana/ABS	Kenaf/PLA	Banana-Coir/Epoxy	Kenaf/PP	Cocoa pod husk/PU	Flax/PP
Process Parameters	Time	X	X	X	X	X	X	X		X	X	X
	Temperature	X	X	X	X		X	X	X	X	X	X
	Force	X	X	X		X	X	X	X	X		X
	Speed				X						X	
	Fiber Percentage					X		X	X	X	X	
Quality Characteristics	Impact Strength		X	X		X			X	X		
	Flexural Strength			X		X	X		X	X		
	Tensile Strength	X		X	X	X	X	X	X	X	X	X
Method	Taguchi	X			X	X			X		X	
	RSM		X	X								
	ANOVA		X	X	X						X	

3 Methods

A detailed description of materials and methods used in the study are presented in the articles (II–IV). The general description of the materials and methods for the articles is presented in Table 3.

Table 3: Material and methods used in the study.

Article	Materials	Methods
I	Published studies about composites of thermoplastics and natural fibers; NFPC applications in the transport sector.	A literature review
II	Recycled ABS and other recycled thermoplastics; 10 and 20% wood fiber and palmyra fiber silane treated and untreated; lubricant.	Surface treatment of fibers to improve interfacial adhesion with the polymer matrix. Compression molding for tensile and impact testing samples and testing the mechanical properties of the composite materials.
III	Recycled ABS and other recycled thermoplastics used in article II; 10% untreated wood fiber; elastomer and lubricant	Compression molding mold design for NFPC components, compressive testing of the NFPC part produced using the designed compression molding mold.
IV	Same material composition as in article III.	Using the Taguchi DOE method and Moldflow® simulation software, optimizing the compression molding process parameters.

Article I focused on a comprehensive literature review of thermoplastics reinforced with natural fibers and their applications in the transport sector. The literature study explains the various types of virgin thermoplastics used in the transportation sector and the potential of their recycling after the lifecycle of the vehicle. It also focuses on various

natural fiber types, benefits, properties, and their applications in automotive, aerospace, and marine applications. Lastly, the literature review describes the common processing techniques employed to manufacture NFPC components for various applications in the transportation sector. The primary motive of the review was to identify the current state of the art of NFPC material in automotive and other transportation applications.

Article II studied the effect of fiber content and silane treatment on the tensile and impact properties of a compression-molded NFPC. The composite material consisted of recycled automotive thermoplastic waste as a matrix, and a varying percentage of untreated and silane treated wood and palmyra fibers were used as a filler material. The recycled thermoplastic material was composed of ABS (80.77%), ABS + styrene-acrylonitrile resin (1.75%), ABS + acrylonitrile styrene acrylate (4.61%), ABS + acrylonitrile ethylene styrene (1.59%), ABS + poly(methyl methacrylate) (7.69%) and ABS + polystyrene (3.59%). The NFPC material composition used in the study is shown in Table 4.

Table 4: Material composition of the composites used in the study.

Designation	Polymer (%)	Wood fiber (%)	Palmyra fiber (%)	Silane (%)	Lubricant (%)
R-ABSmix	100	-	-	-	-
R-ABS/10WF	87	10	-	-	3
R-ABS/10WF-S	84	10	-	3	3
R-ABS/20WF	77	20	-	-	3
R-ABS/20WF-S	74	20	-	3	3
R-ABS/10PF	87	-	10	-	3
R-ABS/10PF-S	84	-	10	3	3
R-ABS/20PF	77	-	20	-	3
R-ABS/20PF-S	74	-	20	3	3

Article III focused on the design considerations for a compression mold for NFPC component manufacturing. An automotive car battery cover manufactured initially by injection molding was selected as the component to be manufactured using the NFPC

material. The original car battery cover and the newly designed part suitable for manufacturing with compression molding are shown in Figure 8. The study explained the design considerations and changes made step-by-step in the existing design to make it suitable for a compression molding process utilizing NFPC material.

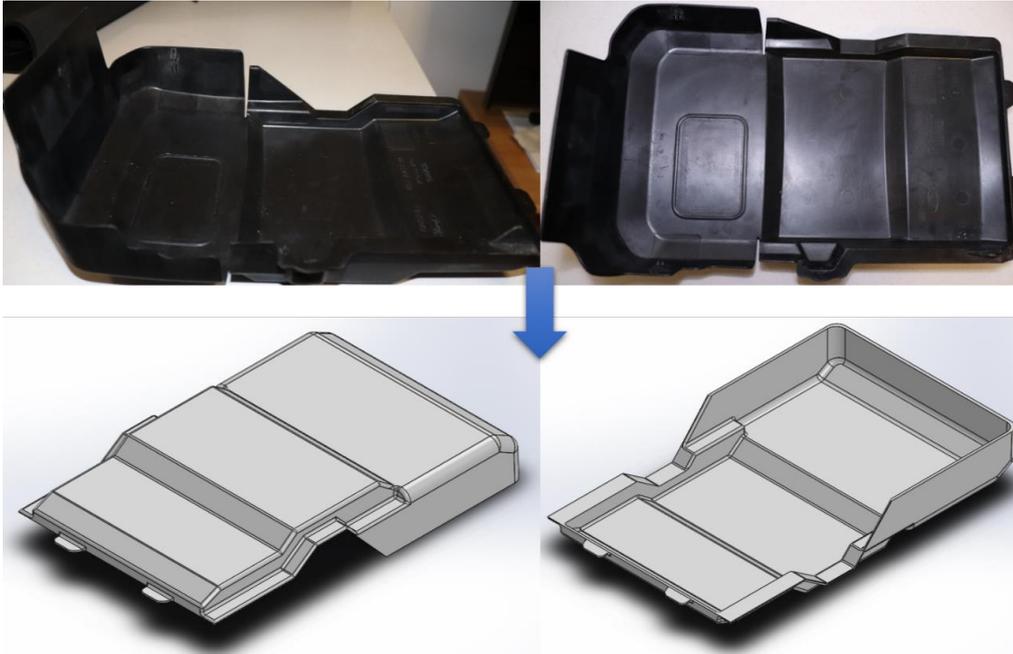


Figure 8: Original injection molded part (up) and newly designed compression molded part.

After designing and machining the new mold for compression molding of the demonstration NFPC automotive part, the part was produced using Stenhøj C-type 40-ton hydraulic press and with the following composition: polymer (82%), wood fiber (10%), lubricant (3%) and elastomer (5%). The processing parameters employed were: material temperature = 200–205 °C, heating time = 15 min, compression time = 120 sec, compression force = 400 kN, and mold temperature = 60–70 °C. The production layout is shown in Figure 9. Later, the NFPC part underwent compressive testing. Finally, the challenges faced during the development of the mold and later during the manufacturing of the NFPC component related to the mold design were documented and reported. Some suggestions for further improvement in the mold were reported in the study.

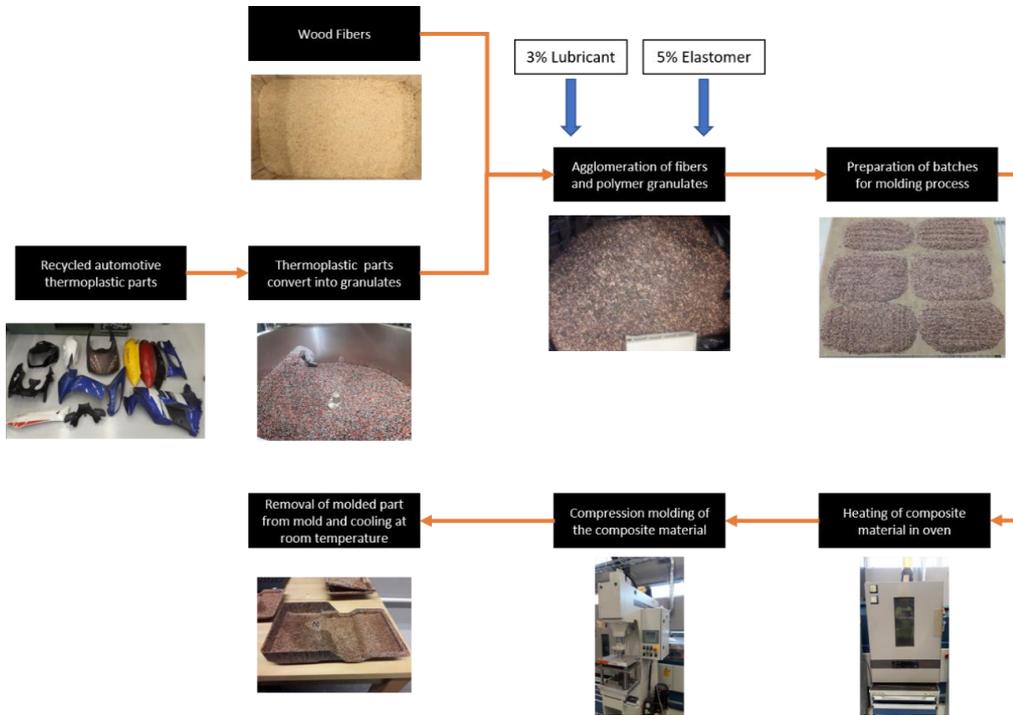


Figure 9: Production layout for manufacturing of the NFPC molded part.

Article IV had the goal of optimizing the process parameters of the compression molding process used in the manufacturing of the test NFPC component in Article III. The optimization of four crucial compression molding process parameters was the focus of the study. These parameters were the mold temperature, melt temperature, compression force, and time. The mold temperature referred to the temperature of the molds, while the melt temperature was the material temperature after heating in an oven. Additionally, the compression force was the force of compression exerted by the press machine, and the time was described as compression time. The study utilized the Taguchi DOE method alongside an Autodesk Moldflow® simulation to determine the optimal process parameters. The Taguchi DOE approach helped to design the initial set of process parameters according to an L9 OA. The levels of process parameters used in the study are shown in Table 5.

Table 5: Levels of process parameters used in the optimization study.

Symbol	Parameter	Unit	Levels		
			1	2	3
A	Mold temperature	°C	60	70	80
B	Compression time	sec	30	40	50
C	Melt temperature	°C	210	220	230
D	Compression force	kN	400	500	600

Later the sets of parameters were run in an Autodesk Moldflow® simulation of the automotive component. The outcomes from the simulation were then transformed into an S/N ratio to determine the optimal parameters. Finally, the percentage significance of the process parameters on the outcome was studied using an ANOVA analysis.

4 Results and discussion

4.1 Effect of the fiber type, fiber content, and silane on the mechanical properties of NFPC

4.1.1 Tensile strength

As shown in Figure 10, the addition of natural fibers to the polymer matrix to form NFPC resulted in a decrease in the tensile strength due to weak interfacial bonding between the fibers and the polymer matrix. In the case of mixed recycled polymer waste (R-ABSmix), which served as the polymer matrix in all the composite blends, the tensile strength was observed to be at 16.42 ± 1.20 MPa. The low tensile strength value for the polymer material, which is predominately ABS and its blends, was attributed to the presence of impurities such as dust, chemicals, or oil found on some surfaces of the recycled automotive waste. These impurities could negatively affect the agglomeration process used to blend fibers and crushed polymer material. Additionally, it is also possible that after years of being in use, the automotive parts which were recycled for this study suffered from the wear and tear from the environment and operating conditions, thereby causing the properties of the polymer material to decline.

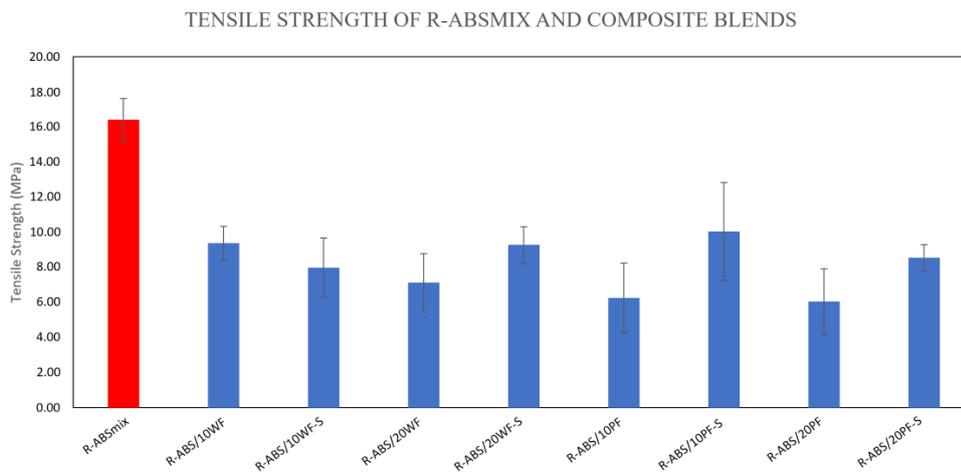


Figure 10: Tensile strength data of R-ABSmix and other composite blends.

When examining the NFPC materials, some interesting results were observed. With an increase in the fiber content for both types of fiber, the tensile strength decreases, which had been documented in many previous studies (Kim, Lee et al. 2011, Chotirat, Chaochanchaikul et al. 2007). Before silane treatment, both R-ABS/10WF and R-ABS/20WF composites exhibited higher tensile strength than R-ABS/10PF and R-

ABS/20PF composites, thus indicating that wood fibers reacted with the polymer matrix more homogeneously than palm fibers. Generally, surface treatment methods such as silane have a positive effect on the tensile strength of NFPC material. Therefore, after the silane treatment of the composites, R-ABS/20WF, R-ABS/10PF, and R-ABS/20PF all exhibited improvements in their respective tensile strength values. However, in the composite R-ABS/10WF-S, an anomaly was observed as after the silane treatment of the fibers, the tensile strength of silane treated composite R-ABS/10WF-S was found to be lower than the untreated composite R-ABS/10WF. Such anomaly is not rare, and few studies have reported it in the past. Such studies attributed this phenomenon to the self-condensation reaction occurring between silane monomers resulting in the formation of polysiloxane molecules on the surface of natural fibers, which negatively affects the silane reactivity to interact with the polymer molecules (Väntsi, Kärki 2015, Chotirat, Chaochanchaikul et al. 2007). Since this anomaly was not observed in any of the other composite blends of wood or palm fiber, it could be safely assumed that such anomalies are not typical, and a decrease in tensile strength could be due to some mishandling during the silane treatment of R-ABS/10WF-S. Additionally, significant variations in the tensile strength values were also observed in most of the composite materials with exception of R-ABS/10WF, R-ABS/20WF-S, and R-ABS/20PF-S. Since the process for producing the composite samples and testing was similar for all the composite blends and R-ABSMix, it is possible that the presence of impurities in R-ABSMix may have caused the improper fusion of the fiber and polymer matrix and possibly created voids or weak spots in the composite structure in some samples. These voids or weak spots could cause variations in the composite structure even amongst the same composite blends since from one composite cake 5–7 tensile testing samples of that particular blend were made.

4.1.2 Tensile modulus

The effect of the addition of natural fiber on the tensile modulus value is shown in Figure 11. In contrast to the tensile strength, the tensile modulus tends to increase when the natural fibers were added to the polymer matrix (Chotirat, Chaochanchaikul et al. 2007, Kimchiang, Threepopnatkul et al. 2011). Since the tensile modulus is the measure of the stiffness of the solid material, it would be correct to state that fibers improve the stiffness of the composite material. This statement is further strengthened as increasing the fiber content increased the tensile modulus of the composite materials. From the study, it is noticeable, that wood-based composites exhibited a slightly higher tensile modulus than the palm-based composites for both 10% and 20% fiber contents, which is in alignment with their tensile strength results.

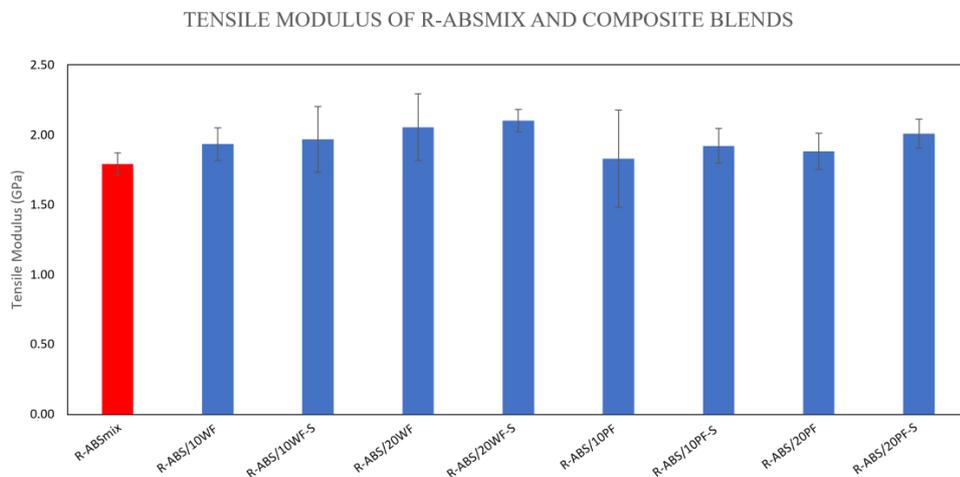


Figure 11: Tensile modulus data of R-ABSmix and other composite blends.

Finally, after silane treatment, it was observed that the tensile modulus for all the composite materials increased, but the increase was relatively small compared to their respective counterparts. This indicates that the silane treatment has less to no effect on the tensile modulus, and even if the self-condensation reaction occurs in the case of R-ABS/10WF-S, it did not negatively affect the tensile modulus value. Similar to the tensile strength, large variations in the tensile modulus were observed in a few composite blends, excluding R-ABS/10WF, R-ABS/20WF-S, R-ABS/10PF-S, R-ABS/20PF, and R-ABS/20PF-S. Such variations could also be attributed to the presence of impurities, voids, or weak spots in the composite structure, which deter the fibers from bonding effectively with the polymer matrix to improve the stiffness of the material.

4.1.3 Impact strength

The effect of the fiber type, fiber content, and silane on the impact strength of composite blends is shown in Figure 12. The impact strength data follow similar trends as seen for the tensile strength data. The impact strength of R-ABSmix was 5.98 ± 0.92 kJ/m², and again significant variations were observed, which were also probably due to the presence of impurities or improper agglomeration. The impact strength for all the composite blends decreased drastically after the addition of fibers, mainly due to the weak interfacial bonding between the fibers and the polymer matrix. Afterward, the silane treatment of the composite blends exhibited some improvement in the impact strength for all of the composite blends except R-ABS/10WF-S. The reason for this decline was probably due to the occurrence of self-condensation or improper silane treatment of the composite, as explained earlier. For wood-based composite blends, R-ABS/10WF exhibited the highest impact strength at 2.90 ± 0.65 kJ/m², but with high variation. Whereas for the palm-based composite blends, R-ABS/10PF-S exhibited the highest impact strength at 3.12 ± 0.87 kJ/m², but it also exhibited the highest variation among all the composite blends. Finally, in almost all the other composite blends, high variations in the impact strength value were also observed. Only R-ABS/20WF exhibited a low amount of variation.

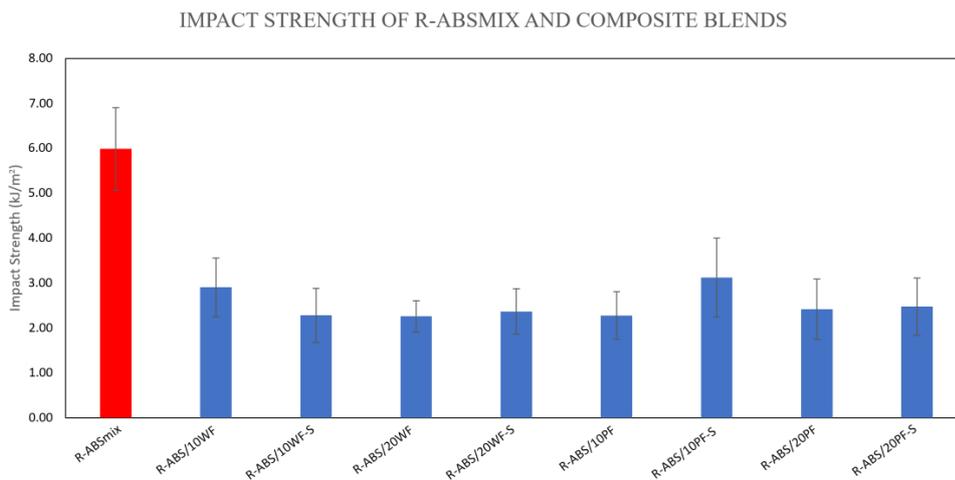


Figure 12: Impact strength data of R-ABSmix and other composite blends.

4.1.4 Density

The density for R-ABSmix and all composite blends is shown in Figure 13. The recycled thermoplastic material mainly consists of blends of ABS and some other engineering polymers which are lightweight. Therefore, the extracted recycled polymer material (R-ABSmix) also has a low density (1032 kg/m^3) since the density of a material is the measure of its mass per unit volume. Inherently, natural fibers have a low density compared to some other synthetic fibers, such as glass fibers, and this makes them an excellent choice in applications where the weight of the final product plays an important role. Typically, the density of natural fibers ranges between $1200\text{--}1600 \text{ kg/m}^3$, while in comparison glass fiber density is 2400 kg/m^3 (Thyavihalli Girijappa, Mavinkere Rangappa et al. 2019). Overall, all the composite blends have relatively similar densities. The highest density was 1087 kg/m^3 observed in the R-ABS/20PF-S composite, and the lowest density composites were R-ABS/10WF and R-ABS/20PF at 1067 kg/m^3 . Apart from the R-ABS/10PF-S composite, for all other composite blends, the density of the composite increased after silane treatment, indicating that the presence of silanol groups in the composite structure increases the density of the composite blend. However, the R-ABS/10PF-S composite exhibited less density in comparison to its untreated counterpart R-ABS/10PF. This could be attributed to the occurrence of self-condensation in the composite blend or more probably due to the presence of voids or other lightweight impurities in the samples of R-ABS/10PF used for the density testing.

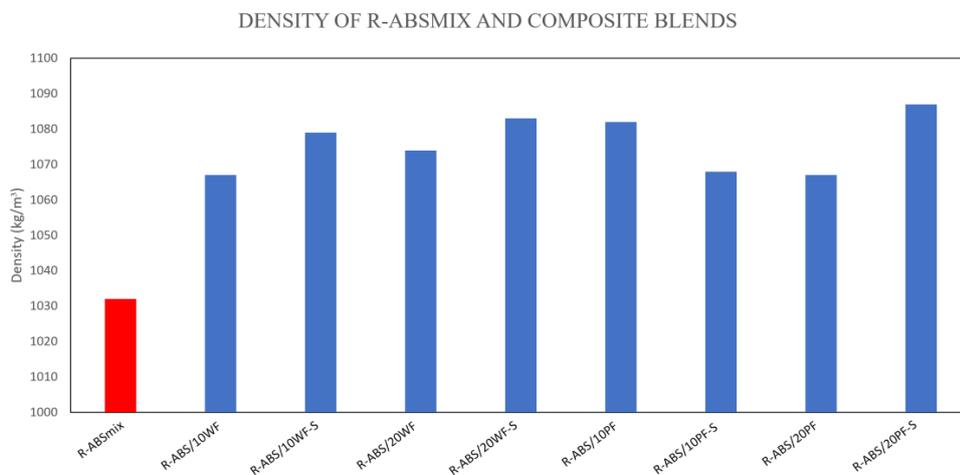


Figure 13: Density data of R-ABSmix and other composite blends.

4.1.5 Optimum fiber type and content

Finally, finding the optimum fiber type and content was crucial for further research work and to further narrow down the scope of the study. The manufacturing of NFPC products with 8 different composite blends would be cumbersome and could have slowed down the entire dissertation. Therefore, only the best performing composite amongst all the available blends was selected for further processing. Table 6 shows the tensile strength, tensile modulus, impact strength, and density of all composite blends. The top three values relative to each other in each category are highlighted.

Table 6: Highlighting best performing composite blends.

Composite	Tensile Strength (MPa)	Tensile Modulus (GPa)	Impact Strength (kJ/m ²)	Density (kg/m ³)
R-ABS/10WF	9.39 ± 0.97	1.93 ± 0.11	2.90 ± 0.65	1067
R-ABS/10WF-S	7.98 ± 1.69	1.96 ± 0.23	2.27 ± 0.60	1079
R-ABS/20WF	7.12 ± 1.66	2.05 ± 0.23	2.25 ± 0.34	1074
R-ABS/20WF-S	9.28 ± 1.03	2.10 ± 0.08	2.36 ± 0.50	1083
R-ABS/10PF	6.26 ± 1.97	1.82 ± 0.34	2.27 ± 0.52	1082
R-ABS/10PF-S	10.04 ± 2.79	1.92 ± 0.12	3.12 ± 0.87	1068
R-ABS/20PF	6.04 ± 1.83	1.88 ± 0.13	2.41 ± 0.67	1067
R-ABS/20PF-S	8.55 ± 0.74	2.00 ± 0.10	2.47 ± 0.63	1087

From Table 5, it is clear that R-ABS/10WF and R-ABS/10PF-S were the best performing composite blends relative to the other composite blends. Even though R-ABS/10PF-S exhibited a higher tensile and impact strength compared to R-ABS/10WF, it is also important to note that the R-ABS/10PF-S composite also exhibited high variation in those values. Meanwhile, the tensile modulus and density of both these composite blends were similar. Another, crucial aspect to note was that R-ABS/10WF just required an agglomeration of fiber and crushed polymers, while R-ABS/10PF-S also required silane treatment which is an additional procedure that could incur costs and increase the processing time. Finally, the wood used in R-ABS/10WF was common European spruce, whereas the Palm fiber used in R-ABS/10PF-S was predominantly indigenous to parts of Africa and Asia. Therefore, keeping in mind the level of variation in the tensile and impact strength, alongside less processing and easy availability of wood fiber, R-ABS/10WF was selected as the best composite blend and was used in further research work.

4.2 Designing a mold for NFPC manufacturing

After selecting the best composite blend, the next step was to select an existing thermoplastic automotive part that could be studied so that it could be fabricated using the composite material. Many possible options were explored, and finally, a car battery cover, as shown in Figure 14 was selected (note: this was not the chosen part for the study, for that see Article III). One main reason for selecting this part was its application area since it is used in under-the-hood applications, thereby making it an interesting prospect since currently, most of the applications of NFPCs are either inside the car or door panels (Holbery, Houston 2006, Zhao, Zhou 2014). After a close examination of the part, the part was redesigned (see Article 3 for more details) to make it more suitable for manufacturing using the compression molding process since the actual part was produced by injection molding.

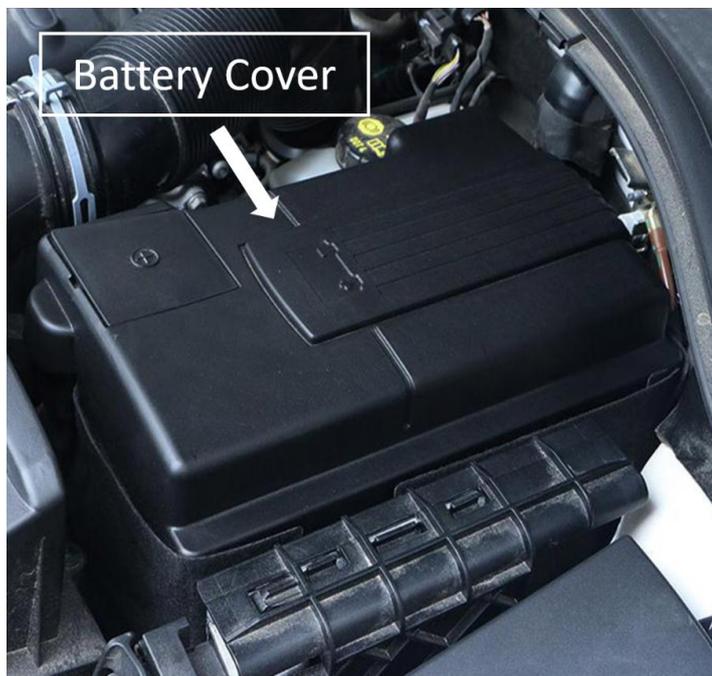


Figure 14: An example of the car battery cover (For the actual part, see Article III).

The NFPC part design was closely linked with designing the mold for compression molding since it was important to keep in mind the considerations for the mold design mentioned in Chapter 2. Additionally, the aim was to design a simple yet effective NFPC part for the desired application. A simple part design was crucial to keep the mold simple and lower the cost. This was mainly from the perspective of keeping the overall cost of operation low since a complex mold could be comprised of multiple pieces and subassemblies. This could directly increase the production time of the mold and part with

the mold-making cost since special tools and cutters would be required for complex shapes. Additionally, a high-cost mold would not complement the low cost of the natural fibers and recycled thermoplastic waste used as raw material in this study. Figure 15 shows both upper and lower mold in the closed stage.

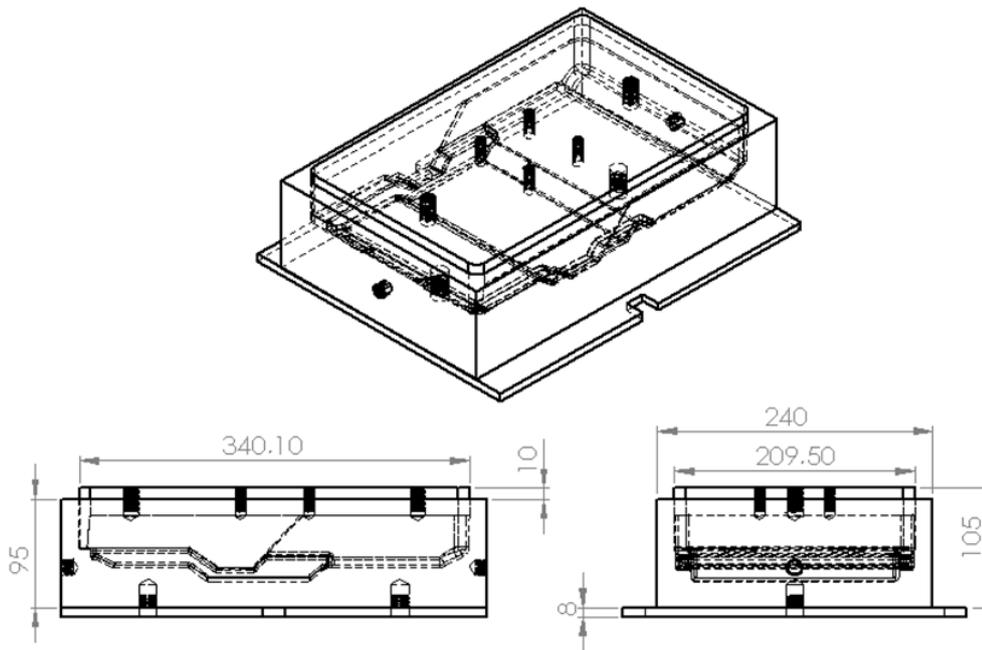


Figure 15: Design of the mold for compression-molded NFPC car battery cover (all units are mm).

Another mold design consideration was the clearance between the upper and lower mold. Since it is of utmost importance that the upper mold could easily slide into the lower mold. Additionally, the clearance would also act as a vent for gas and excess composite material to flow out of the mold when fully compressed. Normally the clearance in a compression mold is between 0.05–0.15 mm. Therefore a clearance of 0.7–0.8 mm on all four sides was used. Furthermore, the clearance also provides additional protection against the collision between the upper and lower mold when the press brings the upper mold down. Figure 16 shows the edges of both upper and lower mold. It can be seen that the edges on both upper and lower mold are curved and not kept sharp. This was important to ensure the smooth and fast flow of material inside the mold once the compression molding began. This was mainly because, after recycling, the melt flow index of the polymer material decreases and could further decrease due to the presence of impurities in the recycled material and due to the addition of fibers to the polymer material. Additionally, the composite material starts to cool rapidly as soon as it comes into contact with the lower mold due to the conduction process. This rapid cooling is more prone to

occur on the edges since the amount of composite material is at a minimum at the edges during the compression stage. Finally, in case of a collision where upper mold collides with lower mold, the presence of curved edges could also minimize the damage caused during the collision.

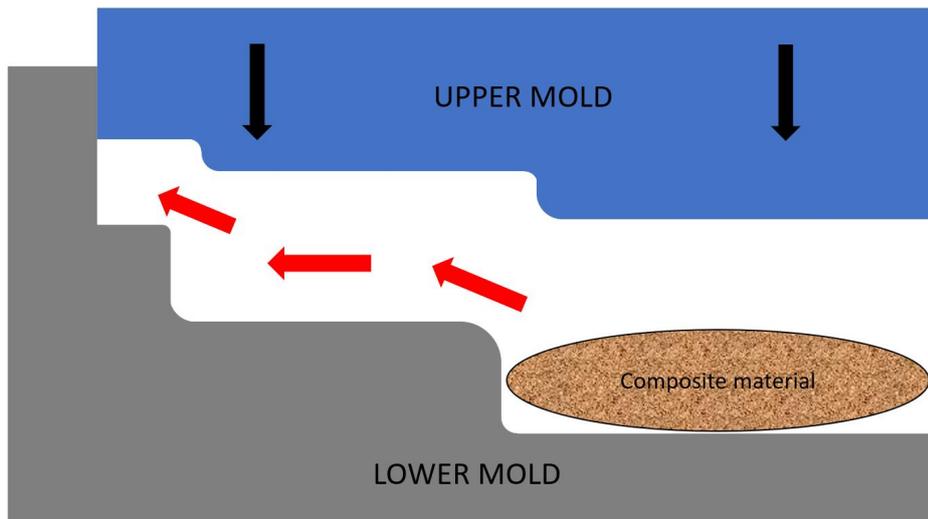


Figure 16: Cross-section view of the upper and lower mold showcasing curved edges.

One major challenge faced during the manufacturing of the NFPC component was the counterforce from the lower mold applied against the force applied by the upper mold. This was mainly due to the difference in the height of the horizontal surfaces of the lower mold as shown in Figure 17. The composite material was placed at the bottom of the lower mold, which was then moved to the sidewalls of the mold once the compression process commenced. The disparity in the heights of the horizontal surfaces of the lower mold led to a non-uniform filling time. This led to some horizontal surfaces filling faster and blocking the upper mold downwards compression action, which in turn resulted in the uneven filling of the material due to less compression force exerted in certain locations of the mold and the rapid cooling of the composite material at the same time. Additionally, in case any extra composite material was present in the mold, it was found that it tended to collect on these horizontal surfaces and solidify instead of coming out of the mold through any runoff channels.

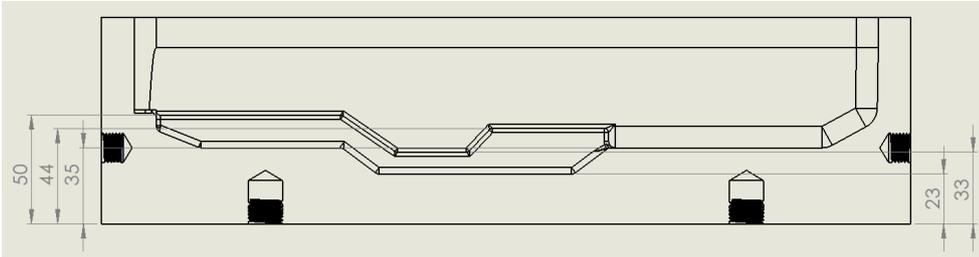


Figure 17: Variation in the height of the mold horizontal surfaces (all units in mm).

One solution suggested for the height disparity of the horizontal surfaces and non-uniform filling of the mold was to make the upper mold work in multiple stages, where different parts of the upper mold would come down at different times and lock in at the desired height, thereby ensuring no extra material could flow and solidify at that particular horizontal level since there would not be any additional space for the material. A small example of such a design is shown in Figure 18. However, it is essential to note that such a mold setup is complex to assemble, operate and maintain due to the multiple separate and independent moving parts involved during the process. Furthermore, due to the moving parts and other sub-assemblies, the overall mold cost would be higher significantly. This directly conflicted with our aim to produce a cheap and simple molding system.

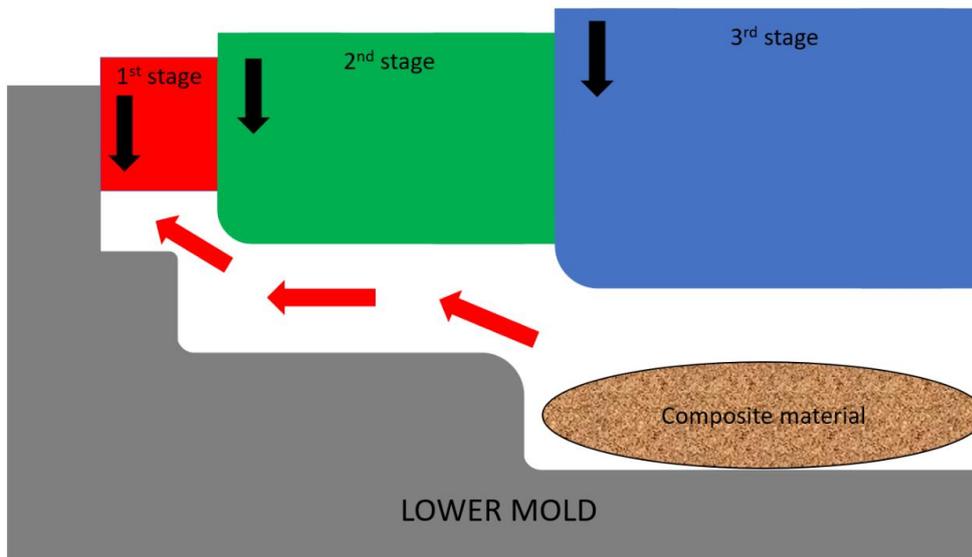


Figure 18: Proposed solution for the mold.

Another crucial challenge faced was the heating of the mold. The mold temperature was crucial for the smooth flowability of the composite in the mold. This is because if the mold temperature was too low, the heated composite material would start to cool the moment it comes into contact with the lower mold due to conduction. Therefore, to avoid the rapid cooling of the composite material both the upper and lower mold were manually heated using a hot air blower up to 60–70 °C. A solution suggested for this problem was the addition of a heating element such as a coil or plate in the lower mold which is quite normal in the compression molding process. However, to ensure simplicity and low cost of the mold no heating element was implemented. For large-scale operation, a heating element would be necessary since it would make the process more automated and increase productivity which in turn could offset the increase in the mold cost incurred by the addition of heating elements.

4.2.1 Compression testing of the demonstration NFPC part

After the demonstration NFPC part was successfully produced using the newly designed compression mold, compression testing of the NFPC part was performed. The average elastic modulus value of the NFPC part with the R-ABS/10WF composite blend was 3156.43 MPa. For comparison, a similar part was also produced using R-ABSmix and the average elastic modulus value was found to be 1568.68 MPa. Since the elastic modulus of a material defines the stiffness or rigidity of the material, this clearly indicates the addition of fibers as the reinforcing material in the polymer matrix increased the rigidity of the structure by 101%. A similar increase in the compressive properties of the NFPC was observed in numerous studies (Vijay, Singaravelu 2016, Kumar, Srivastava 2017, Singha, Rana 2012). Additionally, the R-ABS/10WF composite part also exhibited less deformation in the structure compared to the R-ABSmix part as shown in Figure 19. This indicated that the addition of fibers had a positive impact on both the load-bearing capacity of the structure and the deformation level.

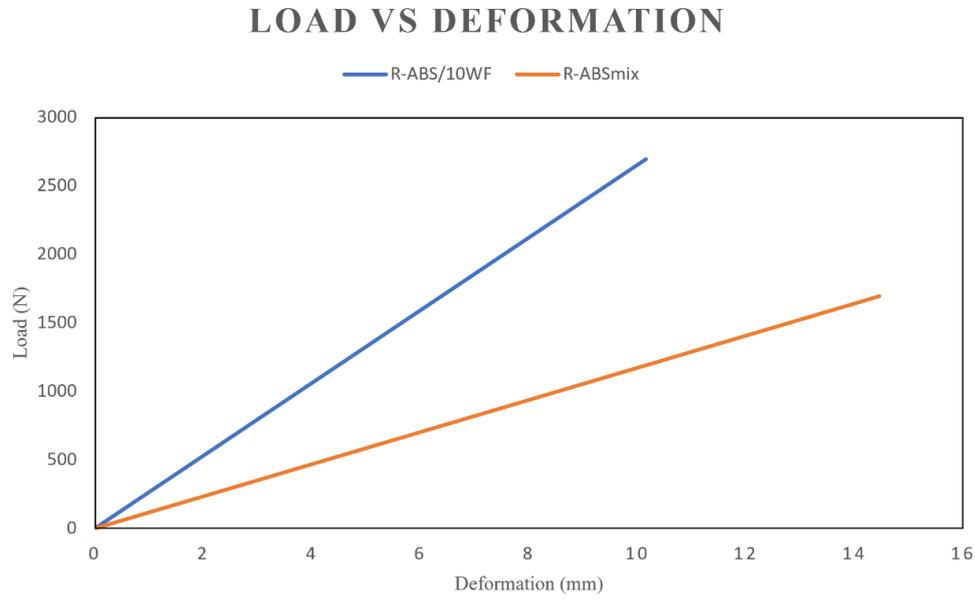


Figure 19: Compression testing results for the R-ABS/10WF and R-ABSmix parts.

4.3 Optimizing process parameters for NFPC compression molding

The next stage of the study was focused on optimizing the processing parameters for the compression molding process of the demonstration NFPC part. As stated earlier, the processing parameters can be optimized for some quality characteristics such as warpage, shrinkage, tensile strength, impact strength, flexural strength, or cycle time. For this study, warpage was selected as the quality characteristic. Warpage and tensile strength are common quality characteristics for NFPC molded parts (Azaman, Sapuan et al. 2015, Sudsawat, Sriseubsai 2017). Since the warpage can severely affect the quality, aesthetics, and application of the molded part, minimizing the warpage was set as the aim. Autodesk Moldflow® software was used to help optimize the processing parameters by simulating the flow of the material inside the mold. The software also provided a warpage measuring capability which can speed up the research work since no additional test setup is required.

Since time, temperature, and the compression force are three crucial processing parameters in compression molding, the mold temperature (A), compression time (B), melt temperature (C), and compression force (D) were selected for optimization. In this study, the melt temperature was defined as the material temperature after it is heated in the oven. A Taguchi L9 OA analysis was implemented using the above-mentioned four parameters and their three levels as described in the methodology section. The L9 OA and the corresponding warpage results are shown in Table 7.

Table 7: Warpage results of the simulation.

Exp. No.	A	B	C	D	Warpage (mm)
1	1	1	1	1	1.506
2	1	2	2	2	1.496
3	1	3	3	3	1.521
4	2	1	2	3	1.512
5	2	2	3	1	1.530
6	2	3	1	2	1.517
7	3	1	3	2	1.575
8	3	2	1	3	1.502
9	3	3	2	1	1.526

After the Taguchi process, the warpage result was transformed into an S/N ratio using smaller-the-better quality characteristics since the aim was to reduce the warpage. For the smaller-the-better quality characteristics,

$$S/N = -\log(MSD), \quad (4.1)$$

where MSD is the mean-square deviation for the output quality characteristics and can be expressed as:

$$MSD = \frac{1}{N} (\sum_{i=1}^n Y_i^2), \quad (4.2)$$

where Y_i is the value of warpage for the i th test, n is the number of tests, and N is the total number of data points.

The S/N ratio response diagram for all four processing parameters is shown in Figure 20. The optimum parameters to minimize the warpage can be identified as the highest value from each parameter. Therefore, the optimal processing parameters obtained were mold temperature 60 °C (A1), compression time 40 sec (B2), melt temperature 210 °C (C1), and compression force 600 kN (D3).

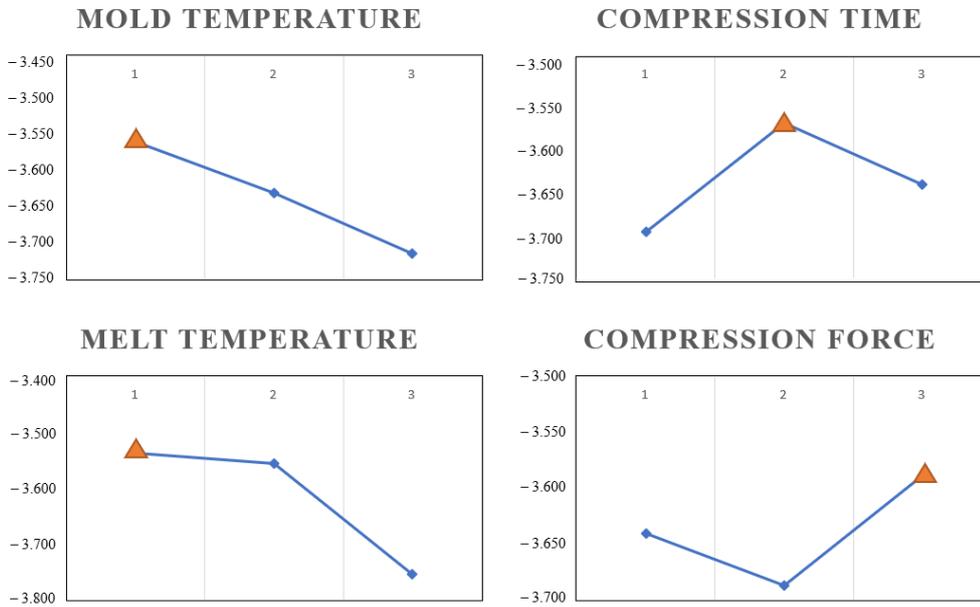


Figure 20: S/N ratio response graph for processing parameters.

Figure 21 shows the optimal temperature result for the NFPC molded part obtained in the study and the other literature. From the temperature result, it was observed that 210 °C was the highest temperature compared to the literature. This could be attributed to the relatively high melting temperature of ABS polymers and also due to the presence of impurities in the recycled thermoplastics.

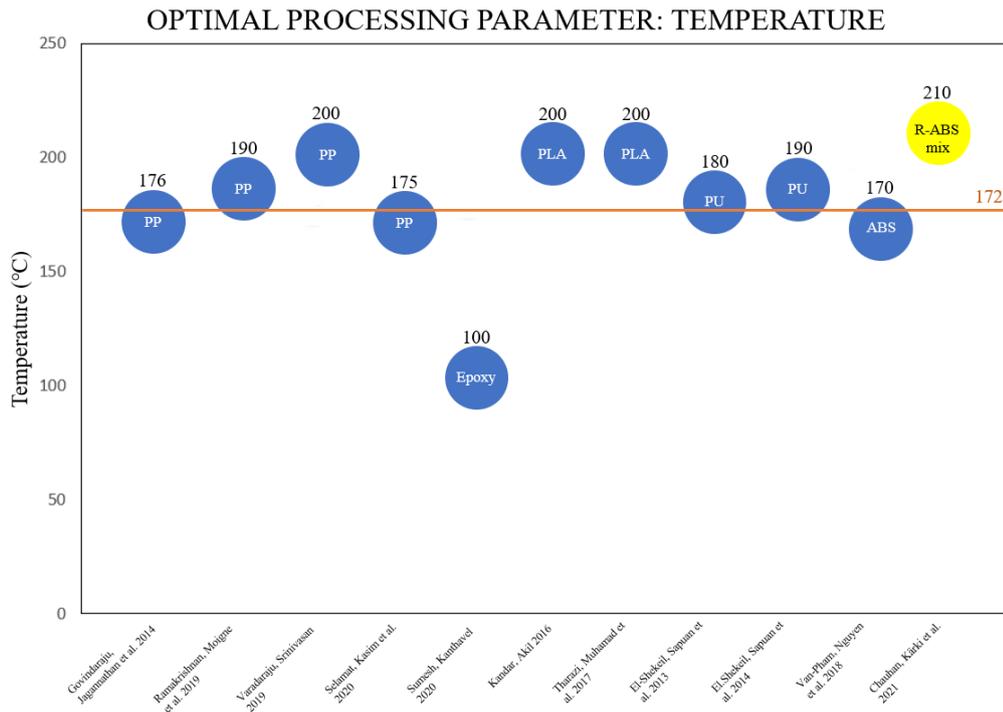


Figure 21: Optimal temperature results for compression-molded NFPC.

The result for the optimal compression time is shown in Figure 22. The optimized compression time obtained from the study was 40 sec in comparison with other studies where the compression time was recorded between 3–20 min. One reason for this deviation from the time in the literature is attributed to the choice of quality characteristics since in this study warpage was used as the quality characteristic for optimizing the process parameters, while in other studies it mainly was tensile or impact strength. Overall, from point of view of productivity, such a low compression time is an excellent positive aspect since time savings during the compression molding process directly leads to higher productivity.

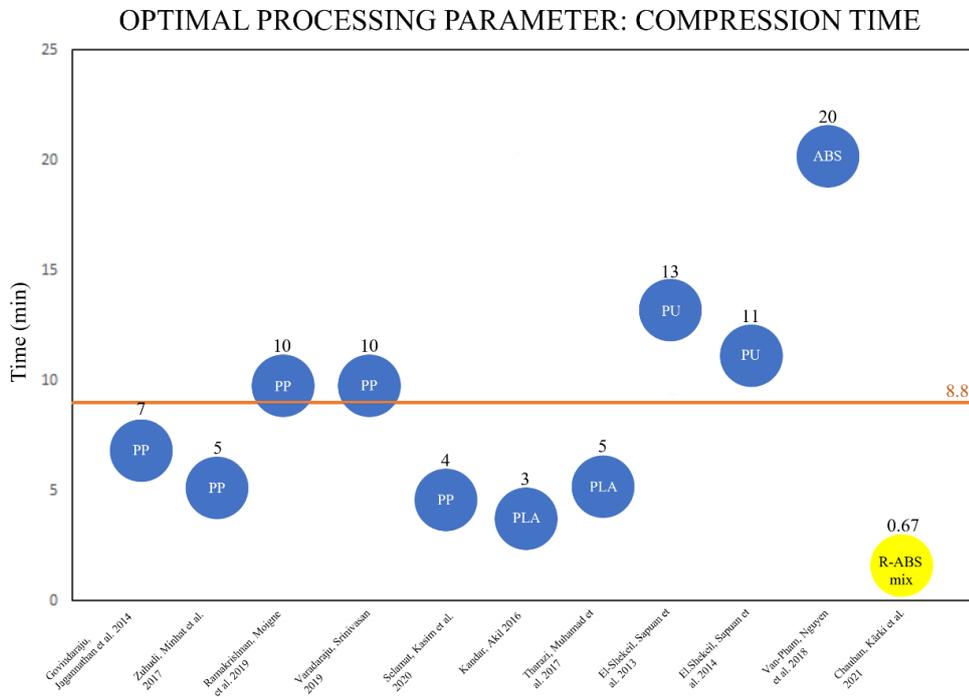


Figure 22: Optimal compression time results for compression-molded NFPC.

The output from the Stenhøj hydraulic press was in the form of force in kN. However, the majority of the existing literature work used pressure in bar as one of the processing parameters in the optimization process. Therefore, to generalize the result with those in the existing literature, the result was converted into bar using equation 4.3. It is also important to understand that converting the literature result into kN would have been a difficult task since the surface area of the parts used in the various literature is an unknown quantity. Meanwhile, in our study, the automotive part was designed in Solidworks and the surface area of the part (0.195 m^2) could be obtained. Finally, the pressure came out to be 3076.92 kN/m^2 which equated to 30.76 bar.

$$\text{Pressure} = \text{Force}/\text{Area} \quad (4.3)$$

Figure 23 shows the optimal pressure or compression force result for the NFPC compression molding. The result of the study was in line with a few other results from the literature. However, there were not many literature references showing high amounts of pressure for NFPC compression molding. This could be due to several factors such as the fiber type, fiber content, polymer type, and due to the availability of the press brake in the research lab. For example, in our study, we had a Stenhøj 40-ton hydraulic press which was used to produce samples for the compressive testing in Article III.

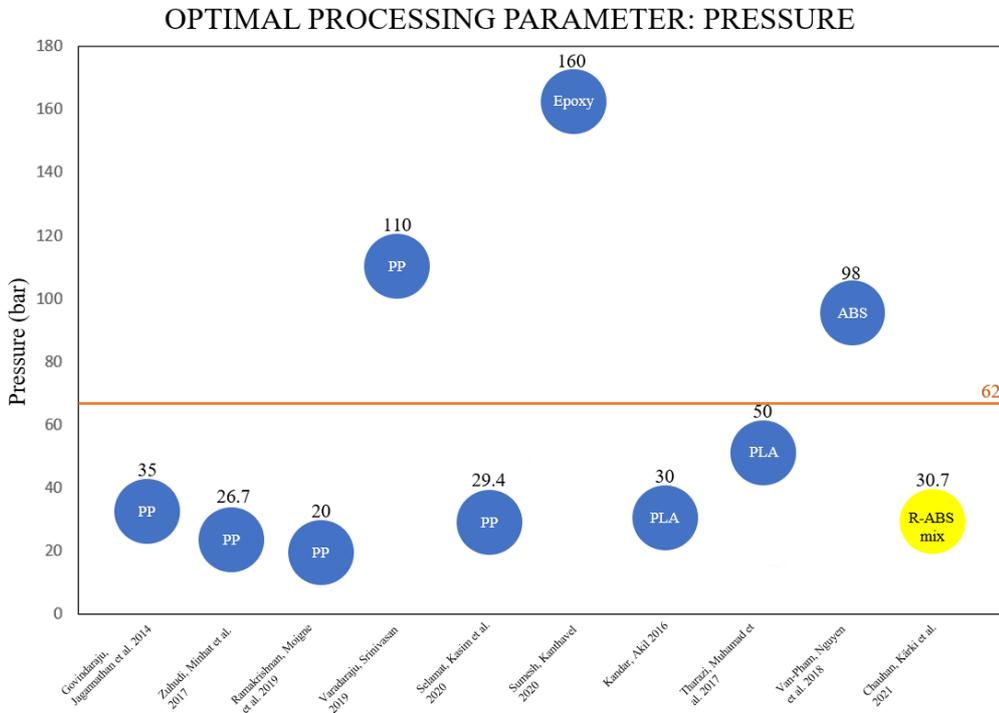


Figure 23: Result of optimizing pressure of compression-molded NFPC.

Later an ANOVA procedure was used to determine the significance of each parameter for the warpage. This was important in understanding which amongst all the processing parameters had the most significant effect on the chosen quality characteristics. ANOVA results consist of the sum of squares (SS), degree of freedom (DOF), variance (V), F-ratio, and percentage contribution (P%). The results of ANOVA are shown in Table 8, and a high P% value indicates a highly significant parameter.

Table 8: ANOVA results.

Parameter	DOF	SS	V	F-ratio	P%
Mold temperature	2	0.00107	0.000535	0.985472	24.73
Compression time	2	0.00071	0.000353	0.585604	16.33
Melt temperature	2	0.00208	0.001041	2.782957	48.12
Compression force	2	0.00047	0.000234	0.363903	10.82

Based on ANOVA results, the order of significance of parameters is as followed: melt temperature > mold temperature > compression time > compression force. The significance of each parameter on the warpage value is shown in Figure 24. From the figure, it is clear that temperature (the mold and melt temperature) exhibited the most effect on the warpage value of the compression-molded NFPC part. The percentage share of both temperatures was approximately 75%, followed by the compression time and lastly, the compression force.

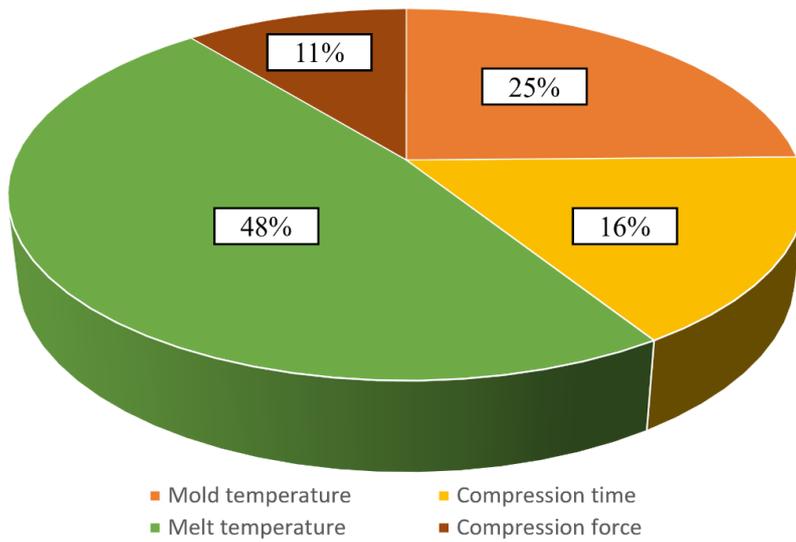


Figure 24: Significance of process parameters on warpage value.

4.4 Cycle time calculations

Initially, the demonstration compression-molded NFPC part was produced using the following parameters: melt temperature = 200–205 °C, heating time = 15 min, compression time = 120 sec, compression force = 400 kN, and mold temperature = 60–70 °C. For the optimization, process parameters such as melt temperature, compression time, mold temperature, and compression force were considered. The optimal parameters obtained from the study were: melt temperature = 210 °C, compression time = 40 sec, mold temperature = 60 °C, and compression force = 600 kN. The melt temperature and mold temperature were almost the same for the initial and optimal parameters. However, after optimization, the compression time was reduced from 120 sec to 40 sec, whereas the compression force was increased from 400 kN to 600 kN, as shown in Figure 25. This implies that using optimal process parameters, the cycle time was reduced by 66.67% compared to the initial set of parameters, while the compression force was increased by 50%.

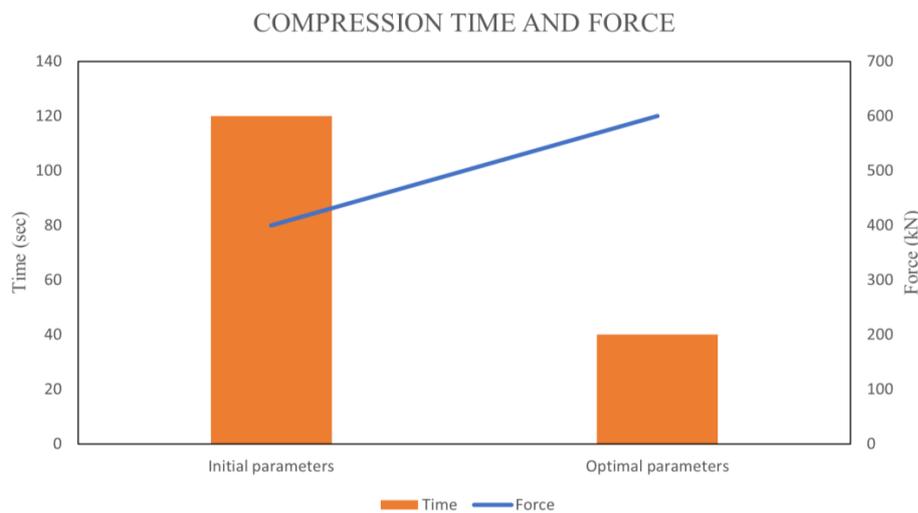


Figure 25: Compression time and force for initial and optimal process parameters.

The compression time is a crucial part of the process cycle time. The cycle time is defined as the time taken to complete one job from the start of the production process until the product is completely ready. Therefore, reducing the cycle time is crucial for improving productivity and thereby increasing profit margins. In the study, the cycle time of the compression molding process was comprised of the material loading and unloading time and the compression time. The material loading and unloading time was 120 sec and were similar for both the initial and optimal parameters. Therefore, the cycle time for the initial parameters was 240 sec, and for the optimal parameters, it was 160 sec. Considering an 8-hour working shift with a single shift and a 5-day working week, approximately 600

pcs can be produced with the initial parameters, and with the optimal parameters, approximately 900 pcs can be produced weekly. Therefore, increasing the productivity of the process by 50%.

5 Conclusion

The aim of the doctoral dissertation was to explore the means to utilize ELV thermoplastic waste for recycling it back into automotive applications in the form of NFPC. The study compiled multiple facets such as material science, new part development, mold design, process parameters, and statistical analyses. For this, the mechanical properties of the composite materials, optimal fiber content, surface treatment methods, automotive part design, and processing parameters of the compression molding process were examined and documented in the study. Additionally, statistical analysis tools such as the Taguchi method and an ANOVA test were employed in optimizing the compression molding process parameters for manufacturing a demonstration NFPC automotive component. The selection of the compression molding process for manufacturing the NFPC automotive component over injection molding or extrusion was due to its low cost and high-volume production. This was also because the overall idea of the study was to keep the cost of the production low due to the low cost of the raw materials used in this study. Finally, the impact of optimizing the process parameters on the cycle time and productivity was reported in the study.

The study is mainly targeted at researchers and design engineers focused on exploring applications for ELV thermoplastic waste. Many previous studies have examined the utilization of thermoplastics and natural fiber composites in various automotive applications. The novelty of the study is from the point of view of using ELV thermoplastic waste in the form of NFPC in automotive applications. Additionally, unlike previous studies, which have predominantly focused on interior or exterior automotive applications of NFPC materials, this study is focused on under-the-hood applications. The lightweight nature and low cost of the raw materials used in this study are also desirable for automotive applications. By replacing heavier automotive components with a lightweight material such as NFPC, the fuel consumption of the vehicle can be improved, which in turn will result in lower emissions. Additionally, using recycled material instead of virgin material is a direct cost saving for car manufacturers. Thus, this study will be especially interesting to car manufacturers and governmental and environmental regulatory bodies. Furthermore, low emission vehicles allow car manufacturers to market them as a more eco-friendly and greener solution in comparison to their competition and thereby giving them a competitive edge in the market. Similarly, governmental and environmental bodies are satisfied with more eco-friendly vehicles on the roads leading to less harm caused to the environment and local surroundings.

This study reports the best fiber-composite blends with good mechanical properties for the manufacturing of an automotive demonstration part. Similar to the results reported in other studies, the strength values of the composite blend decreased significantly with the addition of fibers, which was mainly due to the incompatibility of fibers and polymer material. This decrease was further noticeable when recycled thermoplastic material was used due to the presence of impurities and possible degradation of the polymer material

over its lifecycle. However, certain surface treatment methods such as silane can improve the mechanical properties of the composite blend by improving the interfacial adhesion. In this study, a composite blend with 10% wood fibers without silane was selected over another equally performing silane-treated composite blend with 10% palm fiber. The 10% wood fiber blend exhibited low density and decent tensile and impact strength values with less deviation. The low density of the composite material used was also helpful in producing a lightweight automotive part which would reduce the fuel consumption of the vehicle and thereby reduce the emissions from the vehicle. Therefore, the same composite blend was then later used as the raw material in the manufacturing of the demonstration NFPC automotive part. Before manufacturing, the part was redesigned to make it more suitable for the NFPC material and compression molding process. The compression mold was then designed and machined in-house, and the challenges and the design considerations undertaken during the mold design phase were documented in the study. It was crucial to keep the compression mold simple and the cost low to meet the overall theme of the study with the raw materials consisting of recycled ELV thermoplastic waste and low-cost wood fiber, which in the end was successfully implemented. The challenges and design considerations can help future designers to better understand how to approach mold design for NFPC materials and can save crucial time used in part and mold design. The automotive demonstration part was successfully fabricated using a simple and low-cost compression mold, and the compressive testing performed on the automotive part also yielded positive results. It was observed that the elasticity of the part increased by 101% after the addition of 10% wood fibers to the polymer matrix. Later, the compression molding process used to fabricate the NFPC automotive part was optimized using the Taguchi DOE technique and ANOVA analysis. The optimal processing parameters were: mold temperature = 60 °C, compression time = 40 sec, melt temperature = 210 °C and compression force = 600 kN. Amongst these, the melt temperature was the most significant processing parameter. Finally, optimal parameters were found, which were impactful in reducing the cycle time of the NFPC part by 67%, which in turn resulted in improving the overall productivity of the process by 50%. Any improvement in the productivity of the process is desirable by any manufacturer since an increase in productivity directly correlates to increased revenue and thus more profits.

The limitations of the study are mainly related to the raw materials and the processing method. Since the major raw material was ELV thermoplastic waste composed of ABS and various other ABS blends, it can be challenging to obtain a similar material composition with the ELV thermoplastic waste available at other locations. Additionally, the presence of different types of impurities in ELV thermoplastic waste depends on the environment in which waste material is kept or used previously. Additionally, the study is limited in terms of the type and size of fibers and other additives used in the composite blends since using other fibers and additives could possibly change the outcomes of the study. Finally, the processing method of the study was limited to compression molding, and using other common NFPC manufacturing techniques such as injection molding, extrusion, or resin transfer molding could possibly yield different results.

Future studies related to understanding the detailed chemical interaction of fibers, polymers, and additives in the composite material will be crucial to ensuring a uniform composite property since these materials primarily affect the properties of the composite. It would also be crucial to study and ensure a steady supply of raw material, especially the ELV thermoplastic waste and material composition including the presence of impurities in the waste material since they can heavily influence the final material behavior. A detailed study on the investments and other cost-related actions such as production, storage, quality, and transportation would be helpful from the manufacturers' point of view. Finally, a life cycle study could also further shed light on the ecological impact caused by the entire supply chain when using NFPC materials instead of traditional automotive raw materials.

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Publication I

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Review of natural fiber-reinforced engineering plastic composites, their applications in the transportation sector and processing techniques

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Review of natural fiber-reinforced engineering plastic composites, their applications in the transportation sector and processing techniques

Vardaan Chauhan , Timo Kärki and Juha Varis

Abstract

Interest in natural fiber-reinforced polymer (NFRP) composites is growing rapidly in the transportation sector, especially as a replacement material for metals and synthetic fiber composites. The heightened interest is directly related to a need to produce lightweight and fuel efficient vehicles. Further, stringent legislation and greater environmental awareness is forcing transportation industries to select materials with a smaller carbon footprint. In such a context, NFRP composite materials are a good choice due to their low cost, low environmental impact, and relatively equivalent properties to metals and other composites. Most prior studies have examined commodity plastics such as polypropylene, polyethylene, and epoxy as the primary polymer matrix in NFRP composites and little work has addressed engineering plastics. Engineering plastics, which includes polycarbonate, polyamides, and polystyrene, are high performance thermoplastics with superior properties but relatively higher cost than commodity plastics. It has been claimed that even after recycling, engineering plastics properties are superior to those of commodity plastics, and thus, utilization of recycled engineering plastic in NFRP composites can help reduce waste and lower composite material costs. The aim of this review article is to explore the current status of engineering plastics reinforced with natural fibers such as flax, hemp, jute, and sisal and to examine their use in automotive, aerospace, and maritime applications. Properties and processing techniques of engineering plastics reinforced with natural fibers are also studied.

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Introduction

Transportation is a vital part of the economy and the transport of goods and people from one place to another is hugely dependent on oil and other petroleum-based fuels. A recent report by OPEC in 2017 estimated that worldwide demand for oil for road, rail, air, and maritime transportation was 43.6 mb/d (million barrel per day), 1.8 mb/d, 6.3 mb/d, and 4 mb/d, respectively. As oil is a nonrenewable resource, it is important to restrict its consumption to a minimum, especially as the number of passenger vehicles is projected to rise from 1102 million in 2017 to 1980 million by 2040 alongside a rise in commercial vehicles from 230 million to 462 million during same time period.¹

The transportation sector is responsible for about 23% of total global CO₂ emissions, with road transportation contributing about 72% of emissions, while air and water transportation contribute about 11% each.² Both fuel consumption and CO₂ emissions can be reduced by reducing vehicle weight. A 10% reduction in total vehicle weight leads to an approximate 7% improvement in fuel efficiency and every 1 kg of weight reduction results, on average, in 20 kg less CO₂ being released to the atmosphere.³⁻⁵ Consequently, vehicle manufacturers are making great efforts to reduce vehicle weight by changing the materials used. Further, due to stringent guidelines and legislation in the European Union and Asian countries regarding automotive end-of-life requirements, automobile makers need to consider the ecological impact for the entire lifecycle of the vehicle, including raw material production, vehicle manufacturing, usage, and disposal.⁶⁻⁸ The transportation sector, especially automobile companies, has demonstrated the capability to shift from steel alloys to aluminum alloys and there is now a move from aluminum to fiber-reinforced polymer (FRP) composites in certain applications. Some researchers are already predicting a future where the majority of vehicle weight is composed of polymer composites reinforced with fibers.⁹

FRP composites have been extensively used in applications such as construction for many years, and their market share is growing in a number of different sectors. FRP composites are composed of fibers acting as reinforcement or filler and a polymer matrix. The fibers can be either synthetic fibers, such as glass, carbon, and aramid fibers, or natural fibers, such as plant, mineral, or animal fibers. Synthetic fiber-reinforced polymer composites are widely used in high performance applications such as the aerospace industry and automobiles due to their superior mechanical properties and lightweight. However, these fibers are expensive, with the exception of glass fibers, require large amounts of energy in production, and are not eco-friendly. Thus, their extensive usage can lead to severe ecological problems if not recycled properly.¹⁰ Hence, increased societal pressure to reduce dependency on nonrenewable resources, while simultaneously ensuring cost competitiveness and, reducing energy consumption in production, together with changes in environmental ecological legislation and greater ecological awareness have shifted the focus to natural fibers as a

reinforcement or filler material^{10–13} and natural fiber-reinforced polymers (NFRPs) are gaining increased attention in both the academic and industrial world. Natural fibers like flax, hemp, jute, and sisal are commonly used as reinforcement in polymer composites due to their desirable properties and easy availability.¹⁴ For comparison, a composite part produced with natural fibers will be lighter than one made of glass fibers, mainly due to the difference in their densities, as natural fibers density is between 1.2 and 1.6 g/cm³ and that of glass fibers is between 2.16 and 2.68 g/cm³.^{15,16} Furthermore, production of a flax fiber mat consumes about 82% less energy than production of a glass fiber mat.⁶ Additional benefits of composites made up of natural fibers over synthetic fibers include low cost, renewability, recyclability, biodegradability, carbon neutrality, nonabrasive quality, limited health risks, acoustic, heat and electrical insulation qualities, and ease of dispose.^{5,6,11,13,17–19} On the other hand, natural fibers composites have a number of drawbacks, such as high moisture content, low fire resistance, weak interfacial adhesion, poor resistance to microbial attack, and a need to use low processing temperatures.^{14,20,21} Many of these problems can, however, be resolved with appropriate surface treatment techniques. Interestingly, utilization of NFRP composites varies from region to region, for example, the NFRP market in United States is dominated by structural applications such as fencing, decking, and railing, whereas in Europe NFRP composites are used primarily in automotive applications.²²

The polymer matrices in a NFRP composites can be essentially classified into thermoplastic matrices or thermoset matrices.^{14,23} Thermosets are nonrecyclable polymers possessing stronger bonds and stiffness than thermoplastics. They retain their dimensional stability under high temperature and thus cannot be remolded or reshaped.²³ Thermoplastic polymers can be softened or melted by heating and hardened after cooling, which means that they can be easily remolded into different shapes. In addition, they possess high impact resistance, are easy to recycle, and are chemically inert.^{3,23,24} Furthermore, the processing time of thermoplastic is faster than thermosets^{3,24} and they can be easily joined using by welding techniques such as resistance welding, vibration welding, and ultrasonic welding.³ The most common thermosetting polymers used in NFRP composites are epoxy, polyester, and phenolic, and common thermoplastic polymer matrices used are polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyvinyl chloride (PVC).^{3,7,14,25,26} Polymer resins such as PP, polyamides (PAs), PE, and polycarbonate (PC) are most commonly used in the transportation sector.⁷ Since thermoplastic polymer can be easily recycled and is used in a wide range of applications in the transportation sector in automobile, railway coach, and aircraft manufacture and in other industrial sectors, the use of recycled plastic matrices in place of virgin polymer matrices can help tackle environmental problems.^{7,14,23} Thermoplastic polymers in NFRP composites are further classified into commodity or traditional plastics such as PP and PE and engineering plastics such as acrylonitrile butadiene styrene (ABS), PA, and PC. While the market is heavily dominated by commodity plastics with 80% of total plastic consumed being commodity plastic,²⁶ engineering plastics offer lightweight, flexibility, and higher performance. The market value of engineering plastics market is projected to reach 90 billion USD by 2020. The top three market sectors for engineering

plastics are electrical and electronics products, consumer goods, and transportation. However, there are regional differences, and in Western Europe the largest market for engineering plastics is the transportation sector, whereas in the Asia-Pacific region it is consumer goods such as toys, electrical goods, and household utensils.²⁷

NFRP utilization in the transportation sector has been investigated by several researchers, who have studied the characteristics and properties of different NFRP composites, for example, fiber modification, thermal stability, crystallinity, and biodegradability. However, the majority of work has focused on commodity plastics or thermosets reinforced with natural fibers and few studies have considered engineering plastics reinforced by natural fibers and, specifically, their application in the transportation sector. Furthermore, the main focus of research has been on virgin plastics, in both thermoplastics and thermosets study, and not much work has been published related to the use of recycled plastics in NFRP composites and their characteristics and applications potential. Increased usage of engineering plastics and the replacement of traditional materials such as wood and metal in engineering applications with alternative materials underline the importance of exploring this group of plastics for future potential.

The objective of this article is to examine the current status of the engineering plastics reinforced with natural fiber and their application in the transportation industry. The review is divided into five sections describing types of engineering plastics, types of natural fibers, NFRP in the transportation section, properties of NFRP, and processing of NFRP components.

Engineering thermoplastics

Engineering plastics are of a high performance segment of synthetic plastics with excellent thermal and mechanical properties in comparison to commonly used commodity plastics. However, they are generally more expensive than standard plastics.^{28,29} Engineering plastics are commonly amorphous or crystalline in nature.²⁹ They exhibit characteristics such as excellent strength, good temperature resistance, toughness, stiffness, chemical resistance, lightweight, and wear and abrasion resistance, and they can be easily formed into parts.^{28–30} These advantageous features enable extensive use in electrical goods and electronics manufacturing, consumer goods, and transportation applications where they can easily substitute metal components due to their lightweight and similar properties to metal.²⁸ Worldwide growth in the automotive industry and increasing demand for electrical and electronic goods are expected to promote usage of engineering plastics in those sectors. The most commonly used engineering plastics in the automotive industry (Figure 1) and other transportation sectors are ABS, PA, PC, polyethylene terephthalate (PET), PS, and polybutylene terephthalate (PBT).

Acrylonitrile butadiene styrene

ABS is a thermoplastic terpolymer.³² It consists of styrene–acrylonitrile (SAN) copolymer mixed or grafted with polybutadiene.^{28,33} It is an amorphous polymer and thus does not have a melting point.³⁴ ABS possesses features such as good impact strength,

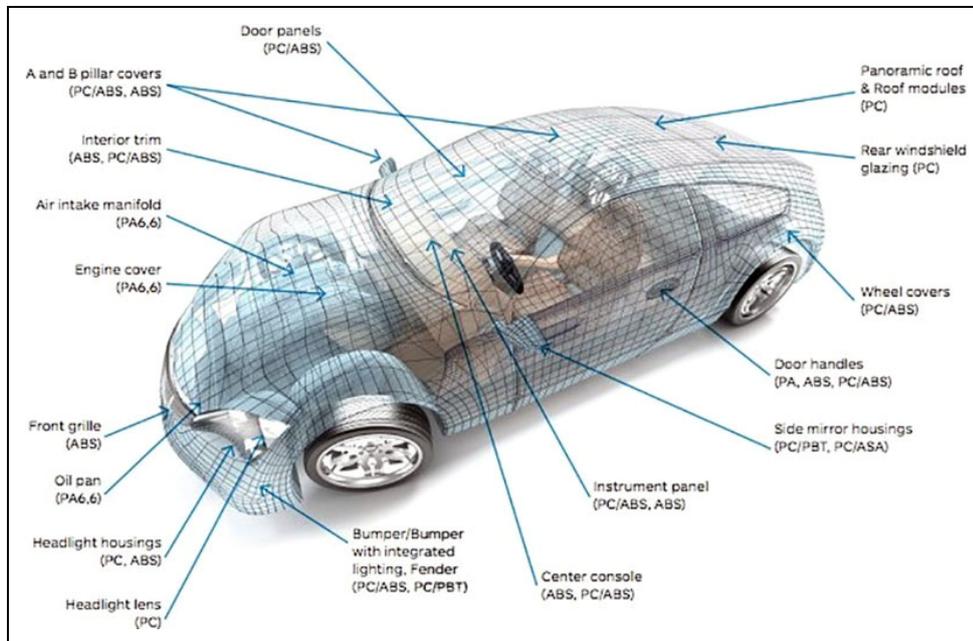


Figure 1. Engineering plastics used in an automotive vehicle.³¹

toughness,^{30,32,35–37} chemical resistance,^{30,32,35,37} heat resistance,^{28,37} adhesion properties,^{32,35} and rigidity.^{32,37,38} ABS is opaque³⁶ and easily processed,^{30,32,35,37,38} and it can be painted³² and recycled.³⁵ Its use is widespread in applications such as housings for electronic appliances,^{30,36,39,40} automobile body parts, wheel covers, instrument panels and interiors,^{30,35,36,38,39} pipes,^{30,36,38} toys,^{35,36} computer and information and communication technology (ICT) components,^{30,35,36,40} as well as medical devices^{30,35} and safety helmets.^{30,36}

Polyamides/nylon

PAs are commonly known as nylons. These polymers can be further classified into PA 6 (Nylon 6), PA 66 (Nylon 66), Nylon 46, Nylon 69, Nylon 610, Nylon 612, Nylon 11, and Nylon 12.²⁹ These polymers exhibit high temperature resistance,³⁰ flexibility, resistance to warping and abrasion,^{29,30,41} toughness, hardness,^{29,30} high strength, and high gloss. They can also be dyed.⁴¹ In addition, they also possess high burst strength when processed into tubes or pipes. Moreover, due to their resistance to bodily fluids and non-toxicity, they do not cause skin or tissue inflammation when used in medical devices such as dilation catheters. Some PA grades are used in blood transfusion equipment as connectors and stopcocks.²⁹ Other applications include: clothing—in socks, lingerie, sportswear,⁴¹ and stockings^{36,41}; household goods—carpets, upholstery,^{36,41} furniture casters, and toothbrush bristles³⁶; and industrial products—rope, gears, bearings, tubes, protective cloths,^{36,41} bushing, sprockets, containers, cables, fishing lines,³⁶ zippers,

pulleys, and parachutes.⁴¹ The automobile sector is the largest consumer of PAs accounting for 35% of PA consumption.²⁷ PAs are used in tires, seat belts, fan blades,⁴¹ car upholstery,³⁶ and in under-the-bonnet applications such as parts of the engine compartment, air-intake manifold,⁴² fuel systems, cooling systems, switch housing,³⁰ and oil pans.⁴³

Polycarbonate

PC is an amorphous polymer with excellent optical transparency.^{36,44–47} It possesses characteristics such as superior toughness,^{30,36,44,45,47,48} high impact strength,^{30,36,37,44,46,48} rigidity,^{36,44} fire resistance,^{28,37} high dimensional stability,^{28,30,37,44,46} high gloss finishing,^{44,47} moderate temperature resistance, low moisture absorption, low mold shrinkage, resistance to corona discharge,⁴⁶ high heat deflection temperature (approximately 130°C)⁴⁷ and it is extremely lightweight.⁴⁸ It is used in applications such as safety hats, helmets, power tool housing, riot shields, automotive sunroofs,^{36,44} aircraft windows,⁴⁴ bulletproof windows, automobile headlights, greenhouse enclosures,^{44,48} and in the electronics sector—in computer housing, housing of cell phones, laptops, pagers, and other ICT equipment^{40,48} and for compact disks.^{36,44,45,48} In comparison with PVC, it is less toxic, recyclable, and eco-friendly.⁴⁸

Polyethylene terephthalate

PET is a thermoplastic polymer belonging to the polyester family and is semi-crystalline in nature.^{49,50} PET exhibits transparency to both microwaves and visible light, heat resistance, high impact strength, lightweight,^{50,51} toughness, and easy moldability and it is impermeable to water and CO₂.^{36,50,51} The main applications of PET are in drinks bottles,^{49,50,52} food containers, and fleece garments.⁵⁰ Additionally, it is also used in electrical connectors, credit cards, photo and video tapes, packaging film, and X-ray films.^{36,52} In the transportation sector, PET is commonly used to produce sails used in windsurfing and sailboats,³⁶ and in automotive parts.⁵²

Polybutylene terephthalate

PBT is a semi-crystalline thermoplastic polyester produced by condensation of either terephthalic acid or dimethyl terephthalate with 1,4-butanediol.^{30,53} It exhibits good chemical resistance, high stiffness, high strength, high heat resistance, easy processability, high dimensional stability, and good electrical properties.^{30,54} PBTs are used in electronic and electrical applications in the production of terminal boards, switches, lamp sockets, and electrical housing. In automotive applications, PBTs are used in electrical, exterior and under-the-hood components such as door mirror system, headlamps, antilock braking systems, electrical control units, plug connectors, pump housings, braking components, locking components, body panels, and windshield wipers.⁴⁴

Table 1. Properties of common engineering plastics used in transportation applications.

Polymer material	Density (g/cm ³)	Tensile strength (MPa)	Modulus of elasticity (GPa)	Elongation at break (%)	References
Acrylonitrile butadiene styrene	1–1.2	27.6–55.2	1.1–2.9	1.5–100	36
Polyamide	1.12–1.14	90–165	2.62–3.2	30–100	36
Polycarbonate	1.14–1.21	60–72.4	2–2.44	70–150	36
Polyethylene terephthalate	1.29–1.40	48.3–72.4	2.76–4.14	30–300	36
Polybutylene terephthalate	1.30–1.38	50–60	1.93–3	250	63
Polystyrene	1.04–1.05	35.9–56.6	1.2–2.6	1.2–3.6	36

Polystyrene

PS is a widely used thermoplastic polymer with excellent transparency and high tensile strength but it is brittle in nature.^{55,56} It exhibits characteristics such as a low barrier to both gases and water vapour,⁵⁵ good chemical resistance, hydrophobicity,⁵⁷ electrical resistance, and ease of manufacture.⁵⁸ PS is widely used in the packaging industry for packaging films,⁵⁵ trays, vending cups, food containers, and lids.⁵⁸ It is also used widely in household appliances and consumer goods, such as television housing, CD cases, cutlery, liner and shelving of refrigerators^{36,40} and houseware, and in the production of test tubes, syringes, and blister packs for pharmaceuticals.⁵⁸ Syndiotactic PS (sPS) is commonly used in automotive under-the-hood applications similarly to PA.⁵⁹ sPS is a semi-crystalline engineering plastics. It possesses excellent properties including high melting temperature, superior crystallization rate, enhanced mechanical performance, low dielectric properties, and high chemical resistance.^{60–62} High impact PS (HIPS) and expanded PS (EPS) are also commonly used in electrical and electronics equipment. HIPS is especially used in applications where PS is unable to meet the standards for impact resistance.²⁸ The properties of common engineering plastics utilized in the transportation sector are shown in Table 1.

Recycled engineering plastics

Extensive usage and rapid growth rates in utilization of engineering plastics especially in transportation and electrical and electronic applications has resulted in the generation of large amounts of plastic waste.⁴⁰ However, unlike recycled commodity plastics, the superior properties and high performance characteristics enable engineering plastics to be effectively reused after recycling. The higher price of virgin engineering plastics compared to commodity plastic can be offset by using recycled engineering plastics which possess superior properties to virgin commodity plastics at similar cost. Additionally, growing environmental concerns and greater regulations by governmental organizations have led to greater emphasis on plastics waste management.⁵² In Europe,

Table 2. Composition of engineering plastics in a selection of WEEE items.⁴⁰

WEEE items	Engineering plastic composition
Printers/faxes	PS (80%), HIPS (10%), ABS
Telecommunication devices	ABS (80%), PC/ABS (13%), HIPS, POM
Televisions	Polyphenyl ether/PS (63%), PC/ABS (32%), PET (5%)
Toys	ABS (70%), HIPS (10%), PA (5%)
Monitors	PC/ABS (90%), ABS (5%), HIPS (5%)
Computer	ABS (50%), PC/ABS (35%), HIPS (15%)
Small household equipment	PA (19%), ABS/SAN (17%), PC (10%), PBT, POM
Refrigeration	PS and EPS (31%), ABS (26%)
Dishwashers	PS (8%), ABS (7%)

WEEE: Waste Electrical and Electronic Equipment; PS: polystyrene; HIPS: high impact PS; ABS: acrylonitrile butadiene styrene; PC: polycarbonate; POM: polyoxymethylene; PET: polyethylene terephthalate; PA: polyamide; PBT: polybutylene terephthalate; SAN: styrene-acrylonitrile; EPS: expanded PS.

the European Commission issued the Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC), which aims to increase rates of collecting, recycling, and recovery of WEEE as a distinct waste stream.²⁸

The recycling of WEEE is a demanding process mainly due to the presence of 10–15 different types of polymers and their blends and because of the presence of different types of additive, flame retardants, fillers, and colorants.⁶⁴ WEEE parts contain various types of plastics resins, including commodity plastics (PP, PE), engineering plastics (PC, PA, ABS, PS), and thermosets. The composition of common engineering plastics found in WEEE is shown in Table 2.⁴⁰

Segregation of engineering plastics from WEEE and other plastics wastes stream is commonly done with mechanical or physical processing. Approximately 95% of plastic by weight can be recovered by manual dismantling of the waste product, although this is a labor-intensive operation and increases labor cost. Alternatively, shredding and multistage separation can be implemented after removal of hazardous elements. One major issue with recycling of WEEE plastics is the presence of foreign material and impurities in the polymer matrix, which can affect the quality of the recycled plastic. Another issue is the presence of metal parts in the WEEE part, which can generate economic pressure to shift the focus toward metal recovery and resulting in extraction of waste plastics of unsuitable size for further processing.⁴⁰

The two most common engineering plastics found in automobiles and electrical and electronic (EEE) are PC and ABS. The main reason for the widespread use of these two polymer in EEE is the excellent fire resistance properties of PC and PC/ABS blends once treated with flame retardants. Additionally, the relatively low cost of these polymers supports blending with more expensive engineering plastics.^{28,37} Blends of PC/ABS found in WEEE have been studied by several researchers. Elmaghor et al.⁶⁵ observed that the toughness of recycled PC greatly increased as a result of melt blending with maleic anhydride-grafted ABS (ABS-g-MA). The authors, investigating waste PC used

for 10 years prior to recycling, found that the value of impact strength of recycled PC/ABS-g-Ma blends was about three-quarters of the impact strength of pure PC.⁶⁵ Further, Balart et al.³⁷ studied the thermal properties of various composition of recycled PC/ABS blends obtained from WEEE using thermogravimetric analysis and determined the kinetic parameters with an autocatalytic model. It was found that in ABS-based blends, the activation energies were independent of heating rates, whereas in high PC-based blends activation energies increased with heating rate, implying that the thermal degradation mechanism in latter comprises several complex procedures.³⁷

In another study, Balart et al.⁶⁶ examined the performance of blends of recycled ABS/PC obtained from WEEE. The authors first investigated prior degradation by Fourier transformed infrared spectroscopy and found out that recycled ABS showed a small amount of degradation, whereas no signs of degradation were seen in the studied PC material. Blends of ABS and PC waste exhibited a decrease in mechanical properties when compared with virgin material; however, the values were still higher than those of commodity plastics. A blend of 10–20% PC concentration gave best mechanical performance. The authors concluded that the main reasons for the decrease in performance values were small previous degradation, low interaction caused by the partial miscibility of the SAN phase with the PC phase, demonstrated in differential scanning calorimetric (DSC) tests, and the presence of a thermo-oxidative degradation sensitive elastomeric phase in the SAN.⁶⁶

It has been shown that the properties of recycled plastics blends can be improved with the addition of modifiers or other additives. For example, Ramesh et al.⁶⁴ investigated the effect of virgin PC and impact modifier on the final performance of recycled blended plastics of PC, ABS, and HIPS. The results showed that addition of 10 wt% virgin PC and 10 wt% impact modifier leads to 167.7% improvement in the impact properties of the recycled blended plastic.⁶⁴

Natural fiber type

In terms of source, natural fibers are classified into animal fibers, plant fibers, and minerals fibers.^{10,16,17,19–22,67–70} Plant fibers in turn classified into six types: bast fibers, leaf fibers, seed/fruit fibers, straw/stalk fiber, grass fiber, and wood fiber.^{10,71,72} Classification of natural fiber is presented graphically in Figure 2. Based on their utility, plant fibers can also be classified into primary and secondary groups. Fibers such as jute, hemp, kenaf, and sisal are the primary group, as they are grown for their fiber contents, while fibers such as pineapple, oil palm, and coir belong to the secondary group, as these fibers are produced as a by-product.^{10,14} Bast fibers are extracted from the outer layer of stems of the plants by the retting process.^{9,71,73} Common types of bast fibers are flax, hemp, jute, kenaf, and ramie.^{5–7,10,13,14,25,26,71,73,74} Bast fibers are used in automotive applications because of their greater strength and low density which results in weight saving.^{70,73,74} Leaf fibers are taken from the leaf of the plant by the process of hand scrapping or mechanical extraction. These fibers are coarse and hard.⁷¹ Leaf fiber possesses high strength and in comparison with bast fibers, they display better impact performance.^{71,73} Some examples of leaf fibers are sisal, pineapple leaf, and banana

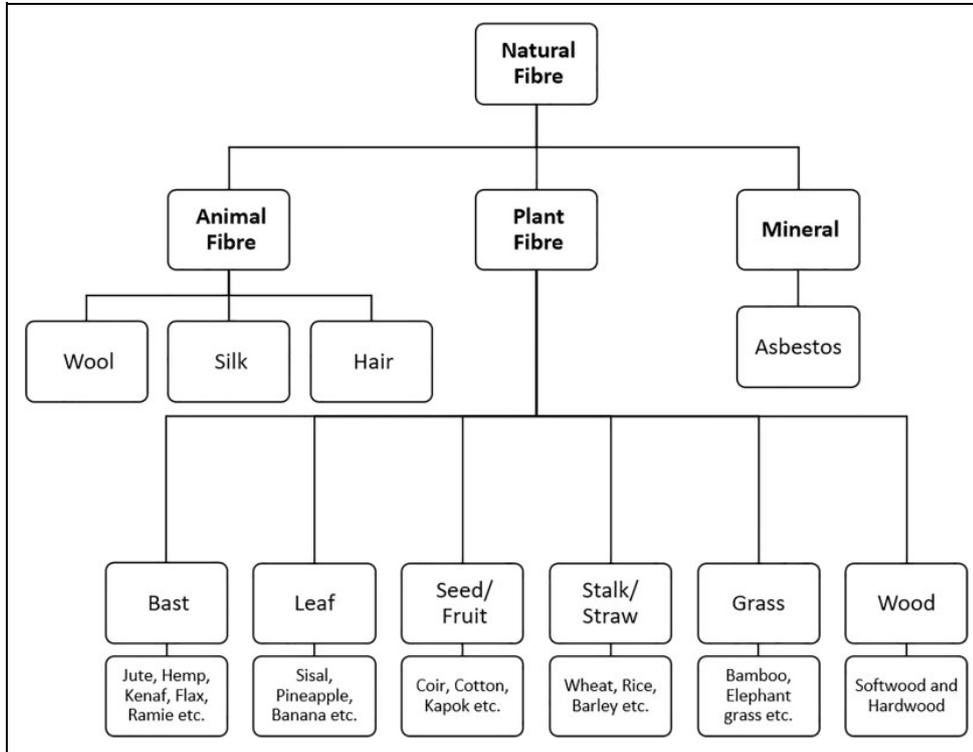


Figure 2. Classification of natural fibers.^{7,10,11,69,71}

(abaca).^{5,10,13,14,25,26,67,71,73} Fruit fiber is derived from the outer husk of the fruit, for example, coir fiber is extracted from coconut husk and seed fibers are produced from the boll or pod of plant seeds such as cotton and kapok. Stalk fiber is extracted from stalks of plants such as sugarcane and maize and also from the straws of wheat, rice, and barley.^{10,67,71,73} Straw fibers possess high strength, high stiffness, and low density.⁷³ Grass fibers are derived from tall grasses such as elephant grass and bamboo plants.^{71,73} Wood fibers are extracted from trees and are categorized into softwood and hardwood fibers.^{10,71} These fibers are commonly used in a mix with virgin or recycled plastics to produce wood–plastic composites, which are used in decking systems, railing, windows, and doors.⁷³ Global production of natural fibers and the largest producing regions are listed in Table 3. The most common natural fibers used as reinforcement in the transportation sector are flax,^{6,7,17,25,87–90} kenaf,^{6,7,25,88,90} jute,^{7,17,25,87–89} sisal,^{6,17,25,87–90} hemp,^{6,7,17,25,87–90} coir,^{6,25,90} abaca,^{6,88,90} wood,^{6,25,88,90} and certain crop residues such as rice.⁸⁸

Flax fiber

Flax belongs to the category of bast fibers and is one of the oldest fiber crops in the world. It is grown in temperate regions, for example, the Mediterranean and the Indian

Table 3. Natural fiber production and major producers.

Fiber type	Production (103 ton)	Major producer	References
Flax	830	Canada, France, Belgium	6,10,14,25,26
Hemp	214	China, France, Philippines	6,10,14,25,26,75
Jute	2300	India, China, Bangladesh	3,10,14,25,26,75
Kenaf	970	India, Bangladesh, United States	6,10,14,25,26,76
Ramie	100	India, China, Brazil, Philippines	10,14,25,26,76,77
Sisal	378	Tanzania, Brazil, Kenya	3,10,14,25,26,75,77,78
Abaca	70	Philippines, Ecuador, Costa Rica	3,10,14,25,26
Coir	100	India, Sri Lanka, Philippines, Malaysia	10,14,25,26,77,79,80
Oil palm	40	Malaysia, Indonesia	10,14,25,26,81,82
Pineapple	74	Philippines, Thailand, Indonesia	10,14,25,26,77
Bagasse	75,000	India, Brazil, China	10,14,25,26
Bamboo	30,000	India, China, Indonesia, Malaysia, Philippines	10,14,25,26,80
Rice husk	160,000	China, India, Indonesia, Malaysia, Bangladesh	83,84
Rice straw	579	China, India, Indonesia, Malaysia, Bangladesh	85,86
Wood fiber	1,750,000	Canada, United States, China	6,22

subcontinent.^{3,5,14,26,69,76,77} Flax fibers possess characteristics such as low density, high toughness, high strength and stiffness, and renewability.^{5,14,91} The tensile strength and compressive strength of elementary flax fiber has been found to be 1500 and 1200 MPa, respectively.⁹² Although the tensile strength of flax fiber is less in comparison with E-glass fiber (2400 MPa), the modulus of elasticity, E , is comparable with E-glass fibers.⁷ Also, due to its low density, flax fibers possess a strength/weight ratio comparable to E-glass and aramid fibers.^{7,9} The relatively high tensile strength and low tensile deformation of flax fibers enables the material to be used in a wide range of applications from automobile and aerospace parts to musical instruments.⁹³ In the automobile industry, flax fibers are used in door cladding, lining of seat backs, floor panels,^{5,6,90} and disk brakes.^{87,90}

Hemp fiber

Hemp belongs to the bast fiber category and is extracted from the Cannabis plant family. It is an annual plant and grows in temperate regions.^{3,10,14,26,69,76} Hemp is one of the earliest plants to be cultivated and is also a most ecological friendly fiber. It originates from central Asia and has been under cultivation for more than 12,000 years. Currently, China produces almost half of the world's industrial hemp supply.^{71,76,89} Hemp fibers possess excellent mechanical strength and higher Young's modulus. Hemp stem consists of 70% long bast fibers, which have a higher concentration of cellulose (55–72%) and lower concentration of lignin (2–5%).^{5,9,14} Due to their high strength and resistance to salt water, hemp fibers were earlier extensively used in canvas sails, nets, and rigging.^{69,76,77} Nowadays, they are primarily used in the textile and paper industries.^{9,10,14,69} In the

automobile sector, hemp fibers are used in door cladding, floor panels, and seat back linings.^{6,90}

Jute fiber

Jute is a bast fiber that is extracted from the ribbon of the stem of plants of genus *Corchorus*.^{5,14,26,80,89} Jute is one of the cheapest natural fibers available and it is produced in high volume in countries such as India, China, and Bangladesh.^{3,6,10,26,69} The jute plant can grow up to 2–3 m in 3–4 months. After separating the outer stem from the inner stem by a retting process, the outer stem is transformed into fibers.^{10,71,77,89} Jute fibers possess good mechanical characteristics, such as high tensile strength and good thermal/insulation properties.^{14,67,80} Their specific modulus is comparable to that of glass fiber. The fibers remain stable up to 200°C temperature and the properties remain unaffected.⁶⁹ Jute fiber can withstand wet environments and are biodegradable. Application for jute fibers includes packaging bags, ropes, yarns, and wall decorations.⁸⁰ Additionally, jute-reinforced polymer composite can be used for producing doors, windows, furniture, corrugated sheets, and water pipes,^{14,67} and in transportation applications, jute-based polymer composites are used in automotive doors and roof panels.⁸⁹

Kenaf fiber

Kenaf belongs to the genus *Hibiscus* of the Malvaceae plant family and is used as reinforcement in composites.^{14,16,26,76,89} Kenaf is a fast growing plant that grows in a wide range of climate conditions and can grow to a height of more than 3 m with base diameter 3–5 cm in 3 months.^{9,10,16,76,89} It is native to Africa and today is cultivated mainly in India, Bangladesh, and the United States.^{6,10} Components of kenaf plants such as leaves, seeds, and stalks are also exploited and are used to produce fiber strands, oils, and allopathic chemicals. Kenaf filament is composed of individual fibers which are distinct from each other and are 2–5 mm long.^{14,16,89} The stem of a kenaf plant consists of bark and a core. The bark of the plant has a dense structure, shows an orientated high crystalline fiber arrangement, and makes up 30–40% of the weight of the stem. The core of the kenaf plant is similar to wood, displays an isotropic amorphous pattern, and constitutes the remaining 60–70% of the stem weight.^{10,14,16} Kenaf fibers have low density, high specific mechanical properties, and good recyclability.^{9,14} In earlier times, kenaf was used to produce rope, sackcloth, canvas, and twine.^{9,14,76} Nowadays, however, it is used in applications such as raw material for the paper industry, in production of woven and nonwoven textiles,¹⁶ and for making nonwoven mats, inner door panels,^{6,16,90} and packing shelves^{6,74} in the automobile sectors.

Sisal fiber

Sisal is a leaf fiber that is extracted from the leaves of the sisal plant (*Agave sisalana*).^{5,14,26,78,89} The plant is native to tropical and subtropical regions of North and

South America. Nowadays, however, it is widely cultivated in Africa, the Far East, and the West Indies. The sisal plant resembles that of a giant pineapple and has a short plantation period.^{14,78,80,94} The sisal plant can also grow in the wild alongside fields and rail tracks.^{14,78,94} Usually a sisal plant consists of 200–250 leaves and each leaf can be composed of over 1000 fiber bundles.^{10,71,78,89,94} The fibers are extracted using a retting process followed by scrapping either by hand or machine.^{80,94} Sisal fiber is coarse and inflexible. It possesses good strength, durability, and high tenacity, and it is resistant to salt water.^{14,17} Thus, products such as carpets, mats, twines, and rope made of sisal fibers are used in the maritime, agricultural, and shipping sectors. It is also used in construction, where it acts as an effective reinforcement for the cement matrix.^{9,78,80,94} Sisal fibers are used in door cladding, lining of seatbacks, and floor panels.⁹⁰

Abaca fiber

Abaca/banana fiber is extracted from the banana plant^{3,9,14,26} which is one of the world's oldest cultivated plants and belongs to the Musaceae family.^{71,89} The plant is native to South East Asia mainly the Philippines.^{3,10,14,26} There are about 300 species of banana plant, out of which only 20 are suitable for consumption.^{71,77,89} Bananas, primarily of Cavendish type, are commercially cultivated across the globe in many tropical and subtropical regions such as the Philippines, Ecuador, and Costa Rica with an approximate annual yield of 70 million metric tons.^{10,71,89} It possesses excellent tensile strength.⁹ It is used to produce ropes, cordages, twines, and mats.^{14,26} In automobile manufacturing, abaca fiber is used in production of floor panels.^{6,74,90}

Coir fiber

Coir is a seed fiber extracted from the husk and outer shell of coconut.^{3,10,14,26,67,69,77,79,80} Eighty-five percent of the total weight of a coconut is fiber and husk.⁷⁹ After 9 months of growth, the coconut husk is green and the white smooth fiber can be extracted and used for the production of yarn, rope, and fishing nets. After 3 more months, the coconut has matured fully and produce strong and less flexible brown fiber, which is used for brushes and mattresses.^{69,80} Coconut grows predominantly in the coastal areas of India, Sri Lanka, Indonesia, Malaysia, and the Philippines.^{5,10,14,77,79,80} The annual production of coir fiber is approximately 250,000 tons.¹⁴ The above-mentioned countries consumed over 50% of total coir fiber production with India being the main consumer.^{14,79} The Indian state of Kerala produces 60% of world white coir fiber and Sri Lanka produces 36% of world brown coir fiber supply. The fiber is available in abundance,²⁶ cheap, renewable, and biodegradable,⁸⁰ and coir fibers are thick and coarse.^{10,14,79} The fiber is resistant to sea water,^{10,69,80} can withstand heat,^{69,80} is resistant to pests and fungi, provide good acoustic resistance, and can withstand wear.¹⁴ Coir fiber is used in production of helmets,^{79,80} bulletproof vests,⁷⁹ seat bottoms and back cushions in automobile,^{6,74,79,90} roofing, postboxes,⁸⁰ yarns, ropes, mats, mattresses, brushes, sacking, rugs, and insulation panels.^{10,14,80}

Rice husk and straw fiber

Rice belongs to the group of dominant cereal grains alongside wheat, barley, oat, and corn.²⁶ It is a major food crop, covering 1% of the earth's total surface.^{84,95} It originated between 6200 and 11,500 BC and is native to Asia, although nowadays, it is cultivated worldwide. Rice husk is the hard protecting encapsulation of the rice grain.¹⁰ Rice husk is an inedible and inexpensive by-product of rice processing and is separated from the edible grain during the refining process.^{83,84,96} For every 1000 kg of rice paddy, approximately 20–23% husk is generated.^{83,84,95,96} For this reason, it is easily available. Rice husk is abrasive and has properties such as low bulk density, toughness, and resistance to weathering.⁸⁴ Rice husk is used as filler in construction as a supplementary material to cement, insulation material,^{10,84} fuel,¹⁰ and as composite in the manufacture of bricks, fences, window and door frames, decks, and panels.^{25,84} Rice straw makes up approximately half of the total rice yield and is separated from the rice either manually or by machine. It is mainly used as a reinforcement in structural board applications. Composites of rice straw are used for their sound absorbing properties.¹⁰

Wood fiber

Wood is the most widely used natural fiber and is extensively used in the paper and pulp industry.¹³ Wood fibers are classified into softwood and hardwood fibers. Hardwood is extracted from the deciduous trees that shed leaves yearly, such as aspen and birch, while softwood is mainly extracted from coniferous trees which do not shed leaves, such as pines and spruces. Softwood tree usually grow at a faster pace than hardwood trees. Hardwood is dense and possesses a more complex cell structure than softwood. Softwood is the predominant type used in composite application, mainly because of its higher aspect ratio.¹⁰ The wood fibers are extracted from the xylem, the tracheid of the woods (both softwood and hardwood), and the vessels (only in hardwoods).^{10,97} Wood from sawmill chips, sawdust, wood flour, and wood residues can be used as the origin of wood fibers.¹⁸ The extraction of wood fibers is done by either pulping process or pulverizing process. The main applications of wood fibers are in paper and paperboard production and in composites for fencing, decking boards, window, and door frames.^{87,97} In automotive applications, wood fibers are used in seatbacks, cargo floor panels, and sliding door inserts.⁶

Chemical composition of plant fibers

Plant fibers are composed of three main components: cellulose, hemicellulose, and lignin. Additional components of plant fiber include pectin and waxes. Cellulose microfibrils are rigid and crystalline in nature.^{14,16,87} Cellulose is resistant to hydrolysis, strongly alkali and contains some oxidizing agents, but can be easily hydrolyzed by acid.^{20,22,87} Cellulose is responsible for characterization of the strength and mechanical properties of plant fibers.^{20,23,90,98,99} Hemicelluloses are polysaccharides^{18,22} and act as a reinforcement matrix between cellulose microfibrils.^{16,20,22,87} It is extremely hydrophilic^{18,20,87} and easily hydrolyzed in both acids and bases.^{20,87} Hemicellulose in plants

Table 4. Chemical composition of natural fibers.

Fiber type	Cellulose (%)	Hemicellulose (%)	Lignin (wt%)	Wax (%)	References
Flax	64.1–75	14.5–20.6	2–2.5	1.5–1.7	3,10,13,14,16,17,25,26,71,91,98
Hemp	68–74.4	15–22.4	3.7–10	0.8	3,10,13,14,16,17,25,26,71,98
Jute	61–71.5	12–20.4	11.8–13	0.5	3,10,13,14,16,17,25,26,67,71,98
Kenaf	31–72	20.3–33.9	8–21.2	—	3,10,13,14,16,25,26,71,98
Sisal	60–78	10–22	8–14	2	3,10,13,14,16,17,25,26,67,71,94,98,100
Abaca	56–63	20–25	7–9	3	3,10,14,16,25,26,71
Coir	32–43	0.15–0.25	40–45	—	3,10,13,14,16,25,26,67,71,98
Rice husk	35–45	19–25	20	14–17	3,10,14,25,26
Rice straw	41–57	33	8–19	8–38	3,10,14,25,26
Hardwood	31–64	25–40	14–34	—	13,97
Softwood	30–60	20–30	21–37	—	13,97

is responsible for the biodegradation, moisture absorption, thermal degradation, and flammability of plant fibers.^{20,23,90,98,99} Lignin is a complex hydrocarbon polymer found in plant fibers, and it also acts as a supportive matrix for cellulose and provides rigidity.^{20,87} It is amorphous,^{14,18,20,87} aromatic,²² and hydrophobic in nature^{14,20,87} and insoluble in most solvents.^{20,87} The role of lignin is to form char and ultraviolet degradation.^{20,90,98,99} Pectin provides flexibility in the plants. Waxes found in plant fibers are composed of various types of alcohols⁸⁷ and are responsible for wettability and interfacial adhesion characteristics of the fiber–matrix.^{10,98} Chemical composition varies significantly from one plant fiber type to other and also differs for different parts of same plant. The variation is also found within the same species of plant depending on geographical regions, age, stages of growth, and weather and soil conditions.^{23,26} Table 4 shows the chemical composition of various plant fibers.

Properties of plant fibers

The properties of natural fibers also vary from one plant fiber type to another and they can differ among the same fiber types coming from different geographical regions, as well as between fibers extracted from the same plant.¹⁰¹ Major factors that affect the properties of natural fibers and cause such wide variation are chemical composition of the fibers,^{10,67} growing conditions such as soil type, weather and aging condition,^{17,26,67} extraction method,^{10,14,26,67,101} surface treatment,¹⁷ cell structure,^{17,26} harvesting period and technique,^{10,101} plant age,^{10,14} source,¹⁴ and storage.¹⁰ The hydrophilic nature and significant moisture content of natural fibers also tends to influence their mechanical properties.^{10,26} Table 5 shows the mechanical properties of various natural fibers.

The physical properties of the natural fibers are crucial in determining the overall performance of the fibers and their composites. These physical properties include fiber length, diameter, strength, structure, defects, and crystallinity. The strength of plant fiber

Table 5. Mechanical properties of natural fibers.^a

Fiber type	Density (g/cm ³)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation to break (%)	References
Flax	1.4–1.5	88–1600	27.6–80	1.2–3.3	6,7,9–11,13,14,16,25,26,67–71,73,75,91,98
Hemp	1.4–1.6	500–900	30–80	1.6–4	6,7,9–11,13,14,16,25,26,67–71,73,75,76,98
Jute	1.3–1.5	385–850	10–55	1.2–1.8	6,7,9–11,13,14,16,25,26 67–71,73,75,98,102
Kenaf	0.6–1.5	223–1191	22.1–60	1.6–6.9	6,7,10,11,13,14,16,25,26,68,69,71,76
Sisal	1.3–1.5	400–700	9–38	2–14	6,9–11,14,16,25,26,67–71,73,75,94,97,98,100,102
Abaca	1.5	400–980	12	3–10	10,11,13,14,16,25,26,71
Coir	1.1–1.6	131–593	4–6	14–40	6,9–11,13,14,16,25,26,67–71,73,75,94,97,98,102
Rice husk	1.4	19–135	0.3–2.6	—	103,104
Rice straw	—	450	26	2.2	105
Softwood kraft pulp	1.5	600–1000	18–40	4.4	6,68,70,71,75,97
Hardwood	1.2	—	37.9	—	97

^aProperties of natural fibers varies depending on fiber preparation, test methods, source of fibers, agricultural parameters, and other factors. Therefore these tables are compiled from various sources given in the column on the far right of the table.

is defined by its higher aspect ratio (fiber length/width) and is an important factor in selecting fiber of a particular application.^{13,26} Therefore, higher strength means lower fibrillar angle, smaller fiber diameter, and higher aspect ratio and thus leads to better mechanical properties.¹⁰ Any major alteration in the structure of plant fiber such as change in density, wall thickness, fiber length, and diameter can result in differences in the physical properties.²⁶ Table 6 shows key physical properties of various fibers.

NFRP composite in transportation

Utilization of NFRP composite is increasing rapidly in applications such as automotive, aerospace, sports, structural components, packing, construction, boats, and electronic goods.^{25,109} The wide range of applications is because of natural fiber's low specific weight, abundancy, low cost, good mechanical properties, biodegradability, renewability, low energy consumption in production, and good corrosion and fatigue resistance compared to synthetic fibers such as glass, carbon, and aramid, which are less biodegradable and derived from nonrenewable sources.^{3,19,25,109–111} A brief comparison of natural fibers with glass and carbon fibers is shown in Table 7. Natural fibers have higher specific strength than glass fiber and equivalent specific modulus. Flax and hemp are of great importance as reinforcing material in production of composites used for automobile, aerospace, and construction applications.¹⁰⁹ The major challenges of NFRP composites in transportation are high water absorption, variation in fiber properties, lack of adhesion between the fiber and matrix, low fire resistance, and limited processing temperature.^{3,20,21,26,74,90,101,109}

Table 6. Physical properties of natural fibers.

Fiber type	Length of fiber (mm)	Diameter of fiber (μm)	Microfibrillar angle ($^\circ$)	References
Flax	9–70	5–38	5–10	7,13,98,106
Hemp	5–55	10–51	2–6.2	7,13,98,106
Jute	0.8–6	5–25	8	7,13,98,106
Kenaf (bast)	1.4–6	11–36	—	7,13,106
Sisal	0.8–8	7–47	20	13,98,106
Abaca	6	20	—	107
Coir	0.3–1	100–450	41–45	13,90,98
Rice husk	0.045–2	—	—	108
Rice straw	0.4–3.4	4–16	—	90
Softwood	2–6	20–40	—	13,97
Hardwood	1–2	10–50	—	13,97

Table 7. Comparison of natural fiber with glass and carbon fibers.^{13,75,112–114}

	Natural fibers	Glass fibers	Carbon fibers
Density	Low	Double that of natural fibers	Equivalent to natural fibers
Cost	Low	Low but higher than natural fibers	Extremely high
Renewability	Yes	No	No
Recyclability	Yes	Difficult	Difficult
Energy consumption	Low	High	High
CO ₂ neutral	Yes	No	No
Biodegradable	Yes	No	No
Health risk	No	Yes	Yes
Abrasion to machinery	No	Yes	Less
Easy to dispose	Yes	No	No

One of the major disadvantages of plant fibers is the poor compatibility between fiber and polymer matrix. This occurs due to the presence of cellulose in natural fibers that are hydrophilic in nature in comparison to the hydrophobic nature of thermoplastics polymers; the incompatibility between polar natural fibers and nonpolar polymer matrix leads to the nonuniform wetting of the fibers in the matrix and their poor interface adhesion.^{13,14,20,70,88,114} Load transfer is therefore insufficient between the fiber reinforcement and matrix, thus resulting in poor mechanical properties of the NFRP composites.⁸⁸ Fibers provide high strength and stiffness in composite structures but fibers are not able to withstand heavy loads by themselves. Consequently, natural fibers are mixed with matrix resin as a reinforcement, where the polymer matrix holds the fibers together

and is responsible for transmission of shear forces. Thus, the efficiency of composites depends on the interface between fiber and polymer matrix.^{5,25,111,114}

Another disadvantage of NFRP composites is the high water absorption of natural fibers. This is more prevalent in the natural fibers with greater cellulose content such as sisal, flax, and hemp due to the hydrophilic nature of cellulose. Additionally, the moisture content of certain natural fibers can reach as high as 20%. Presence of high moisture content and absorption of high amount of water from environment can cause fiber swelling within the composites resulting in dimensional instability and thus affecting mechanical properties of the composites material.^{13,21,88} Also, the presence of water can create voids in the matrix and further decrease the adhesion between fiber and matrix.⁸⁸ Thus using NFRP composites in external application in vehicles or aircrafts and in maritime industry, the water absorption can greatly affect the performance requirement of the component.^{21,88} However, methods can be adopted to reduce moisture intake such as chemical treatment of natural fibers to improve adhesion with the polymer matrix to reduce moisture intake by wicking at the fiber–matrix interface and laminating inner layer of NFRP composites with an outer layer of synthetic fiber composites that reduce the ingress of water by protecting the NFRP composite.¹¹⁵

These two challenges of NFRP composites can be tackled by surface treatment methods such as chemically treating natural fibers for improving interfacial adhesion and thus reducing moisture intake and ensuring good mechanical properties.^{13,21,25,70,74,88} The surface treatment can be done either by adding compatibilizers/coupling agents or by chemically treating the fibers.⁷⁴ The various kinds of chemical treatment include silane, alkaline, acetylation, maleated coupling, anhydride, and benzylation.^{13,21,25,88} These chemical treatments aim to improve the interfacial adhesion between fiber and polymer matrix by increasing the surface roughness of fibers and modify fiber surface to mitigate moisture intake. Silane treatment involve introduction of silane to the natural fibers to react with hydroxyl groups and improve fiber wetting.^{74,88} Alkaline treatment also known as mercerization focuses on decreasing moisture intake of natural fibers by decreasing the hydrogen content of cellulose. Alkali also modifies the hemicellulose of the fiber which is one of the most water absorbing constituent of the fiber.^{21,74} It is one of the most widely used chemical treatment to increase surface roughness and improving fiber wetting.⁸⁸ One disadvantage of alkaline treatment is higher pH level, high surface contamination with waste water, and chemical and mechanical degradation of cellulose.²¹ Acetylation or esterification decreases the hydrophilic nature of the natural fibers by introducing an acetyl functional group to react with hydroxyl groups of natural fibers⁸⁸ and aids in improving the dimensional stability of the composite. In few cases, some of the mechanical properties of the fibers start to increase with rise in acetylation degree of 15% to 18%, but any further increase results in detrimental effects on the properties.²¹

Apart from moisture absorption and interfacial adhesion, NFRP composites faces other challenges in transportation sector. These include limitation against processing temperature and low fire resistance. Natural fiber has certain limitations with processing temperature since they are composed of various organic materials (primarily cellulose, hemicellulose, and lignin) and thus processing temperature above 200°C can cause

cellulose degradation and emission of various gaseous and volatile products. These gases can cause high porosity in the composite and reduce mechanical properties of the composite. Although it is possible to use temperature higher than 200°C but only for short periods. Hence, to ensure better thermal stability, techniques like coating or grafting the fibers with monomers are implemented.^{13,70} Additionally, alkaline treatment and acetylation can also improve the thermal stability of natural fibers.⁸⁸

Natural fibers by their nature have very poor fire resistance, which is a big disadvantage especially in aircraft parts and even automobiles. It is important for the natural fibers to fulfill the necessary safety measures in order to compete with artificial fibers as a composite material for transportation sector. Natural fibers with high cellulose content have higher flammability capacity, whereas fibers with higher lignin content has greater chance of char formation. The decomposition temperature of natural fibers is very low at 240°C. However, the components of fibers, such as cellulose, hemicellulose, and lignin, degrade at different temperature ranges.^{21,25} For example, temperature range for cellulose decomposition is between 260°C and 350°C, while for hemicellulose, it is between 200°C and 260°C,²¹ and lignin decomposes slower from 200°C up to 500°C.¹¹⁶ Studies show similar decomposition behavior of jute and sisal fiber since they have similar percentage of lignin content and both degrade at very high temperature. In comparison, poor lignin content of flax fibers leads to lower decomposition temperatures.²¹

Currently, fewer studies have been done on fire resistance of NFRP composites and thus very limited literature is available. Factors such as high lignin content, large fiber orientation angle, and low crystallinity can improve the fire resistance of natural fibers.²¹ Further, to improve fire resistance of NFRP composites, various methods can be employed. Fire barrier treatments such as coating and additives used as intumescent are very promising and expand on heating leading to surface that is evenly charred. This charred surface protects the underlying component against the heat. By coating NFRP composites with some fire retardant is another method for enhancing the fire resistance property of composites. This coating is usually done at the end or finishing stage or impregnation. For composites, two most widely used fire retardants are aluminum hydroxide ($\text{Al}(\text{OH})_3$) and magnesium hydroxide ($\text{Mg}(\text{OH})_2$), with the latter displaying better thermal stability since the temperature range given off by the decomposition of magnesium hydroxide is nearly 300–320°C compared to 200°C by aluminum hydroxide.²⁵

Automotive

In automobile manufacturing, selection of an appropriate material for construction of the vehicle is key, as the material determines the vehicle's weight, cost, and crashworthiness. Increased environmental concerns and stringent government regulations regarding CO_2 emissions and recyclability of automotive components have driven many automobile producers to invest in development of lightweight sustainable material for their vehicles. Reducing the weight of the vehicle, which directly corresponds to reduction in fuel consumption, brings better environmental performance by reducing CO_2 emissions.¹¹⁷ Weight reduction of vehicles can be achieved by either design change or

material change. At the same time, it is important for automotive manufacturers to keep the cost of the vehicle in check in order to be relevant in a competitive market. Some lightweight materials used in the transportation sector, for example, carbon fiber, are expensive, which increases the price of the vehicle. The cost of any new material should be equivalent or lower than that of the existing material. Thus, simply choosing the lightest material for construction of the vehicle is not justified. However, if the new material offers additional benefits and improved performance, then an increase in the cost may be reasonable. It should be noted that any change in material should maintain the crashworthiness of the vehicle by absorbing impact energy during a collision and thus offering passenger safety.³

Every year many vehicles reach the end of their useful life, generating about 8–9 million tons waste, of which only 65–75% is recycled effectively. The European Union has issued an end-of-life vehicle directive, 2000/53/EC, which mandates that 85% of the vehicle weight should be recyclable by 2005 and 95% of the weight by 2015. Additionally, vehicles should be composed of 95% recyclable material with 85% recoverable by mechanical recycling and 10% through energy recovery/thermal recycling by 2015.^{4,68,70,90} Further, the European Commission and the European Automobile Manufacturers Association comprised of BMW, Daimler Chrysler, Fiat, Ford, GM, Porsche, Renault, VW, and PSA Peugeot Citroën jointly passed legislation for new passenger cars to reduce average CO₂ emissions to 130 g/km by 2015 and to 95 g/km by 2020. For light commercial vehicles, average CO₂ emissions were set to 175 g/km by 2017 and to 147 g/km by 2020.^{4,118} Following this legislation, the Japan Automobile Manufacturers Association and Korea Automobile Manufacturers Association also set CO₂ emissions limits to 140 g/km by 2009 for all vehicles sold in Europe.⁴

NFRP composites are used in both internal and external components such as panels for floor and doors, backrest of seats, parcel shelves, luggage compartments, door linings, and headliners of vehicle roofs.^{25,70,88,109,111} Natural fibers such as kenaf, flax, sisal, and jute are most commonly used as the reinforcing material.^{89,111} However, the choice of natural fiber to manufacture automobile composites is mainly influenced by source proximity; thus, jute, ramie, and kenaf are used mostly in India and Asia, whereas in Europe NFRP composites components mostly include flax or hemp. South American automobile producers mainly use sisal or ramie.^{7,119} The application of NFRP in automobiles helps to reduce the weight of vehicle and improves the efficiency without compromising the safety aspect of vehicle. By using natural fiber composites, approximately 30% of the vehicle's weight and 20% of cost can be saved.¹¹¹

European car manufacturers have been using natural fibers such as flax, hemp, jute, and sisal in producing door panels, headliners, and dashboard for decades, resulting in weight savings, cost reduction, and lower CO₂ emissions.⁶ Nylon and flax composites are used in making floor mats, while fibers such as hemp and cotton are used in inner cladding, seat back lining, and floor panel.⁴ Flax, sisal, and hemp fibers are used in door cladding, seatbacks lining, and floor panel. Flax is also used in car disk brakes as a replacement for asbestos.^{70,90} Coconut fibers are used for seat bottoms, back cushions, and head restraints.⁹⁰ Abaca fibers are used for floor body panel and kenaf for door

Table 8. Amount of natural fibers used in automobile industry.¹¹⁹

Parts of automobile	Amount of natural fibers
Front door linens	1.2–1.8 kg
Rear door linens	0.8–1.5 kg
Boot linens	1.5–2.5 kg
Parcel shelves/packaging tray	Up to 2 kg
Seat backs	1.6–2.0 kg
Sunroof sliders	Up to 0.4 kg
Headliners	2.5 kg

**Figure 3.** Components of Mercedes E-Class produced by using natural fiber composites.^{4,6,74}

inner panel.⁹⁰ Typical amounts of plant fibers used in automobile industry are shown in Table 8.

Some German car manufacturers like BMW, Audi, Volkswagen, Daimler Chrysler, and Mercedes make great use of NFRP composites in both interior and exterior components of their vehicles.^{3,25,70,89,90,109,120} Other international car producers such as GM, Ford, Volvo, Opel, Renault, and Peugeot are also following the trend of greater utilizing NFRP composites in their products.^{70,88,90} Daimler Chrysler is leading the way in terms of global natural fibers usage. Flax, hemp, sisal, wool, and other natural fibers are used in 50 Mercedes-Benz E-Class components^{6,74,109} (Figure 3). Currently, Daimler is using natural fibers such as flax, hemp, sisal and coconut-reinforced components for its A, C, E and S Class models.⁸⁸ However, the majority of these NFRP composite materials are primarily composed of virgin commodity plastics or thermosets rather than engineering plastics.



Figure 4. Composite door panel of S-Class Mercedes-Benz.^{74,120}

The Mercedes-Benz A-Class model uses a seat made up of coconut fibers and a rubber latex composite. The E-Class model uses flax–sisal fiber mat-reinforced epoxy door panels.^{25,121} A further example is found in the Travego coach from Mercedes-Benz, which uses engine encapsulations made of flax/PP-reinforced composite,^{3,121} resulting in 10% weight reduction of the cover and 5% cost reduction.¹¹⁹ Daimler Chrysler uses a composite spare tire carrier in the Mercedes-Benz A-Class car. The component is produced using Abaca (banana) fiber reinforced with a PP matrix instead of glass fibers. The composite component results in cost saving and mass reduction, which reduces CO₂ emission and saves energy needed for production of glass fiber.^{4,88,109,121}

In 1996, Daimler Chrysler used jute/epoxy composite for the door panels of its Mercedes-Benz E-Class model.^{70,88,89,109,119} In 1999, the S-Class Mercedes-Benz had an inner door panel composed of 35% polyurethane (PU) and 65% blend of flax, hemp, and sisal fiber¹⁰⁹ (Figure 4). The 2006 model of Mercedes S-Class has wood fiber-reinforced components in the front door lining and back rest of the driver seat and some components are made of flax fiber-reinforced material, for example, the trunk cover and parcel shelves. In total, the vehicle contains 27 interior components that include natural fiber, which results in 73% increase in use of renewable material over previous models and weight reduction of the vehicle by 43 kg.^{4,121}

Natural fiber composites have been used since 1990s in BMW models in the 3, 5 and 7 series for making the linings of interior doors and panelling.^{4,121} For example, door panels of the BMW i3 electric vehicle are produced with hemp fibers and about 95% of the components are recyclable.¹²² Recently, BMW has started using biocomposites in structural components such as bumpers, suspension parts, and fender liners.^{4,121} BMW produced the inner door panels of its 7 series sedan in 2010 using acrylic reinforced with sisal fiber mats.⁸⁸ BMW 7 series was manufactured with 24 kg of raw renewable material, including flax and sisal fibers, used in the inner door lining panels.^{4,25} On average, BMW utilizes 8–13 kg natural fibers per vehicle.³ In 2004, BMW used 10,000 tons of natural fibers (flax, sisal, cotton, and wood) in production of its vehicles.^{6,25,123}

General Motors used flax reinforced with PP composites in trim panels and back shelf components for the Chevrolet Impala 2002 model.^{25,88} Wood fiber composites are used in seatbacks of the Cadillac DeVille and the cargo floor of the GMC Envoy and Chevy Trailblazer.^{4,6,88} Ford uses kenaf-reinforced PP composites in the door panels of its Mondeo models, which reduces the weight of the door by 5–10%.^{25,88,109} Currently, Ford uses 5–13 kg natural fiber per vehicle.^{3,119} Volkswagen uses natural fiber in seatbacks, door panels, inserts, and boot liners of Passat, Golf, A4, and Bora models.^{4,25} The 2000 model of Audi A2 used PU reinforced with a flax/sisal mixed fiber mat in its door trim panels.^{3,4,25,70,88,89,109,119} The spare tire cover of the 2003 model of the Toyota Raum multi-purpose vehicle was made of a polylactic acid (PLA) matrix reinforced with kenaf fibers.^{4,88,119,121} In 2011, Toyota developed a new bio-PET material from sugarcane and used it to produce luggage trunk liners and parcel shelves for the new Lexus CT 200h hybrid-electric compact car.^{4,6,88,89,109} Opel uses a mixture of kenaf and flax in the package tray (parcel shelves) and door panels of the Vectra.²⁵ Findlay Industries, a manufacturer of automobile and truck parts, make the headliner of the Mack truck with a hemp, flax, kenaf, and sisal mixture.⁶ Mitsubishi Motors, in collaboration with Fiat SPA, manufactured some interior components of its vehicles using a bamboo fiber-reinforced polybutylene succinate bioresin. Nylon fibers and bio-PLA produced from sugarcane molasses and PP fibers were used for floor mats.^{4,88,121} High strength PA is used to produce front end carriers of the Skoda Octavia. The front end carriers are thin walled, possess high stiffness, and high mechanical strength and good surface quality. Newer models of Porsche include engines whose intake system, airframe, and accessories are produced with PA.¹²³ The Saturn L300 used kenaf and flax fibers in package trays and door panels. Honda uses wood fibers in manufacture of floor components of the Pilot SUV.^{6,88} In 2008, Lotus manufactured a concept car named Eco Elise (shown in Figure 5) with body panels made of hemp-reinforced polyester composite, a carpet made up of woven sisal fibers, and interiors upholstery made with eco-wool.^{88,109,110}

A brief overview of natural fiber composites used by various automobile manufacturers is given in Table 9.

Aerospace

The modern aerospace industry is heavily reliant on synthetic and nonrenewable materials. Nowadays, carbon fiber composites are used as the principal construction material of aircraft, followed by metals such as aluminum and titanium, and to a lesser extent, fiberglass. For example, in the Boeing 787 Dreamliner (shown in Figure 6(a)) and the Airbus 350 XWB, (shown in Figure 6(b)) components such as the fuselage, turbine housing, wings, and stabilizers are fabricated mainly using composites, thereby making the aircraft lightweight and giving improved service life, reduced noise levels, and increased comfort.¹²³ Composite materials are used in the aviation industry to produce lightweight structural designs due to their excellent specific strength, good stiffness properties, and ease of fabrication into complex shapes.¹²⁶ The current trend in the aerospace industry is to produce more fuel efficient aircrafts by increasing usage of composite materials.¹²⁷ Currently, composite materials are used in cabin interiors, due to



Figure 5. Lotus Eco Elise with hemp components.¹²⁴

their high strength-to-weight ratio, thus makes the aircraft more fuel efficient. Common interior applications of composites include seats, cabin separators, lavatories, ceiling and wall panels, floor boards, galleys, and storage bins.^{121,128}

NFRP composites have recently started to be used in the aerospace industry.^{110,121} The increased usage of natural fibers in aerospace applications over carbon and glass fibers is due to their low cost, low weight, and biodegradability.²¹ In the aviation industry, aspects such as reducing aircraft weight, improving fuel economy, increasing carrying capacity, and improving aircraft maneuverability are of great importance.^{110,121,123} These requirements can be accomplished by using NFRP composites, as they offer high aircraft efficiency through lower fuel consumption and reduced emissions. Their high strength and stiffness allow fabrication of complex shapes, which enables improvement in aerodynamic efficiency. Furthermore, they have good fatigue and corrosion properties.^{121,129} Over 200 aircraft components are currently manufactured from NFRP composite, these include cabin equipment, side walls, and propeller system.^{21,110} The European Union has funded research and development of the use of NFRP composites in aviation parts. The Cayley project, an EU co-funded project in a collaboration with Boeing Research and Technology, Invent GmbH, Aimplas, and Lineo, aims to develop eco-friendly interior panels for aircraft using flax fiber and recycled thermoplastic sheets.¹²⁷ Another EU-funded project, named Eco-Compass, is working on developing eco-friendly composites from biosources and recycled materials

Table 9. Natural fibers in automotive components.^{3,25,111,119}

Manufacturers	Model	Applications	Natural fiber
Daimler Chrysler	A, C, E, and S Class	Door panels, floor panels, trunk panels, dashboards, pillar cover panels, seat back rests, insulation	Flax, sisal, coir, wood, banana, cotton
BMW	3, 5, and 7 series	Door panels, boot linings, seat backs, headliner panels, noise insulation panels	Flax, sisal, cotton, wood, hemp
Audi	A2, A3, A4, A6, A8, A4 Avant, Coupe, Roadster	Seat backs, back door panels, side door panels, boot liners, spare tire liners	Flax, sisal
Volkswagen	Golf, Passat, Bora, A4	Seat backs, door panels, boot liners, boot lid finish panels	Flax, sisal
Ford	Mondeo, Focus	Door panels, boot liners, floor trays, door inserts	Kenaf, wheat, castor
Toyota	Raum, Brevis, Harrier, Celsior	Floor mats, spare tire covers, door panels and seat backs, luggage compartments	Kenaf, sugarcane, bamboo
General Motors	Cadillac DeVille, Chevy Trailblazer, Chevy Impala, GMC Envoy	Seat backs, cargo area floor mats, noise insulation, door panels, trim	Cotton, flax, wood, kenaf, hemp
Opel	Vectra, Astra, Zafira	Door panels, head liner panels, instrumental panels	Flax, kenaf
Lotus Peugeot	Eco Elsie 406	Body panels, interior mats, seats Front and rear door panels, seat backs, packaging trays	Hemp, sisal
Fiat	Punto, Brava, Alfa Romeo 146, 156, 159	Door panels	
Volvo Mitsubishi	V70, C70	Seat cushions, cargo floor mats Cargo area floors, door panels, instrument panels	

for the aviation sector. The focus of the project is to find alternative solutions to reduce carbon- and glass-reinforced polymer composites in aircrafts by utilizing bioresin, natural fibers, or recycled carbon fibers in secondary structure and interior applications. Flax and ramie fibers have shown potential for use in NFRP composites for aircraft with some enhancement to their tensile strength and flame-retardant properties.¹³⁰

In high performance and lightweight applications, epoxy resin is generally used in combination with either carbon fiber or natural fibers.¹²⁶ The sidewall panels of Boeing 737 were manufactured using flax/epoxy sandwich composites, where the flax fabrics were treated with halogen-free fire retardants. Flax/epoxy prepregs are 35% lighter than

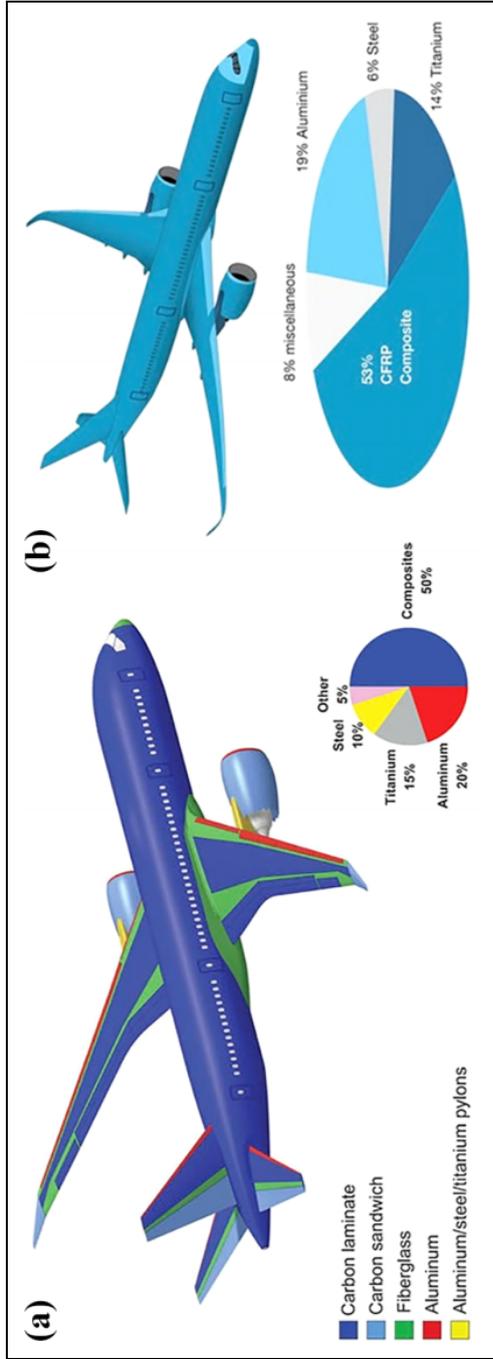


Figure 6. Material compositions of: (a) Boeing 787 Dreamliner; and (b) Airbus 350 XWB. ¹²⁵

carbon/epoxy prepregs and have a cost similar to glass/epoxy unidirection prepregs.^{121,127} Apart from epoxy, phenolics and engineering thermoplastics such as polyphenylene sulfide, polyether ether ketone, PA, and PC are the main focus of attention in aviation industry, mainly due to their good fire resistance properties, which comply with the flame, smoke, and toxicity regulations of the aviation sector.^{127,128} For example, Victrex Europa GmbH developed VICTREX[®] PEEK (polyether ether ketone) and reinforced it with chopped glass and carbon fiber for aircraft applications. The material possesses mechanical strength and dimensional stability comparable to metal alloys. The material is capable of withstanding high temperatures and has a melting point of 343°C. In addition, the material is corrosion resistant, chemical resistant, wear resistant, and abrasion resistant.¹²³

Maritime

The introduction of greater composites use in the maritime industry is primarily driven by the need to replace wood, which degrades when subject to attack by biological agents, and steel and aluminum alloys, which can corrode and difficult to weld respectively, and further to reduce the weight of the construction. Composites in maritime applications are used in both external and internal equipment, including bulkheads, decks, propellers, heat exchangers, valves, pumps, watertight doors, ducts, and pipes. Composites of glass- or carbon-reinforced polymers are utilized in racing boats and yachts due to their lightweight construction and good resistance to wave impacts.¹²¹ The introduction of FRP composite materials in the maritime sector has had a great impact on the construction of modern ships. FRP composites help to reduce weight, which in turn reduces fuel consumption and improves maneuverability. FRP also has good water and corrosion resistance, which is crucial for maritime vessels. Carbon composite, which can absorb electromagnetic waves and thus gives stealth characteristics, has been used in some specialist boat hulls. Carbon fiber reinforced with polymer resins such as epoxy, vinyl ester, polyester, or phenolic are commonly used to produce racing boats and yachts such as the Revolver 42 speedboat, which weighs 7500 kg yet is able to reach 68 knots, and the Vestas Sailrocket 2, which broke the sailing speed record with an average speed over 65 knots, are some classic examples.¹²³ The major disadvantages of carbon-reinforced polymer composites in maritime application are cost, environmental concerns, poor reparability, and recyclability. Thermosetting polymer resins such as epoxy, polyester, and vinyl ester, which are difficult to recycle, are quite commonly used in FRP composite structures. Nowadays, recyclable thermoplastics such as PA, PP, PET, and PBT are also used in maritime construction.¹³¹

Waste disposal regulation and environmental concerns means that attention has shifted to using biocomposites in boat construction instead of synthetic composites, and natural fibers have recently come back into use in maritime applications.¹²¹ In the past, natural fibers were used in form of ropes, nets, and sailcloth. In earlier times, timber rafts were lashed together with ropes and twines prepared from natural fiber resources. Flax, cotton, and hemp fibers were commonly used in weaving of canvas sails, until being replaced by synthetic fibers.¹¹⁵ Further, flax fiber possesses better vibration absorption

than Kevlar or glass fiber, which is quite crucial as vessels are subjected to vibration when cruising at high speed.¹²¹ In earlier times, natural fibers were used in maritime application mainly because there was no alternative. Natural fibers are returning back to maritime applications due to their low cost and low environmental impact. Problems such as moisture absorption remains a major concern even in modern maritime vessels using natural fibers as reinforcement in composite materials. Earlier, waterproofing of sailcloth made of by natural fibers was done by applying linseed oil or paraffin wax.¹¹⁵

A major concern with NFRP composites in maritime applications is moisture intake by natural fibers while operating in the marine environment.¹¹⁰ The problem stems mainly from weak interfacial adhesion between natural fibers and the polymer resin matrix. A number of techniques attempt to address this issue, for example, chemical treatment of the natural fibers to optimize adhesion with the polymer matrix or the production of hybrid composites with a laminating layer of NFRP composites and synthetic fiber composites.¹¹⁵

Although natural fibers are slowly receiving more attention in maritime applications, the industry is still dominated by glass and carbon fiber. Nevertheless, much research and development is being done with the aim of enabling greater natural fiber use in future maritime applications.¹³¹ An example is the NavEcoMat project, which is a joint research between marine construction companies and material research laboratories. In the project, an eco-friendly composite material has been developed that uses PLA and flax fibers and has properties similar to glass or carbon composite material. The developed technology was used to produce a Naskapi-style canoe made of PLA films reinforced with flax fiber mats.¹²³ In other research, an epoxy matrix reinforced with 50% flax fiber was used to produce a racing boat prototype, "Araldite". Flax with bioresin (EcoComp[®] UV-L) has also been used to manufacture kayaks and canoes.^{115,121} Basalt fiber, which has superior mechanical characteristics and is eco-friendly, is also suitable for maritime applications, and thus basalt and balsa wood composites have been used in the hull and deck of sailing yachts.¹²¹

Properties of NFRP composites

The mechanical properties of NFRP composites depends on fiber orientation, moisture absorption, impurities, and interfacial adhesion.^{21,25} However, the interfacial adhesion between natural fiber and polymer matrix is extremely poor.^{21,25,67} The poor adhesion mainly because of incompatibility between the plant fiber and polymer matrix as a result of the of hydroxyl group present in plant fibers, which makes them hydrophilic and thus incompatible with the hydrophobic nature of polymer.^{9,14,20,26} Additionally, high moisture content (Table 10) in fibers leads to poor processing and the creation voids in the matrix, which result in poorer mechanical properties.^{9,20} The high moisture content of plant fibers also greatly affects the biological degradation of NFRP composite.²⁶ Processing methods and parameters affect the properties and surface characteristics.²⁵ The performance of NFRP composites depends largely on factors such as the structure and chemical composition of the fibers, cell dimensions, physical and mechanical properties of the fibers, microfibrillar angle, defects, and the interaction of fibers with the

Table 10. Moisture content of plant fiber.

Fiber type	Moisture content	References
Flax	7–12	9,16,98
Hemp	6.2–12	9,16,76,98
Jute	12–13.7	9,16,98
Kenaf (bast)	10–12	76
Sisal	10–22	9,16,98
Abaca	5–10	16
Coir	8–10	9,98
Rice husk	5–10	132
Rice straw	6.5–9.8	86,105

polymer.^{25,26,71} Good interfacial adhesion allows efficient stress transfer between the fiber and matrix material.^{5,25} The fiber surface properties, and more specifically, the interfacial adhesion between the fiber and polymer matrix can be improved by appropriate chemical treatments, additives or coatings, which improve the surface wettability of the fiber.^{20,23}

Several researchers have examined the mechanical and physical properties of composites made up of engineering plastics and natural fibers. Yeh et al.¹³³ compared the mechanical properties of wood–plastic composites reinforced by virgin ABS and recycled ABS with 50 wt% wood fiber concentration. The virgin and recycled ABS composites were treated with three different coupling agents, styrene maleic anhydride (SMA), styrene ethylene butylene midblock-grafted maleic anhydride (SEBS-g-MA), and styrene acrylonitrile-grafted maleic anhydride (SANMA). The authors reported that the tensile strength and tensile modulus for both the virgin and recycled ABS increased in the presence of SMA and SANMA. This change was ascribed to the coupling agents improving adhesion between the fiber and matrix. However, SEBS-g-MA resulted in a decrease in tensile strength and tensile modulus, because SEBS-g-MA is an elastomer and has a much lower tensile modulus than either ABS or wood. The authors also observed that elongation at break values of the composite material made with recycled ABS did not differ much from that of composites manufactured using virgin ABS. Elongation at break value of composite made with virgin ABS was in the range of 1.05% to 1.4% and that of composite made with recycled ABS was in the range of 0.8% to 1.02%. However, the addition of coupling agents SMA and SANMA increased the elongation at break value by a considerable amount. Impact strength of virgin ABS wood composite without any coupling agent and recycled ABS wood composite was 63 and 55 J/m, respectively. Addition of the coupling agent SANMA (10 wt%) increased the impact strength value to almost 90 J/m for virgin ABS.

Chotirat et al.¹³⁴ investigated the adhesion mechanism and interfacial strength of an ABS matrix and wood sawdust. Prior to blending, the wood sawdust particles were treated with two different silane coupling agents: 3-methacryloxypropyl trimethoxysilane (KBM503) and N-(2(aminoethyl)3-aminopropyl trimethoxysilane (KBM603).

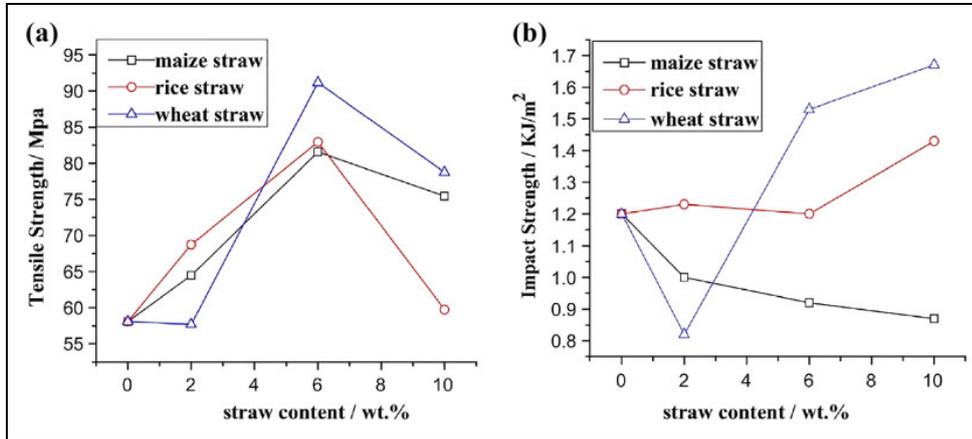


Figure 7. (a) Tensile; and (b) Impact strength values of straw fibres/PA 6 composites.¹²⁵ PA: polyamide.

The effects of the coupling agents and different wood sawdust concentration on the mechanical properties of the composite were investigated. The authors reported that an increase in sawdust concentration resulted in an increase in the values of tensile and flexural moduli; however, the values of elongation at break, tensile, flexural and impact strength decreased with increase in wood content. An ABS/sawdust composite with 9.1 and 33.3 wt% sawdust content were selected for coupling agent tests. The sawdust was treated with 0.5 wt% KBM603 and 1.0 wt% KBM503 solution. It was found that coupling agent KBM603 improved the interfacial strength between the matrix and wood particles better than KBM503.

Huang et al.¹³⁵ examined the mechanical properties and crystallization behavior of PA6 composite reinforced with wheat, maize, and rice straw. The three straw fibers were treated with NaOH solution and KH-550 (γ -aminopropyltriethoxysilane) was used as coupling agent. The authors observed that the tensile strength for all fiber types peaked at 6 wt% fiber content as shown in Figure 7(a). The wheat straw fiber composite demonstrated 56.9% higher tensile strength than a composite with pure PA6, followed by rice fiber at 42.8% higher, and finally maize straw fiber at 40.4% higher. According to the authors, this result is mainly due to good interfacial adhesion caused by alkali treatment of fibers. They also found that the impact strength of the composites with wheat and rice fiber initially declined but later increased with increase in fiber content (Figure 7(b)) and the impact strength peaked when the fiber content in the composite was highest, that is, 10 wt%. The results mean that wheat fiber increased the impact strength value by 39.2% in comparison with pure PA6, while rice fiber increased it by 19.2%. However, the addition of maize fiber reduced the impact strength with increase in fiber concentration. The authors attributed this finding to weak consistency between the maize fibers and matrix, whereas both rice and wheat fibers displayed good consistency. DSC analysis showed that the crystallization temperature of composites with straw fibers was

higher than that of pure PA6. It was observed that the crystallization temperature tends to decline slightly with increase in fiber content. However, the melting temperature of the composite structure remained the same as that of pure PA6, indicating that fiber type and content had no effect on the melting process of the composite structure. The authors also found that rice straw fibers had a negative effect on the degree of crystallinity, whereas wheat and maize fiber had varying effects. The 10 wt% wheat straw fiber composite had the highest degree of crystallinity followed by the 10 wt% maize straw fiber composite. X-ray diffraction analysis showed that the PA6 mainly consisted of γ crystals and there was only one diffraction peak located around $2\theta = 21.4^\circ$ in the (001) plane for the γ crystal. The addition of three types of straw fibers resulted in two diffraction peaks located at $2\theta = 20.4^\circ$ and 23.2° with varying intensities in the (001) and (002) planes of the PA6 α crystal. The three straw fibers resulted in the formation of α crystal in the structure of PA6 and produced several diffraction peaks at 2θ . The author attributed these varying diffraction peaks to the cellulose present in the fibers. Further, it was also observed that there was no noticeable change in the interplanar spacing d of pure PA6 and the composite, which led to the conclusion that the addition of fibers had no effect on the γ crystal structure.

One problem claimed to hinder the use of natural fiber in high end applications is their susceptibility to environmental effects. Salazar et al.¹³⁶ examined the biodegradation of coir, coir with latex, and sisal fibers used in manufacturing of automotive components by monitoring CO₂ generation and fungal growth. The authors observed approximately 10% biodegradation in all three samples after roughly 45 days, which is a clear signal that all three materials do not easily degrade. However, all the materials showed degradation of cellulose, which was 95% degraded in nearly all samples. The fungal growth test found that after 90 days, the sisal sample had greatest fungal growth, followed by the coir sample. The sample of coir with latex inhibited the least amount of fungal growth because of the anti-bacterial and anti-fungal properties of latex. A chemical composition test showed that the sisal fiber had the highest percentage of holocellulose, which is a key factor affecting the decay rate. Thus, the sisal sample had the greatest degradation of all the samples for the same period of time.

Processing techniques for NFRP composites

The processing techniques used to fabricate NFRP composites depend largely on the type of polymer matrix used. Processes such as hand lay-up,^{3,24} compression molding (for producing sheet molding compounds),^{3,6,9,76,137} resin transfer moulding,^{3,6,9,24,88,137} filament winding,³ pultrusion,^{24,137} and vacuum-assisted resin transfer moulding^{6,9,24,88} are suitable for thermosetting polymers. Processes such as injection moulding,^{3,6,9,24,88,137} extrusion,^{3,24,88,137} thermoforming,^{3,6,24} and compression moulding^{3,137} are appropriate for thermoplastic polymers. In addition, processing of thermoplastic composite depends also on fiber length, for example, injection molding is preferable for short fiber (approximately 3 mm long) thermoplastic composites, extrusion and, compression molding for long fibers (3–25 mm) and thermoforming, pultrusion, thermo-stamping, filament winding for continuous fibers.¹³⁸ Pultrusion can be used to produce

composite with both thermoset and thermoplastics matrices.^{137,138} However, the high melt viscosities of thermoplastic polymer cause difficulty in the melt impregnation of the reinforcement and thus the process is seldom used for thermoplastics.¹³⁸ Additional parameters for the appropriate process selection are cost, part geometry, ease of manufacturing, and performance attributes.⁶

Extrusion

Extrusion is a high speed fabrication technique and is commonly used to manufacture thermoplastic polymer composites.²⁴ In the process, polymer granules (either virgin or recycled) are fed into the heated barrel of an extruder, where mixing is performed. The extruder can be a single-screw or twin-screw extruder. The molten material flows inside the barrel of the extruder and is forced out through a die to produce the desired shape.^{3,6,137} Extrusion is a continuous fabrication process and can be fully automated. Crucial parameters are pressure, temperature, and delivery rate. The extruder removes any air trapped between the granules.³

Thermoforming

Fibers such as sisal, coir, jute, and ramie fiber can be processed by thermoforming.²⁴ This processing technique is used in the fabrication of thermoplastic sheet and film of varying thickness from 0.5 to 5 mm. The process involves heating the sheet and forming the sheet over a male mold or in a female mold. Low pressure is required to hold the sheet in place. Thermoforming molds are commonly made of aluminum and wood, or sometimes epoxy polymer. A vacuum or pressure is applied to ensure that the sheet stays against the surface of the mould.^{3,24,138} Once the part has formed, it is trimmed from the sheet and the leftover trimmed material is reprocessed. The advantages of thermoforming are the flexible tooling setup, flexibility in terms of adapting to customer needs, cost effectiveness, weight savings, suitability to be fully automated, suitability for small- and large-scale designs, and that paintable surface components can be formed.²⁴ Disadvantages includes higher cycle times and inability to make remedial changes to the part after forming.³

Injection molding

The process is mainly suitable for fabrication of thermoplastic polymers but processing of thermosets is also possible. During the process, the polymer granules are melted and injected at pressure into dies made up of either steel or aluminum. The material cools and solidifies into the desired shape inside the mold.^{3,138-140} The process of injection molding is suitable for complex shapes and minute details.^{3,6,9,138} The components produced have excellent surface quality and dimensional accuracy.^{3,6,9,24,140} Correct parameter setting is important for quality products, and key parameters include filling time, mold temperature, pressure, and melting temperature.²⁴ The advantages of injection molding includes short cycle time,^{3,6,9,24} little scrap,⁹ low cost,²⁴ higher production

volumes, low labor cost,^{6,9} and suitability to be fully automated.³ The disadvantages are the expensive machine setup and long lead times to fabricate the moulds.³ Injection molding is used for making products like houseware, automotive parts, furniture, and medical equipment.¹⁴⁰

Compression molding

Compression molding is a closed molding operation under high pressure. The reinforcement and the matrix are placed in a metallic mould.³ The compression molder consists of two parts, the bottom plate, which is stationary, and the upper part, which is movable. The setup is supplied with the necessary heat and pressure according to the shape and size of the part. The reinforcement and the matrix are mixed together by the application of heat and pressure. The curing process takes place at room temperature and the composite part is removed from the mould.¹⁴⁰ Compression molding is used to produce thin, light, and strong automotive panels and structures. The compression molding process has high processing speed and low fiber abrasion.^{6,140} Other advantages includes short cycle time and low scrap amounts. For example, a 3-D component with a high forming degree can be processed in under 2 min.⁹

Conclusion

The manufacture of lightweight and fuel efficient vehicles is vital for the future of the transportation sector and to meet increasingly strict environmental regulations aiming to reduce the global carbon footprint and create a sustainable future. In the past, the transportation sector has always been able to adapt to changed demands of consumers, governments, and environmental organization by making changes to the design, material, or manufacturing techniques while at the same time remaining competitive in the market. Already, vehicle manufacturing has witnessed a shift from metal to aluminium and, more recently, from aluminium to polymer composites. Thus far, the majority of polymeric composites are reinforced with glass or carbon fibers, however, a shift is already in full swing from synthetic fibers reinforced with polymer composites to NFRP composites, primarily due to the unique characteristics, low cost, and environmental impact of the latter. The main goal of this review article was to determine the status quo of NFRP usage in the transportation sector, especially in the three primary transportation sectors of automotive, aerospace, and maritime transport, with emphasis on recycled engineering plastics as the polymer matrix.

From previous studies, it can be seen that PP, PE, and epoxy are the most common polymer matrices used in NFRP composites for transportation applications. These commodity polymers are generally not suitable for structural and high performance applications, and thus, they are commonly used in nonstructural applications such as body panels, headliners, seats, interiors, and dashboards. In contrast, engineering plastics such as ABS, PA, PS, PC, PET, and PBT are widely used in under-the-hood operation and other structural applications. However, due to their relatively higher cost, most vehicle manufacturers are reluctant to use these plastics on a large scale. However, the

use of recycled material may offer a solution to address these misgivings. Many researchers have reported that these engineering plastics have superior properties like greater strength, toughness, heat resistance, and stability than commodity plastics, and they have noted that even after recycling, engineering plastics exhibit slightly superior properties than commodity plastics. These properties can be enhanced further by reinforcing engineering plastics with natural fibers to match industry standards. Consequently, by utilizing recycled engineering plastics, manufacturers can not only contribute toward meeting environmental targets by reusing material which would otherwise have been disposed of by incineration or dumping in landfill, thus causing more emissions, but can also reap potential financial benefit from reclaiming a high value waste stream.

Some major challenges hindering wide usage of NFRP composites in transportation sector are poor interfacial adhesion, high moisture absorption, and low fire resistance. Poor interfacial adhesion between the polymer matrix and fiber is caused by the incompatibility of hydrophilic natural fibers with hydrophobic nature of engineering plastics. On other hand, the high moisture absorption rate of natural fibers greatly limits their usage in maritime transportation, where contact with water is inevitable. Finally, the low fire resistance of NFRP composites is a great challenge in aircraft and automobile applications where the operating conditions generate heat and the safety of the passengers is the prime condition. However, it has been observed that certain surface treatment techniques such as coupling agents, coating, and fire retardants for natural fibers can greatly aid in overcoming these challenges. Therefore, it is important to further investigate the properties of NFRP composites and surface treatment techniques for composites made of natural fibers and recycled engineering plastics with the aim of improving their suitability for high end applications such as transportation as a part of worldwide efforts to produce lightweight vehicles, improve fuel efficiency, and reduce carbon emissions.

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Publication II

Chauhan, V., Kärki, T., and Varis, J.

**Effect of Fiber Content and Silane Treatment on the Mechanical Properties of
Recycled Acrylonitrile-Butadiene-Styrene Fiber Composites**

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Article

Effect of Fiber Content and Silane Treatment on the Mechanical Properties of Recycled Acrylonitrile-Butadiene-Styrene Fiber Composites

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Abstract: The aim of the present study was to investigate the effects of fiber content and then silane treatment on the mechanical performance of the natural fiber composites of recycled acrylonitrile-butadiene-styrene (ABS) provided by the automotive sector. Wood and palmyra fibers were used as fillers in 10% and 20% fiber content composites. The fibers were treated with *N*-(2-Aminoethyl)-3-aminopropyltrimethoxysilane to improve the interfacial adhesion between fibers and polymer matrices. The mechanical properties of the composites were determined by tensile and impact tests. Morphological analysis was later performed using a scanning electron microscope (SEM). According to the experiment results, the tensile and impact strength of both wood and palmyra fibers increase after silane treatment. However, for the low-wood-fiber-content composite, the tensile and impact strength decrease after silane treatment due to the presence of an excess amount of silane relative to fiber content. The addition of wood and palmyra fibers significantly improved the tensile modulus of composite material and further increases slightly after silane treatment. Finally, SEM analysis shows a homogenous mix of fibers and polymer matrices with fewer voids after silane treatment, thereby improving interfacial adhesion.

Keywords: recycled ABS; natural fibers; mechanical properties; automotive



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1. Introduction

Acrylonitrile-butadiene-styrene (ABS) is one of the most used thermoplastic terpolymers. It is prepared by polymerizing styrene and acrylonitrile in the presence of polybutadiene [1–3]. Acrylonitrile offers heat and chemical resistance, while butadiene provides impact resistance and styrene provides processability and surface properties [4–6]. Thus, ABS terpolymer exhibits good mechanical properties, easy processing, chemical resistance, good surface appearance, and recyclability [1,3,4,7,8]. ABS is widely used in electrical and automotive applications such as household appliances, automobile body parts, and wheel covers [1,5,9]. Due to the widespread applications, a large volume of ABS is also available in the form of post-consumer polymer waste after the end of life of its applications. This post-consumer polymer waste is utilized as an aggregate in concrete and road construction [10–12] as well as a matrix material in natural fiber polymer composites (NFPCs).

NFPCs are composed of natural fibers as a reinforcement/filler material and a polymer matrix. NFPCs are used as an alternative material in furniture, automotive parts, window panels, household equipment, and decking [8,13,14]. Previous studies related to ABS as a polymer matrix in NFPCs primarily focused on using virgin ABS [2,3,7,13,15–17]. However, recycled ABS extracted from automotive or electrical waste streams can also be utilized in making composite materials. Additionally, due to strict legislation in European and several Asian countries regarding waste from electrical and electronic equipment and automotive end-of-life requirements, product manufacturers need to consider the

environmental impact of materials used in manufacturing goods [18,19]. Therefore, the material recovered from such high-value waste streams can easily be reused in other applications or even in similar ones.

One main issue with using recycled ABS or any other polymer extracted from post-consumer applications is that these polymers are not free of impurities which are either caused by their usage during the product life cycle or due to mixing during the collection process. These impurities lead to detrimental effects on the mechanical properties of the recycled material and change the thermal characteristics of the material [20]. Studies have also found that with even 1 wt.% of impurities in the polymer material, the properties' reduction was severe and unacceptable when compared to virgin polymer material [21,22].

Natural fibers are derived from plant, animal, and mineral sources. Plant fibers such as wood, hemp, kenaf, sisal, flax, palm, and jute are commonly used for reinforcement in composites. Wood is the most common plant fiber used as a filler in NFPC materials. However, the choice of a natural fiber as a filler in NFPCs largely depends on the source proximity; for example, jute and palm are commonly used in Asia, whereas in Europe wood and hemp are the most common. Furthermore, for wood fibers the type of source wood can differ drastically from one species of tree to another, which can also affect the overall properties of NFPC materials [23]. In Europe, spruce is the most common type of wood fiber used in composites. On the other hand, palmyra is very economical and widely cultivated in tropical regions of Africa and Asia for its usage in over 800 applications. Palmyra fibers are extracted from the leaf stalks of the palmyra tree and used in producing ropes, painting brushes, broom bristles, fans, baskets, mats, and hats. Due to their low cost and abundance, these fibers could easily be utilized as fillers in NFPC materials [24–26]. The chemical composition of both spruce wood and palmyra fiber is shown in Table 1.

Table 1. Chemical composition of spruce and palmyra fibers.

	Spruce [27]	Palmyra [24]
Cellulose (%)	42.00	58.58
Hemicellulose (%)	27.30	22.80
Lignin (%)	27.40	13.48
Extractives and Wax (%)	2.00	0.53

The main issue with NFPC materials is the incompatibility of fibers and polymer matrices. This is due to difference between the hydrophilic nature of fiber and hydrophobic nature of polymers, which results in weak interfacial adhesion [1,13,15–17]. Surface modification methods, such as silane treatment of natural fibers, can improve interfacial adhesion, thus improving the properties of composite materials [1,4,15–17,28]. Other surface modification methods include alkaline treatment [29], acetylation [30], enzyme treatment [31], plasma treatment [32], and grafting [33]. The introduction of silane reduces the hydroxyl groups of natural fibers and thus improves interfacial adhesion [34]. For example, Wang et al. [28] studied the effects of silane treatment on the mechanical properties and thermal behavior of bamboo-fiber-reinforced polypropylene composites, and they found that the tensile and flexural strength of silane-treated fibers composites were increased compared to the composition of untreated fibers. However, during the silane treatment silane monomers can also undergo a self-condensation, where they form polysiloxane molecules by reacting with each other, and these molecules are less reactive [35]. As observed by Orue et al. [36], who studied the effect of alkaline and silane treatment on the mechanical properties of sisal fiber/poly(lactic acid) composites, untreated fiber composites and silane-treated fiber composites exhibit low mechanical properties compared to alkaline-treated and alkaline + silane-treated composites at varying fiber contents.

Mechanical properties of NFPCs with ABS as the polymer matrix have been extensively investigated by various research groups across the world. Neher et al. [2,37] examined the mechanical and physical properties of ABS/palm fiber composites and concluded that the tensile strength and flexural strength of the composite material decrease with

the introduction of fiber content, but that the stiffness of the material increases. Similarly, Martins et al. [38] observed that the introduction of fiber content considerably increases the stiffness of ABS/textile fiber composite materials. Chotirat et al. [15] investigated the mechanical properties of ABS/wood sawdust composites and observed that increasing the wood sawdust content in the composite material increases the modulus but reduces the strength of the material. They also found that a low concentration of silane treatment can improve mechanical properties while a higher percentage has an adverse effect on them. However, most of the previous studies had been focused on virgin polymer materials, and not much focus has been given to prospects of using recycled polymer material in composite manufacturing. Therefore, in this study the effects of fiber content and silane treatment on the mechanical properties of recycled ABS composites containing wood and palmyra fibers were investigated. The main objective of the study was to study how mechanical properties are influenced by silane and varying fiber contents and to identify suitable composite compositions for future automotive applications. The mechanical properties were examined using tensile and impact testing. Furthermore, a scanning electron microscope (SEM) was used to analyze the tensile-fractured surfaces of the composites.

2. Materials and Methods

2.1. Materials

The waste polymers, primarily consisting of ABS and other styrene-based polymers such as ABS + Polymethyl methacrylate (PMMA), ABS + Polystyrene (PS), ABS + Styrene acrylonitrile resin (SAN), ABS + Acrylonitrile styrene acrylate (ASA), and ABS + Acrylonitrile ethylene styrene (AES) were supplied by Oili Jalonon Oy (Finland) and Majava Group Oy (Finland). These waste materials were extracted from automotive, motorbike, and truck polymer parts such as motorbike frontal fairings, side fairings, tank covers, truck bumpers, car emergency triangle reflectors, and door panels, as shown in Figure 1. The composition of the recycled polymers is shown in Table 2. The density and melt flow indices of the recycled ABS mixture (R-ABSmix) were 1032 kg/m^3 and 14.98 g/10 min , respectively.



Figure 1. Scrap polymers from various automotive parts.

Table 2. Composition of recycled polymers.

Polymer Type	Weight (kg)	Composition (%)
ABS	63.47	80.77
ABS + SAN	1.38	1.75
ABS + ASA	3.62	4.61
ABS + AES	1.25	1.59
ABS + PMMA	6.04	7.69
ABS + PS	2.82	3.59
Total	78.58	

The wood fiber was obtained from Stora Enso Oy, and palmyra (bassine fiber) was obtained from Mirja Dahl Ky (Finland). The fiber size used for composite preparation was less than 1 mm for both fibers, as shown in Figure 2. The density of wood fiber and palmyra fiber was 457 kg/m³ and 365 kg/m³, respectively. In addition, the moisture content for wood and palmyra fibers was 12% and 10%, respectively. The lubricant used was STRUKTOL[®] TPW 113, and *N*-(2-Aminoethyl)-3-aminopropyltrimethoxysilane was used for the surface treatment of fibers.

**Figure 2.** (a) Sample of wood fibers. (b) Sample of palmyra fibers.

2.2. Silane Treatment

Both wood and palmyra fibers were treated with 3% (*w/w*) *N*-(2-Aminoethyl)-3-aminopropyltrimethoxysilane. The most common method used for silane treatment is immersing the fibers in the silane solution [28,39]. However, since the fiber size was small, a spraying method was chosen, which is a relatively easy way to conduct fiber treatment with silane [40]. In this method, a thin layer of fibers was placed on a silicone sheet as shown in Figure 3 and sprayed with a silane solution using a handheld pump sprayer [35]. The spraying method is preferred over soaking the fibers in the silane because it ensures an even distribution of silane and controls the amount of silane in the material. Afterward, the mixture was dried for 30 min at 50 °C before making the composite agglomerate.



Figure 3. Thin layer of fibers sprayed with silane solution.

2.3. Composite Preparation

Both treated and untreated fibers were compounded with polymers using STRUKTOL[®] TPW 113 3% (*w/w*) in Plasmec COMBIMIX-RV/100/200/FV/W. The material composition, density, and designation code for different composite samples are shown in Table 3.

Table 3. Composition of recycled polymer composites.

Designation	Fiber	Polymer (%)	Fiber (%)	Lubricant (%)	Silane (%)	Density (kg/m ³)
R-ABS/10WF	Wood	87	10	3	-	1067
R-ABS/10WF-S	Wood	84	10	3	3	1079
R-ABS/20WF	Wood	77	20	3	-	1074
R-ABS/20WF-S	Wood	74	20	3	3	1083
R-ABS/10PF	Palmyra	87	10	3	-	1082
R-ABS/10PF-S	Palmyra	84	10	3	3	1068
R-ABS/20PF	Palmyra	77	20	3	-	1067
R-ABS/20PF-S	Palmyra	74	20	3	3	1087

Initially, the polymer–fiber mixture was first heated in a convection oven at 260 °C for 20 min. Then, the mixture was transferred into the mold of a Stenhoj 40-ton compression molding machine to produce composite cakes, as shown in Figure 4. The dimensions of composite cakes were 110 × 110 × 10 mm. The composite cakes were compressed for a minute in the compression molding machine. After removal from the mold, the composite cakes were air cooled and kept for over 24 h in a humidifier at 23 °C and at 50% relative humidity.

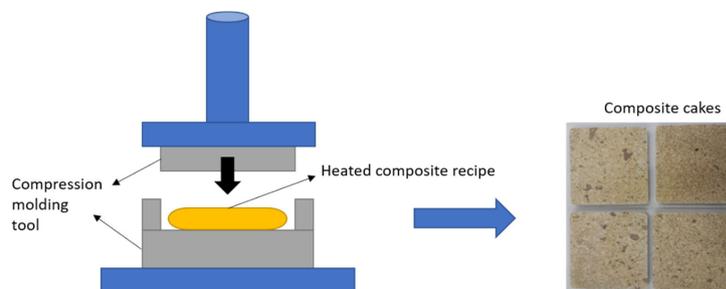


Figure 4. Illustration of the compression molding process.

2.4. Mechanical Properties Testing

Composite cakes were cut into the shape of a dog bone for tensile testing according to ASTM D638. The samples were prepared according to type 1 specifications. Ten samples for each composite recipe (overall 8, as shown in Table 3) were prepared for tensile testing purposes and kept in a humidifier for 24 h at 23 °C and at 50% relative humidity. All the composite samples were tested at room temperature for tensile properties using a universal testing machine (Zwick Roell Z020) at a test speed of 2 mm/min according to ASTM D638. The experimental data were collected and processed automatically using testXpert II software.

The impact strength of the samples was tested using a Zwick 5102 impact testing machine according to SFS-EN ISO 179 1 by using Method ISO 179 1/1fU. Prior to testing, the samples were prepared according to type 1 test specimens— $80 \pm 2 \times 10 \pm 0.2 \times 4 \pm 0.2$ mm, under Charpy unnotched impact strength. Fifteen samples of each composite recipe were prepared and kept for 24 h at 23 °C and at 50% relative humidity in a humidifier before testing. The testing was performed using a pendulum impact tester, with a hammer size of 5 Kpcm for all composite recipes. The Charpy impact strength was calculated according to the equation:

$$a_{c,u} = \frac{E_c}{h \times b} \times 10^3$$

where E_c is the energy absorbed by test specimen (in joules), h is the thickness of the test specimen (in mm), and b is the width (in mm) of the test specimen.

2.5. Surface Morphology Analysis

The morphology of both wood and palmyra fiber composites before and after silane treatment was examined using a Hitachi SU3500 scanning electron microscope (SEM) at 10.0 kV accelerating voltage using a backscattered electron (BSE) detector. Tensile-fractured specimens of all material samples were examined, and SEM images were obtained at $\times 500$ magnification. All the samples were imaged without coating.

2.6. Statistical Analysis

Due to the variation in the observed mechanical properties a statistical analysis of the untreated composite samples with reference to the R-ABSmix material was performed, to see the impact of fiber content on the mechanical properties. Similarly, treated composite recipe results were analyzed with reference to corresponding untreated composite recipes to see the impact of silane treatment on mechanical properties. The statistical analysis was performed in Daniel's XL Toolbox Excel software and by using a one-way ANOVA Bonferroni–Holm post hoc testing algorithm.

3. Results

3.1. Wood Fiber Composites

The strength of natural-fiber-reinforced composites is affected by various factors, such as fiber type, fiber length, fiber content, matrix, and interfacial adhesion between fiber and matrix [2,41]. The fiber content and effect of silane treatment on the mechanical properties of the wood-fiber-reinforced composites are shown in Table 4. As per the statistical analysis, tensile strength, tensile modulus, and impact strength of both untreated 10% and 20% wood fiber composites were found to be significantly impacted. On the other hand, only the tensile strength of treated 10% and 20% wood fiber composites was significantly changed and there was no significant change in the tensile modulus values of the silane-treated composites. The impact strength of 10%-silane-treated wood fiber composites also display a significant change, while no significant change was observed in the 20%-silane-treated wood fiber composites. The tensile strength of untreated wood-fiber-reinforced composites decreased with an increase in fiber content. This is mainly attributed to the weak interfacial bonding between wood fibers and the ABS matrix due to the incompatible nature of ABS, which is hydrophobic, and wood fibers, which are hydrophilic. This interfacial bonding strength further decreased with an increase in fiber content in the composite [15,42].

Table 4. Mechanical properties of the wood fiber composites and R-ABSmix.

	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)	Impact Strength (kJ/m ²)
R-ABSmix	16.42 ± 1.20	1.79 ± 0.07	1.00 ± 0.19	5.98 ± 0.92
R-ABS/10WF	9.39 ± 0.97 ^s	1.93 ± 0.11 ^s	0.48 ± 0.06	2.90 ± 0.65 ^s
R-ABS/10WF-S	7.98 ± 1.69 ^s	1.96 ± 0.23 ^{ns}	0.49 ± 0.12	2.27 ± 0.60 ^s
R-ABS/20WF	7.12 ± 1.66 ^s	2.05 ± 0.23 ^s	0.39 ± 0.12	2.25 ± 0.34 ^s
R-ABS/20WF-S	9.28 ± 1.03 ^s	2.10 ± 0.08 ^{ns}	0.46 ± 0.07	2.36 ± 0.50 ^{ns}

^{s, ns} statistically significant and no significant changes with a 95% confidence level, respectively.

However, as stated in previous studies [4,15,42–44], surface modification methods such as silane treatment tend to improve the interfacial adhesion between wood fibers and polymer matrices and thus result in better mechanical properties. Similar results were observed for R-ABS/20WF-S, as the tensile strength value increased by 30.33% after silane treatment. However, for R-ABS/10WF-S, the tensile strength decreased by 15% after treatment. This observation had been reported in some previous studies [15,17,36,42,45]. Kimchiang et al. [16] studied low-fiber-content samples, where silane concentration was above 0.5–1.5%, and cases where a certain type of silane was used. The self-condensation reaction occurring on the surface of composites causes the formation of flexible polysiloxane molecules, which reduces the interfacial adhesion between treated wood fibers and matrices at low fiber contents [16]. Similarly, Chotirat et al. [15] observed that the optimum silane concentration for wood sawdust/ABS composites is around 0.5–1.0% for both low and high fiber content [15]. Furthermore, Raj et al. [45] observed that silane-treated wood/PP composites exhibit poor tensile strength and expressed that the availability of silanol groups necessary for the formation of covalent bonds was restricted by the lower surface coverage of wood fibers.

On the contrary, the tensile modulus of wood composites is usually higher than that of polymer materials and further increases as the fiber content of the composite increases [4,15,16]. The tensile modulus indicates the rigidity of the composite material [46]. During the experiment it was observed that tensile modulus increases by 5.8% with an increase in fiber content from 10 to 20%. Later, it was observed that after silane treatment the tensile modulus of R-ABS/10WF-S and R-ABS/20WF-S increased marginally compared to the untreated recipes, indicating that silane treatment has a marginal impact on the tensile modulus of composite materials and that the modulus largely depends on the fiber. Chotirat et al. [15] observed similar observations, where the tensile modulus slightly increases in both low- and high-fiber-content samples.

The impact strength of a polymer is influenced by the presence of a rubbery phase in the copolymer [4]. In the case of NFPCs, the impact property is dependent on many factors such as geometry, fiber arrangement, and fiber matrix interface [41]. Thus, the introduction of wood fibers reduces the toughness of the material. This is due to the restriction of polymeric chain mobility caused by the addition of wood fibers, thus reducing energy absorption capacity [4]. Further, poor interfacial adhesion causes voids in composite structure and thus leads to crack propagation during impact testing [42]. The impact strength of R-ABS/10WF was reduced by 50% in comparison to R-ABSmix and further decreased by 60% as the fiber content increased. Improvement of the impact strength of fiber-reinforced polymer composites is achieved by improving the interfacial adhesion between fiber and matrix or by adding an impact modifier that is flexible and elastic in nature [15]. However, the impact strength of R-ABS/10WF-S was 21% lower than the R-ABS/10WF composite. It is assumed that, similar to tensile strength, the reason for this decrease was the presence of an excess amount of silane concentration during fiber treatment. For higher fiber contents, silane treatment can improve the bonding between fibers and matrices, thus reducing voids and cracks [42]. As observed for R-ABS/20WF-S, the impact strength increased by about 5% in comparison with R-ABS/20WF.

3.2. Palmyra Fiber Composites

Table 5 shows the effect of silane and fiber content on the mechanical properties of the palmyra-fiber-reinforced composites. After statistical analysis, it was observed that the tensile strength and impact strength of both 10% and 20% untreated palm fiber composites and the tensile strength of silane-treated palm composites exhibit statistically significant changes. Meanwhile there were no significant changes observed in the tensile modulus of all the palm composite recipes. Finally, the impact strength of 10%-silane-treated palm fiber composites displays significant change, while no significant change was seen in 20%-silane-treated palm fiber composites. Similar to the wood fiber composites, the introduction of untreated palmyra fiber into the polymer matrix causes the tensile strength to decrease drastically and further decrease with an increase in fiber content [2,41,47]. However, in contrast to R-ABS/10WF-S, the introduction of silane into the composite causes the tensile strength of the R-ABS/10PF-S composite to drastically increase by 60.38%. In addition, the tensile strength for R-ABS/20PF-S also increases by 41.55% after silane treatment. Similar observations were found in previous palmyra- and other palm-fiber-related studies [26,46,48]. It is assumed that palmyra fibers, even at low fiber content, react to silane solution in a positive manner, and no self-condensation process takes place. Thus, even at a higher silane concentration with low palmyra fiber content, the interfacial adhesion between fibers and matrices improves. Additionally, it can also be assumed that the surface coverage of palmyra fibers was better than wood fibers, which does not restrict the availability of the silanol group for the formation of covalent bonds.

Table 5. Mechanical properties of the palmyra fiber composites and R-ABSmix.

	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)	Impact Strength (kJ/m ²)
R-ABSmix	16.42 ± 1.20	1.79 ± 0.07	1.00 ± 0.19	5.98 ± 0.92
R-ABS/10PF	6.26 ± 1.97 ^s	1.82 ± 0.34 ^{ns}	0.41 ± 0.12	2.27 ± 0.52 ^s
R-ABS/10PF-S	10.04 ± 2.79 ^s	1.92 ± 0.12 ^{ns}	0.76 ± 0.20	3.12 ± 0.87 ^s
R-ABS/20PF	6.04 ± 1.83 ^s	1.88 ± 0.13 ^{ns}	0.48 ± 0.27	2.41 ± 0.67 ^s
R-ABS/20PF-S	8.55 ± 0.74 ^s	2.00 ± 0.10 ^{ns}	0.52 ± 0.13	2.47 ± 0.63 ^{ns}

^{s, ns} statistically significant and no significant changes with a 95% confidence level, respectively.

As with wood fiber composites, the tensile modulus increases with an increase in fiber content in the composite material. It was observed that tensile modulus increases by 3.3% with an increase in fiber content. After silane treatment, tensile modulus values increase by 5.5% and 6.3% for R-ABS/10PF-S and R-ABS/20PF-S, respectively, compared to untreated composite recipes. The impact strength of composites decreased when the

fibers were introduced; this is mainly because of the poor wetting of fibers by the polymer matrix [49]. However, in contrast to wood fibers, the impact strength of untreated palmyra fiber composites increased slightly as the palmyra fiber content increased. Similar results were seen in a previous study [47]. Furthermore, after silane treatment, the impact strength for both R-ABS/10PF-S and R-ABS/20PF-S composites increased by 37.45% and 2.5%, respectively. Similar observations were reported in previous studies related to palmyra fibers [49,50].

3.3. Morphological Investigations

In order to further understand the effect of silane treatment, the tensile-fractured surfaces of both wood and palmyra fiber composite specimens were examined with an SEM. Figure 5 shows the surface morphologies of (a) R-ABS/10WF, (b) R-ABS/10WF-S, (c) R-ABS/20WF, and (d) R-ABS/20WF-S. From the untreated material sample images, it is visible that due to the presence of voids, pullout fibers, and poor agglomeration of fibers throughout the polymer matrix, the material property of the composite materials decreases. This also confirms the incompatibility of fiber and polymer materials in the composite. However, after silane treatment for both 10% and 20% wood fiber content, the surface was found to be slightly more homogeneous, especially for R-ABS/20WF-S, thereby leading to an improvement in the tensile strength value. Finally, the presence of a high silane concentration relative to fiber content in R-ABS/10WF-S causes the tensile strength to decrease.

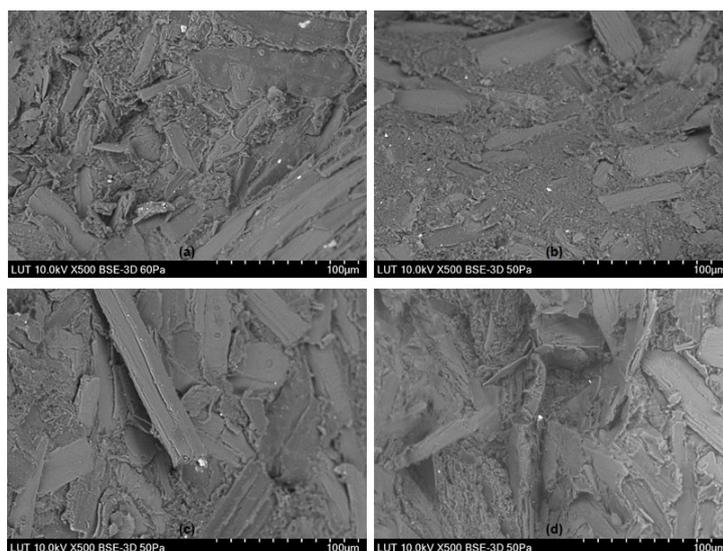


Figure 5. SEM images of wood-fiber-reinforced composites (a) R-ABS/10WF; (b) R-ABS/10WF-S; (c) R-ABS/20WF; and (d) R-ABS/20WF-S.

Figure 6 shows the surface morphologies of (a) R-ABS/10PF, (b) R-ABS/10PF-S, (c) R-ABS/20PF, and (d) R-ABS/20PF-S. For untreated palmyra fiber composites, the presence of large voids, pullout fibers, and porous fiber structures caused the tensile strength of R-ABS/10PF and R-ABS/20PF to decrease. However, the introduction of silane treatment to both 10% and 20% fiber content composites saw a drastic increase in tensile strength. This is also visible in the somewhat homogenous mixture for both R-ABS/10PF-S and

R-ABS/20PF-S; thus, adding silane improves the interfacial adhesion between fibers and polymer matrices.

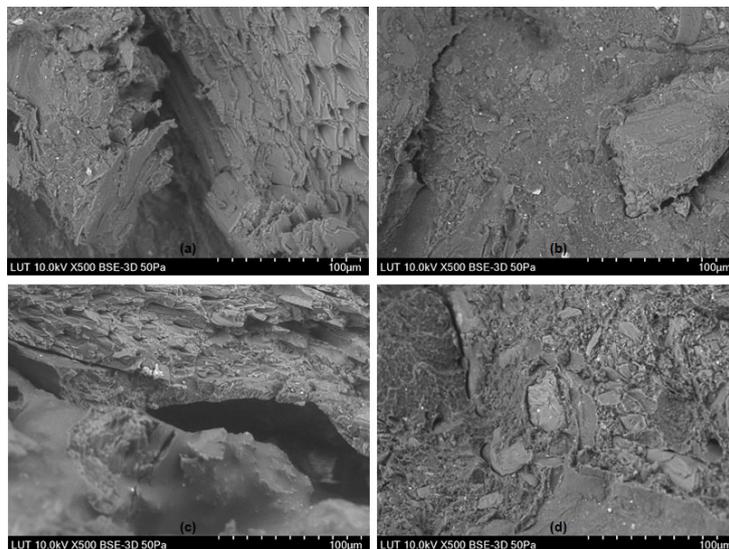


Figure 6. SEM images of palmyra-fiber-reinforced composites (a) R-ABS/10PF; (b) R-ABS/10PF-S; (c) R-ABS/20PF; and (d) R-ABS/20PF-S.

4. Conclusions

This study investigated the effect of fiber content and silane treatment on the mechanical properties of recycled ABS composites containing wood and palmyra fibers. Tensile and impact properties of the composite samples were examined. For the wood fiber composites, the tensile strength and impact strength decrease with an increase in fiber content. After silane treatment, the tensile strength for 20% wood fiber content increases due to improved interfacial adhesion between wood fibers and the polymer matrix. However, for 10% wood fiber content the tensile strength decreases after silane treatment due to the relatively high concentration of silane used, which results in a self-condensation reaction occurring on the surface of the composite, causing the formation of polysiloxane molecules, further negatively affecting the fiber and matrix bonding. The tensile modulus of untreated wood fiber composites increases with an increase in fiber content in the composite. Further, it was observed that silane treatment has a marginal positive effect on the tensile modulus values for both 10% and 20% wood fiber composite recipes. Similarly, tensile strength decreases with an increase in palmyra fiber content. However, after silane treatment the tensile strength increases for both 10% and 20% palmyra fiber contents. This indicates no self-condensation reaction occurring on the surface of the composite for 10% palmyra fiber content. Similar to wood fiber composites, the tensile modulus of the palmyra fiber composite increases as the fiber content increases. After silane treatment, the tensile modulus value for both 10% and 20% palmyra fiber contents increase slightly, further indicating the marginal effect of silane on the tensile modulus values of NFPCs. In contrast to wood fibers, the impact strength of the untreated palmyra fiber composites increases with an increase in fiber content. In addition, after silane treatment the impact strength of both the 10% and 20% palmyra fiber composites increases further, indicating improved interfacial bonding between fiber and matrix. The morphological analysis of both wood and palmyra fiber composites showed

that surfaces of silane-treated composite specimens were more homogenous with fewer voids and pullout fibers compared to untreated composite specimens. Further, the presence of a relatively high concentration of silane in the composite with respect to fiber content causes certain specimens' properties to deteriorate. Considering the relatively low quality of the recycled materials, the properties of R-ABSmix and natural fiber composites were at an acceptable level, especially for the tensile modulus, which indicates high stiffness of the material. For further research, a proposal could be made to identify applications of this recycled material composite.

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Publication III

Chauhan, V., Kärki, T., and Varis, J.

**Design of Tooling System and Identifying Crucial Processing Parameters for
NFPC Manufacturing in Automotive Applications**

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Article

Design of Tooling System and Identifying Crucial Processing Parameters for NFPC Manufacturing in Automotive Applications

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Abstract: The aim of this study was to design a tooling system for manufacturing automotive components using a natural fiber polymer composite (NFPC) material. As a case study, an automotive battery cover was selected and a compression molding tool was designed, keeping in mind the need for the simplicity of the tool and ensuring the low cost of this process. However, since the original part was injection-molded with virgin polypropene, some vital changes made in the part and tool design process were documented as a guideline to show new designers how to approach the design of parts and tools using a natural fiber polymer composite material. Additionally, the challenges faced during the manufacturing of composite parts with the new tool were also documented and solutions to these challenges were suggested for large-scale production. Finally, compressive testing was performed to evaluate the performance of the structure of the designed part and to compare the recycled polymer with NFPC material. Both wood and palm fiber composite material perform better in compression testing compared to the recycled polymer material.



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Keywords: natural fiber; recycled polymer; automotive; design methods; tooling system; processing parameters

1. Introduction

Nowadays, polymer matrix composites are used extensively in the automotive industry due to their lightweight and superior properties. These polymer matrix composites are reinforced by either synthetic or natural fibers. Synthetic fibers such as glass and carbon fiber are widely used in manufacturing fiber-reinforced polymer composites for the automotive industry. However, these fibers have certain disadvantages, such as the high density of glass fiber; the high cost of carbon fiber; they are difficult to recycle, non-renewable, and non-biodegradable; and the high energy consumption in their production [1]. Due to these reasons, many car manufactures are shifting towards the use of natural fibers, such as plant fibers or mineral fibers, in producing components for their car models.

Natural fibers possess desirable properties, such as being low-cost, abundant, renewable, and biodegradable, as well as requiring less energy in their production. However, natural fibers also have several drawbacks, such as their high moisture content, low fire resistance, and weak interfacial adhesion with the polymer matrix in the composite [2,3]. The efficiency of the interfacial bonding of fibers and the polymer matrix in the composite determines its overall mechanical properties [4]. In a composite, fibers provide strength and stiffness, but they cannot withstand heavy loads directly, thus a polymer matrix's primary function is to aid in the transfer of stress [1]. Surface treatment methods have been reported to improve the interfacial adhesion between fibers and the polymer matrix. Hence, numerous studies [5–8] have been conducted on this issue in the past and an improvement in the mechanical properties of composites after the surface treatment of fibers has been reported. Ma et al. [6] observed that alkali treatment of bamboo fibers improves the adhe-

sion between the fibers and acrylonitrile-butadiene-styrene (ABS) matrix, which resulted in a significant increase in impact strength of the composite. Similarly, Saidah et al. [7] observed an increase in tensile strength after alkali treatment of rice straw fiber-reinforced composites for varying fiber content compared to untreated fiber-reinforced composites. Kim et al. [8] observed significant improvements in tensile, flexural and impact strength of the wood fiber-reinforced polypropylene (PP) after chemical treatment of wood flour with various types of silane.

In automotive, natural fiber polymer composites (NFPCs) can be used as a substitute for both metal and polymer components. Currently, car manufacturers use NFPCs in both internal and external applications, such as door panels, luggage compartments, seat backrests, headliners, and door linings [9–13]. Natural fibers such as wood, flax, hemp, sisal, and kenaf are most commonly used in the automotive sector as reinforcing materials in polymer composites. Daimler uses flax, hemp, sisal, and coconut fibers in their A-, C-, E-, and S-class models [9]. Similarly, BMW uses flax and sisal in their 7-series model [13].

Previous studies have applied both synthetic fiber composites [14–18] and NFPCs [5,7,19–25] in various automotive applications. Saidah et al. [7] examined the effects of fiber loading and alkali treatment on a rice straw fiber-reinforced composite for an automotive bumper beam application. They concluded that alkali-treated rice fiber-reinforced composites with a 30% fiber loading possess superior mechanical properties compared to a standard bumper and can be a viable alternative to the existing material used. Nayak et al. [25] investigated the thermo-mechanical properties of Ceiba pentandra bark fiber/poly (vinyl) alcohol composites for dashboard and door panel applications. They found that 20 wt.% fiber content composites exhibited superior mechanical and thermal properties and were highly suitable in the fabrication of automotive dashboard and door panels. However, there are few under-the-bonnet applications for NFPCs in the automotive sector. This is mainly due to the harsh working environment of the bonnet area, which can negatively affect NFPC components due to their low fire resistance.

Epoxy, PP, ABS, polyamide, and polycarbonate are commonly used in polymer matrixes in NFPCs in the automotive sector. For example, a flax/PP composite was used to produce engine encapsulations for Mercedes-Benz Travego coaches and a flax-sisal/epoxy composite was used to produce the door panels of their E-class model [26]. Currently, virgin polymers are primarily used as polymer matrixes in NFPCs in automotive applications [5,7,25]. However, due to the sheer volume of polymers used in various components of automobiles, it is also possible to reuse some polymers extracted from automobiles after their end of life and reuse them in automotive in the form of NFPCs. These recycled polymers might have a slightly lower performance value in comparison with virgin polymers, but they are an easily available, cheap, and sustainable option and, more importantly, can be used to create viable matrixes for NFPCs.

The most common manufacturing process used for producing NFPCs is injection molding [8,27–29], compression molding [27,30–33], and resin transfer molding [34,35]. Compression molding is a high-volume production process used to manufacture composites for automotive components. In this process, the composite material is placed into the cavity of a fixed female tool and formed by applying pressure to the material with the help of a moving male tool. The material is cured utilizing pressure and heat and formed into the desired shape within the gap of male and female tools [36]. Overall, it is a low-cost, simple, and high-volume process that is suitable for manufacturing a variety of shapes. Due to the simplicity of the process, the tooling system for compression molding also costs less compared to injection molding [37]. Thereby, compression molding is a low-cost and viable process for manufacturing NFPCs composite for automotive applications.

It is important to study the role recycled polymers can play in the automotive industry and how car manufacturers can use these recycled polymers as composite materials for industrial-scale production. Thus, it is necessary to identify the key processing parameters that promote the use of recycled polymers in composite materials to be used as primary raw materials for manufacturing automotive parts. This also includes ensuring the low

cost of the entire new part development process, especially in terms of the processing and tooling cost, because high costs will not promote the use of recycled polymers. Therefore, the aim of this article is to design a compression molding tooling system for NFPC component manufacturing and identify key areas of improvement for future research work. Additionally, the crucial processing parameters required in the manufacturing of the component were also studied and documented. Finally, the part produced using the new tooling system was tested to determine its mechanical properties, and the challenges faced during the design phase were documented.

2. Materials and Design

The goal of the study is to develop a compression mold tooling system to manufacture an NFPC component. Therefore, for the purpose of the study, an automotive battery cover was taken into consideration and a compression molding tool was designed for the battery cover. Additionally, the processing parameters required for producing the component were identified and studied. This case study of an automotive battery car will help us to understand the key factors influencing the material, design, and process parameters when using NFPC material for the development of a new part. The methodology of the new part development is presented in Figure 1.

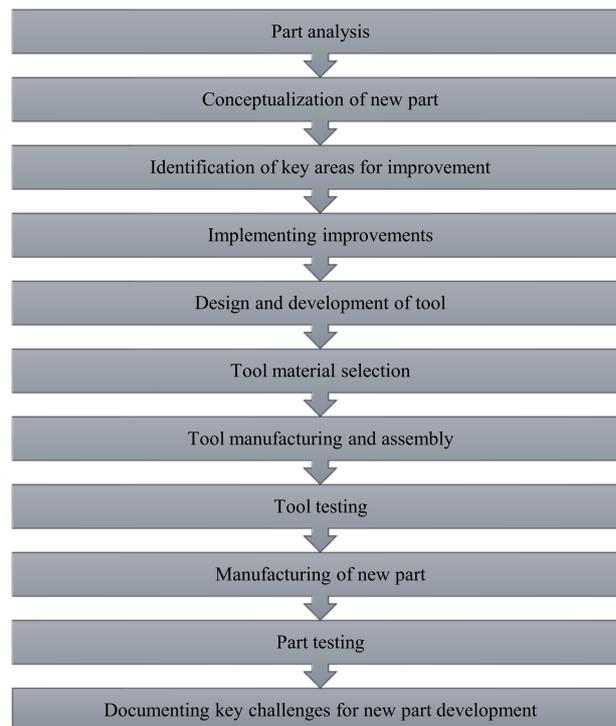


Figure 1. Methodology of the new part development.

Table 1 consists of all the key parameters that will be taken into consideration in developing the new part and tooling system for NFPC material. Previous studies related to the fabrication of NFPCs indicated that there were three crucial parameters in the compression

molding process; these include temperature, holding time, and pressure [4,38,39]. However, considering the experimental setup of the study, few additional process parameters were added. From the design aspect, basic tool parameters such as tool clearance, draft angle, and parameters related to the part design were taken into consideration. Finally, the material parameters mainly involved the fiber content, melt flow index, and additives.

Table 1. Key parameters involved in the development of the new part for the automotive application.

Material	Design	Process
Fiber %	Height of the part	Compression time
Polymer %	Radius of edges	Pressure
Density	Thickness of part	Mold temperature
Melt flow index	Tool clearance	Material temperature
Additives	Draft angle	Material heating time

2.1. Design of Automotive Part and Tooling

In order to design a new tooling system, it is important to analyze the construction and manufacturing process of an existing polymeric automotive component and then design a similar part for the same purpose as that of the existing part, but which will use recycled polymer composite material. Furthermore, a change in the manufacturing material will not only result in the adaptation of the design of the part but will also require an adaptation in the manufacturing process, since the material, design, and process are interlinked and any change in one aspect may affect the other two.

Therefore, for the purpose of this study an automotive battery cover, as shown in Figure 2, was selected as a testing specimen. An automotive battery cover was used as a lid on top of the car battery alongside the battery casing to act as an extra measure of protection from any mishap related to the battery, such as leakage or preventing a dead short. The design of the battery casing and cover is mostly generic and is suitable to most of the batteries available on the market. The original part was manufactured from virgin PP using an injection molding process. The original part consisted of many hotspot points (shown in Figure 2) from which the material was filled into the cavity of the mold. These hotspot points improve the flowability of the material in the mold but, at the same time, increase the overall tooling cost and make the tool more complex. However, fiber-based composite materials are difficult to manufacture with the use of an injection molding process because the flow of the material inside the chamber will be greatly reduced due to the presence of fibers, while fibers can also cause the blocking of the orifice of the machine at the output end. Secondly, the high cost of the injection molding tooling system does not compliment the purpose of using recycled plastics and low-cost natural fibers from a cost saving point of view. Furthermore, compression molding is a common manufacturing process used to produce NFPC automotive parts and is highly compatible in producing large shapes [11]. Therefore, keeping such issues in mind, a compression molding process was selected for use in this study.

Additionally, many previous studies related to natural fiber polymer composites have found that the compression molding is a viable means to produce NFPC parts for automotive applications. Liu et al. [28] compared the samples produced by compression molding and injection molding and found out that the samples produced by the compression molding process exhibited superior mechanical properties. Tungjitpornkull and Sombatsompop [40] also found similar results; they observed that the samples produced by the compression molding process exhibited higher tensile strength than those produced by a twin-screw extrusion process. Bledzki and Faruk [29] compared the mechanical properties of wood fiber–PP composites produced using compression and injection molding processes. They found that compression-molded samples exhibited a better impact strength, while injection molding samples showed better tensile and flexural strength. Finally, Zampaloni et al. [32] stated that the optimal manufacturing process for producing NFPCs is compression molding.



Figure 2. Existing hotspot points in the virgin polypropylene (PP) automotive battery cover.

However, compression molding also has some limitations when used to manufacture fiber-based composite materials. One main issue is the height of the part, which in the case of the original part was relatively high; however, it is difficult to obtain such a height in compression molding when using a fiber-based material because the melt flow index of fiber-based composite materials is quite low compared with that of virgin polymer, which can easily be melted and compressed at a high pressure to fill the entire cavity of the mold. Similarly, it is also difficult to keep the thickness of the material constant because of the low mobility of fiber-based composite materials in the mold. It is possible that the thickness could be increased if additional material is present in the mold. Therefore, it is important that only a fixed amount of material is put in the mold for compression. Additionally, it is impossible to achieve complex features, such as those marked by red circles in Figure 3, using compression molding, because such features will obstruct the path movement of male tools and can also cause an increase in tooling costs if complex tooling is required.



Figure 3. Complex features in the existing component.

Therefore, during the re-design of this battery part, it is important that the existing design issues due to the limitation of the manufacturing process and material properties

and behavior are taken into consideration in both the design and processing stages. Figure 4 shows a Solidworks representation of the battery cover design, considering all the existing issues. As shown in the figure, the height of the end of the cover was greatly reduced so that the composite material will flow smoothly up into the mold cavity. This is important from both a process and material point of view. If the height is too large, it is expected that the pressing machine will require a large amount of force to push the material up into the cavity of the mold, and it is also possible that a greater amount of plasticizer will be required in composite preparation to improve the mobility of the material. Additionally, the side features were also removed and both sides were made uniform; we believe that this will not impact the function of the battery cover, whose main purpose is to provide extra measure of protection to the battery. However, practical applications are yet to be tested. The bottom thickness was kept uniform at 4 mm for the entire component.

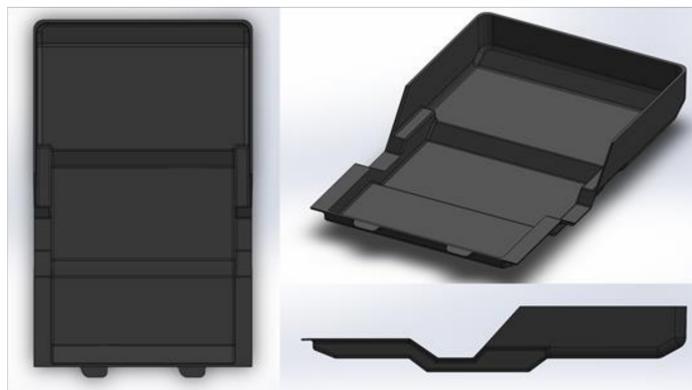


Figure 4. New automotive part design for the natural fiber polymer composite (NFPC) application.

As mentioned earlier, injection molding tools are generally complex and high cost, thus it does not make sense to have a costly tool, which would undermine the purpose of using recycled plastics and low-cost natural fibers. Therefore, it is important to keep the focus on developing a tool for the compression molding process that is simple and low cost. The mold (shown in Figure 5) for the newly designed part was produced at the Lappeenranta University of Technology (LUT) machining center using Toolox 34 engineering-grade steel. The mold design was a challenging aspect. The manufacturing of the mold required special long and small radius machine tooling because of the depth of the female mold. The long machining tool caused vibration during the machining process; therefore, a low feed rate was used, which resulted in longer cutting times. Furthermore, it was difficult to produce the inner radiuses of the female tool, especially for smaller dimensions. Some of angles of the designed parts were steep and not gradual.



Figure 5. Compressing molding tool manufacturing to produce natural fiber polymer composite (NFPC) components.

The cross-section view of the design compression molding tool in the assembly alongside the base plate by which it is connected to the compression molding machine is shown in Figure 6.

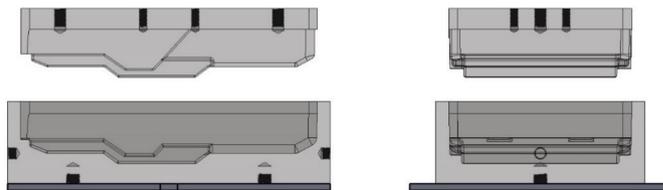


Figure 6. Representation of the compression molding tool in Solidworks.

2.2. Materials

The waste polymers, primarily consisting of ABS and other styrene-based polymers, such as ABS + polymethyl methacrylate (PMMA), ABS + polystyrene (PS), ABS + styrene acrylonitrile resin (SAN), ABS + acrylonitrile styrene acrylate (ASA), and ABS + acrylonitrile ethylene styrenes (AES), as shown in Figure 7, were supplied by Oili Jalonen Oy (Finland) and Majava Group Oy (Finland). These waste materials were extracted from automotive, motorbike, and truck polymer parts, such as motorbike frontal fairings, side fairings, tank covers, truck bumpers, car emergency triangle reflectors, and door panels. The waste automotive components were crushed into smaller granulates using a low-speed granulator

SHINI SG-1635N, as shown in Figure 7. The density and melt flow indexes of the recycled ABS mixture (R-ABSmix) were 1.032 g/cm³ and 14.981 g/10 min, respectively. The wood fiber was obtained from Stora Enso Oy (Finland) and palmyra (bassine fiber) was obtained from Mirja Dahl Ky (Finland). The wood and palmyra fiber size used for the composite preparation were 20 mesh (0.85 mm). The density of the wood fiber and palmyra fiber was 0.457 g/cm³ and 0.365 g/cm³, respectively and the moisture content of the wood and palmyra fibers was 12% and 10%. STRUKTOL[®] TPW 113, a blend of complex modified fatty acid esters, was used as a lubricant, with a density of 1.005 g/cm³ and a dropping point of 70–88 °C. EXACT elastomer was used as a plasticizer to improve the melt flow index of the composite material in the mold.



Figure 7. Illustration of the crushing process for the waste automotive components.

2.3. Agglomeration of Composites

The fiber material, R-ABSmix, lubricant, and plasticizer were compounded in a Plasmec COMBIMIX-RV/100/200/FV/W turbomixer with a cooler. Table 2 presents the composition of the prepared composite blends of R-ABSmix and natural fibers. Three different blends, including R-ABSmix, 10% wood fiber, and 10% palmyra fiber, were prepared for the experiment.

Table 2. Material composition of the recycled acrylonitrile-butadiene-styrene (R-ABS) and natural fiber composites.

Blend	Polymer	Fiber	Polymer (%)	Fiber (%)	Lubricant (%)	Plasticizer (%)
Blend 1	R-ABSmix	-	92	-	3	5
Blend 2	R-ABSmix	Wood	82	10	3	5
Blend 3	R-ABSmix	Palmyra	82	10	3	5

2.4. Compression Molding

All the blends were processed in a Stenhøj 400 kN compression molding machine. Six samples of each blend were produced for the experiment. Illustrations of the process setup and compression molding machine are shown in Figure 8. The process is a closed molding operation under high pressure. The blends were placed in the lower tool and spread evenly. Before the compression molding process, the blends were heated in a convection oven at 260 °C for 15 min and manually transferred to the compression molding tool. It is also important to note that even though the fiber degradation temperature was around 200 °C for most of the materials [38], it is important to ensure that the recycled polymer reaches its melting point. Therefore, considering the melting point of ABS and the possibilities of impurities in the recycled polymer mixture, a higher heating temperature was selected. However, the temperature of the composite material before compression was recorded using the Mastercool Infrared Thermometer OUTPUT < 1 mW AT 630–670 nm CLASS II and documented in Table 3. Additionally, both male and female tools were constantly

heated using a hot air blower. The compression time was maintained at 120 s to achieve a perfect profile for the battery cover. Furthermore, the bottom position of the male tool was set up to achieve a final thickness of 4 mm for the component. Due to this, the pressure applied by the compression molding machine was the outcome of the entire process.

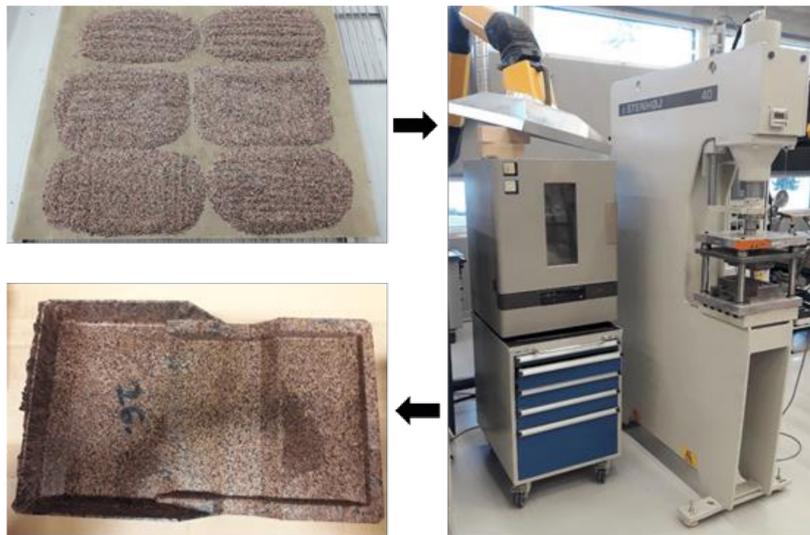


Figure 8. Compression molding setup and the production of the new automotive part.

Table 3. Processing parameters for the manufacturing of the new automotive parts.

Blend	Material Wt (gms)	Oven Temp (°C)	Material Temp (°C)	Heating Time (mins)	Female Tool Temp (°C)	Male Tool Temp (°C)	Compression Time (s)	Pressure (kN) *
Blend 1	400	260	205	15	71.5	62	120	392
Blend 2	400	260	204	15	68.5	60	120	392
Blend 3	400	260	202	15	70.5	63	120	395

* Pressure was measured as the outcome from the pressing machine.

The processing parameters used in the manufacturing of each composite component are described in Table 3.

2.5. Mechanical Testing

Since the primary function of an automotive battery cover is to act as protection for the top portion of a car battery, a basic compressive strength test was performed. The manufactured part from all three blends underwent compression testing to check the design and structural integrity of the part. The compression testing of the part was conducted in a universal testing machine (Zwick Roell Z202, Ulm, Germany) at a test speed of 50 mm/min with continuous loading. The experimental data were collected and processed automatically using the testXpert II software. Before testing, all the samples were cut into equal-height specimens, as shown in Figure 9.

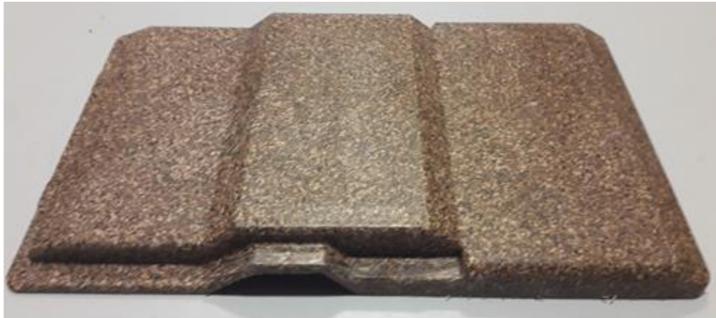


Figure 9. A specimen of wood fiber composite cut into shape for the compressive strength test.

3. Results and Discussion

3.1. Challenges in Design and Part Manufacturing

An initial design-related issue was the relatively small radius at the edges of the tool, which was kept at 3 mm during the tool design stage. The smaller radius made it difficult for the material in the mold to flow, and this was especially noticeable in the vertical sections of the mold. In some cases, the walls and the side horizontal surfaces of the final product did not come out of the mold filled. Usually, the melt flow index (MFI) of the polymer material decreases slightly after recycling [41], and it decreases further due to the presence of impurities in the recycled material. Moreover, adding fibers to the material to form the composite further decreases the flowability of the material in the mold. One solution to this issue is the better placement of material in the mold [42], as shown in Figure 10. It was observed that having an even distribution of the material in the mold results in the better flowability of the material compared to putting all the material in the center of the mold. Finally, to fully improve the flowability of both the polymer and composite material in the mold 5% *w/w* plasticizer was added to the material blends; this eased the flow of the material in the mold greatly. However, this led to a decrease in the overall mechanical properties of the material, because the addition of the plasticizer tends to make the material slightly softer. Finally, using a heavier pressing machine can also slightly improve the flowability, but, at the same time, this increases the cost of the machine drastically.

Another challenge observed with the designed tool involved the horizontal surfaces of the female tool, which, once filled with material and compressed by the male tool, acted as a counterforce to the force applied by the male tool. This primarily occurred due to the uneven levels of the horizontal surfaces. For instance, differences in height sometimes caused the material to be filled earlier at higher elevations and lock the tool in its place. This also affected the thickness of the material at the horizontal surfaces. If extra material is present in the mold, it can move towards these horizontal surfaces and accumulate there instead of coming out of the mold via runoff channels. The solution to such an issue is to have a multi-stage male tool, where the first part of the tool will come down slightly earlier than the main and lock all the horizontal surfaces at the desired height, apart from the main base of the female tool, where the working material is kept. An illustration of this is shown in Figure 11. However, such drastic and complex changes to the tool design which involve combining multiple tool pieces can significantly increase the tool cost. However, in our case, the focus was on developing a cheap and simple tooling system, and the results obtained from the designed tool were desirable for this study.



Figure 10. Evenly spread material placement, especially on the edges of the tool, provides optimal results.

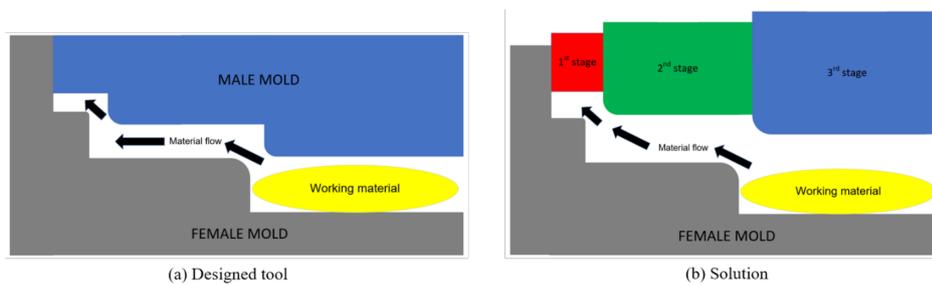


Figure 11. Illustration of (a) the designed tool for the production of the new automotive part and (b) the suggested change in the tooling system.

Then, it was found that the removal of the manufactured part from the tool was also difficult due to the depth of the female tool. This was more difficult when the part had just been pressed and was still hot, but it tended to come out easily once it had cooled. However, it can take several minutes for the material to cool down due to the high temperature of the material before putting it into the press with the heated tool. One solution we found was to add lubricant to the material blend, which greatly improved the removal process from the tool but, similarly to a plasticizer, had an adverse effect on the overall properties of the final product. Finally, another solution is to add an ejector pin to the base of the female tool as shown in Figure 12, which was proposed as a further modification for future testing [42].

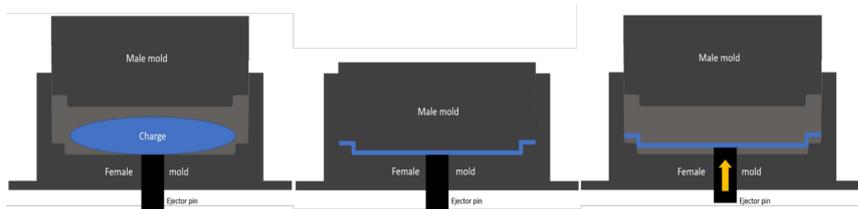


Figure 12. Proposed ejector pin for the new tooling system.

The next challenge was with the temperature difference between the heated material and the cold tool. Initial experiments were performed with the tools at room temperature; however, the results were not acceptable, since the manufactured product did not come out filled from the mold. Such a large and sudden temperature difference can affect the mechanical properties of the product and the flowability of the material in the mold. Finally, it was decided to manually maintain the mold temperature between 60 and 70 °C using a hot air blower. One easy solution for this problem is to add a heating coil to the female tool as shown in Figure 13 and use an induction process. This can make the tool more complex increase the tooling cost but can entirely replace the convection oven heating process. Syahirah et al. [43] designed and developed a heating system compression molding process and selected induction heating as the best heating method for the pressing process.



Figure 13. Proposed heating element for the new tooling system.

Numerous studies [4,33,39,44] have reported the preparation of NFPC specimens through the use of a compression molding process with an inbuilt heating system in the mold and analyzed the influence of the mold temperature on the mechanical properties. Yalaw et al. [4] studied the effects of mold temperature on the tensile properties of fiber-reinforced PP composites. They found out that at 175 °C temperature and 1 MPa pressure all three fiber-reinforced PP composite types exhibit the highest tensile strength value. Takemura and Minekage [33] used heating plates in both male and female tools and then compared the effect of varying molding temperature on the tensile properties of hemp reinforced composite and jute reinforced composites. Although most of these studies used a mold temperature between 165 and 195 °C, most focused on polypropylene (PP) or high-density polyethylene (HDPE), which have relatively lower melting points. Since ABS melts between 200 and 220 °C, it is important to use a mold temperature in that range. For the experiment, we found that heating with hot air blowers produced acceptable results, but for full-scale manufacturing a proper heating system is required.

The last challenge was related to the handling of the heated material, since all the handling was conducted by hand using protective gears. However, implementing an automated system with conveyors can ease the handling of the material, speed up the overall process, and avoid any unnecessary injuries to the operator. This is especially important in this case because the oven, material, and the mold were all relatively hot. Therefore, for the purposes of our test, the setup can be viable, but for large-scale production a conveyor system would be preferable.

3.2. Mechanical Testing

The result of the compression testing of 6 samples of blend 1 (R-ABS mix) is shown in Figure 14. The maximum load required for breaking the parts was found to be between 1400 and 2100 N, with an average load of 1693 N and an average deformation of 14.38 mm. The average elasticity value for all the samples of blend 1 was observed to be 1568.68 MPa.

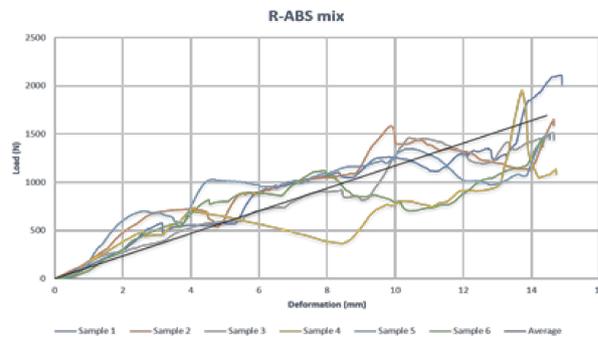


Figure 14. Compression testing results of the R-ABS mix blend.

The results of the compression testing of 5 samples (one sample was damaged during the handling phase) of blend 2 (10% wood) and 6 samples of blend 3 (10% palmyra) are shown in Figure 15a,b, respectively. For the wood fiber composite, the maximum load required for breaking the parts was found to be between 2350 and 3600 N, with an average load of 2690 N and an average deformation of 10.15 mm. The average elasticity value for all the samples of blend 2 was observed to be 3156.43 MPa. Meanwhile, for the palmyra fiber composite, the maximum load required for breaking the parts was found to be between 1450 and 2400 N, with an average maximum load of 1802 N and an average deformation of 12.97 mm. The average elasticity value for all the samples of blend 3 was observed to be 2032.95 MPa.

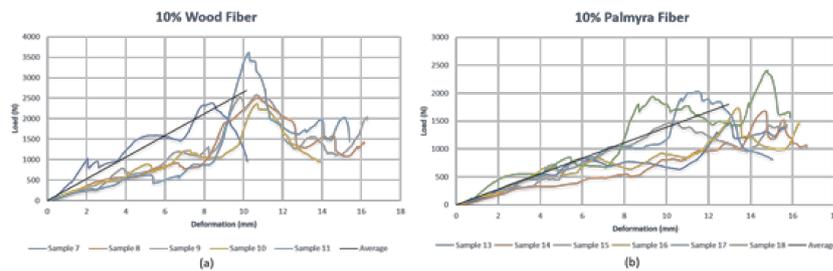


Figure 15. Compression testing results for (a) wood and (b) palmyra fiber composite blends.

It is evident that the addition of natural fibers had a positive effect on the compressive properties of the composite material. Numerous studies in the past have also observed similar results [27,30,31,45,46], where the addition of natural fibers to the polymer matrix increased the compressive strength of the composite. Kumar and Srivastava [45] observed that the addition of jute fibers to epoxy resin increases the compressive strength of the composite in comparison to the epoxy material. They attributed this increase to the interfacial bonding between the fibers and the polymer matrix. Similarly, Singha and Rana [27] found that composites reinforced with either 10% wt. raw agave fiber or MMA-grafted agave fibers exhibited high compressive strength values compared to virgin polystyrene. They also observed that irrespective of using short or long fibers, the compressive strength value was higher for the composite material. Also, Singha and Thakur [30] observed that the compressive strength of urea-formaldehyde resin increased when reinforced with natural fibers. They found that particle reinforcement exhibits better compressive strength values than short or long fiber reinforcement.

However, in the case of the palmyra fiber composite, the increase in compressive strength was less than the increase in the wood fiber compressive strength. It was assumed that the interaction between the palmyra fibers and polymer matrix was relatively weak compared to the interaction between the wood fibers and polymer matrix, indicating that wood fibers have more affinity for polymer interaction than palmyra fibers.

Finally, a statistical analysis was performed in Daniel's XL Toolbox Excel software, using the one-way analysis of variance (ANOVA) Bonferroni–Holm post hoc testing algorithm to verify the significance of the variations in the results. Table 4 displays the average values of elasticity, load, and deformation for all three blends. It was observed that the wood fiber composite exhibited significant changes, while the palmyra fiber composite exhibited non-significant changes in comparison with the R-ABSmix material for all the values.

Table 4. Compressive testing results of all three blends; ^s and ^{ns} denote statistically significant and not statistically significant changes at the 95% confidence level.

	Elastic Modulus (MPa)	Load (N)	Deformation (mm)
Blend 1	1568.68	1692.70	14.45
Blend 2	3156.43 ^s	2690.36 ^s	10.15 ^s
Blend 3	2032.95 ^{ns}	1802.66 ^{ns}	12.97 ^{ns}

4. Conclusions

This study focused on designing a tooling system for the use of NFPCs in automotive applications and identifying the crucial parameters involved in the design and processing of the part. For the purposes of the study, an automotive car battery cover was selected as a case study, because little focus has been given to NFPC applications in under-the-bonnet applications. The design of the new part and the tool has been explained in detail, considering various elements during the design and manufacturing phase when working with NFPC as a material. Additionally, all the challenges faced relating to the design, material, and processing of the part were also documented, which in turn can aid designers in implementing solutions for these challenges in their work.

The challenges faced during the study related to the design, material, and processing. They included the low melt flow index of the material due to the introduction of fibers, the small radiuses at the edges of the tool, the horizontal surfaces of the tool, the lack of an ejector pin at the bottom of the tool, and the lack of a heating element in the tool. However, it is also important to understand that implementing certain features to overcome some of these challenges will drastically increase the cost of the product. Therefore, due to the scale of this experiment, such features were not pursued but could be implemented during mass-scale production. The primary aim of this study was to evaluate the possibility of using NFPCs from recycled polymers in automotive applications and identifying key areas for improvement.

The compressive testing of the NFPCs and recycled ABS displayed that both wood and palmyra fiber composites possess a high compressive strength and exhibit low deformation values. Such results clearly indicate that the composite materials exhibit a higher compressive strength than recycled ABS due to their high elasticity/stiffness and the interfacial bonding between the fiber and matrix, which aids in stress transfer. This further increases the potential of NFPCs from recycled polymers to be used as construction materials in automotive part manufacturing. In future research, the focus should be on the cost and lifecycle analysis of the case study component and material to understand the economic and environmental aspects related to use of recycled materials.

Finally, it is important to understand that the purpose of this study was to explore the possibility of using recycled polymers from automobiles combined with natural fibers and provide a guideline on how such material can be utilized again in the automotive industry. We were able to produce an automotive battery cover with desirable properties using a simple tooling system and manufacturing process. However, we made some changes to the original design of the automotive battery cover in order to fulfil the goal of the study, which was to use recycled material to produce a low-cost and simple part. Hopefully, this will help future researchers to focus their attention on these materials and promote their usage in both experimental and practical applications.

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Publication IV

Chauhan, V., Kärki, T., and Varis, J.

**Optimization of Compression Molding Process Parameters for NFPC
Manufacturing Using Taguchi Design of Experiment and Moldflow Analysis**

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Article

Optimization of Compression Molding Process Parameters for NFPC Manufacturing Using Taguchi Design of Experiment and Moldflow Analysis

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Abstract: This paper presents the application of Taguchi design of experiment and Autodesk Moldflow[®] simulation in finding the optimal processing parameters for the manufacturing of natural fiber–polymer composite products. The material used in the study is a composite of recycled thermo-plastic reinforced with 10% wood fibers. For the study, four critical processing parameters, namely compression time, mold temperature, melt temperature, and pressure, were selected for optimization. Process analysis was carried out in Moldflow[®] utilizing a combination of process parameters based on an L9 orthogonal array. Later, the warpage output from Moldflow[®] simulation was converted into a signal-to-noise (S/N) ratio response, and the optimum values of each processing parameter were obtained using the smaller-the-better quality characteristic. The results show that the optimum values were 60 °C, 40 s, 210 °C, and 600 kN for the mold temperature, compression time, melt temperature, and pressure, respectively. Afterward, a confirmation test was performed to test the optimum parameters. Using analysis of variance (ANOVA), melt temperature was found to be the most significant processing parameter, followed by mold temperature, compression time, and pressure.

Keywords: compression molding; design of experiment; Moldflow; optimization; process parameters



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1. Introduction

The popularity of natural fiber–polymer composite (NFPC) as a primary construction material in various applications has been increasing rapidly. NFPCs are not limited to basic household and construction applications; they are rapidly gaining a market share in various other engineering fields, such as automotive, electronic, and aerospace applications [1]. The main reason for this growth is the demand for lightweight and robust components [2,3]. NFPCs are relatively lightweight due to the low density of natural fibers and offer good mechanical and dielectric properties. Additional benefits include their low cost, good specific strength, recyclability, carbon neutrality, biodegradability, and ease of disposal [4]. Furthermore, environmental concerns and strict government regulations are also a driving force in the shift towards more sustainable materials [3,5]. Therefore, incorporating NFPCs as a material not only enables manufacturers to save costs by using low-cost and easily available material but also increases their brand image as compared to their competitors.

The polymer matrices in NFPCs are divided into thermoplastics such as polypropylene (PP), acrylonitrile-butadiene-styrene (ABS), and polyamide (PA); and thermosetting, such as epoxy and polyester. The polymer material in the composites is usually virgin, however, recycled polymer material is also receiving increasing interest from researchers and manufacturers [6,7]. NFPCs are fabricated using various manufacturing techniques, including injection molding, compression molding, extrusion, and resin transfer molding [2,8,9]. Compression molding is widely used for the fabrication of NFPCs due to its high-volume productivity, simplicity, and low-cost operation [2,10]. The low cost of

operation of compression molding perfectly complements the low cost of NFPC materials, which can be further reduced when using recycled polymer materials. In comparison, injection molding, which is also a high-volume operation, requires complex and expensive tooling systems [10]. This is usually good for the processing of polymer material alone because with NFPCs there is always a possibility of fibers becoming stuck at the gate or runner due to their small size [9]. Meanwhile, compression molding requires the composite charge to be placed in between male and female tools, and the material is formed by the application of pressure and heat [8,10,11].

Previous studies have found that temperature, time, and pressure (compression force) are crucial parameters when processing NFPCs via compression molding [12,13]. From an industrial point of view, it is important to optimize these parameters in order to achieve the maximum efficiency from the production line while maintaining the desired quality and making sure all cost-saving measures are incorporated in the production of the final product. During optimization, it is vital to focus on reducing the processing time, since that will contribute directly to higher productivity and lower production costs. Meanwhile, it is also important to ensure that excessive reductions in processing time do not cause any defect risks in the final product [14].

Few studies have focused on optimizing the compression molding process parameters of NFPCs manufacturing [11,15–18]. Taguchi design of experiment (DOE) is the most commonly used method for optimizing the processing parameters of both compression molding [11,17] and injection molding [19,20]. Selmat et al. [11] determined the optimum compression molding parameters for obtaining the highest tensile strength from a pineapple leaf fiber–PP composite. They found that 30 kg/cm² pressure, 175 °C temperature, 6 min preheating, and 4 min compression duration yielded the desired output. Similarly, Shekeil et al. [17] optimized the processing temperature, time, speed, and fiber size using the Taguchi method to determine the highest tensile strength of a kenaf-reinforced polyurethane composite. The reported optimum parameters were 180 °C temperature, 50 rpm speed, 13 min time, and 125–300 µm fiber size. Ibrahim et al. [20] employed the Taguchi approach to find the best combination of processing parameters using orthogonal array (OA) and signal-to-noise (S/N) ratio.

At present, Autodesk Moldflow[®] software is also widely used in the optimization of injection or compression molding processes by simulating the flow, cooling, and warpage. It aids in resolving the challenges in injection and compression molding and aids in optimizing the part, mold, and process to reduce delays. Numerous studies related to process optimization using Moldflow[®] simulation combined with Taguchi DOE have been conducted; however, most of them are limited to the injection molding of polymer material and polymer composites [14,21–26]. These studies primarily focus on finding optimum injection molding processing parameters by reducing cycle time, warpage, or shrinkage. Even though there are not many studies related to the use of Moldflow[®] as an optimization tool for the compression molding of NFPCs, the software can simulate the compression molding process with fiber-based material, and the setup is relatively easier than that for injection molding.

In this study, an alternative approach based on a combination of Taguchi DOE and Autodesk Moldflow[®] simulation is implemented to optimize the compression molding process for the fabrication of NFPCs with the aim of minimizing warpage and achieving the desired product quality. The S/N ratio of the warpage value was selected as the objective function of the DOE. Furthermore, the significance of the processing parameters' influence on the warpage value was determined using analysis of variance (ANOVA). Therefore, with S/N ratio and ANOVA, the optimal combination of process parameters could be determined.

2. Material and Methods

2.1. Material Composition and Properties

The polymer material used in the study was waste polymer extracted from various automotive, motorbike, and truck parts. The waste polymer was mostly composed of ABS (80.77%) and other styrene-based polymer blends such as ABS–Polystyrene (3.59%), ABS–Styrene acrylonitrile (1.75%), ABS–Acrylonitrile styrene acrylate (4.61%), ABS–Acrylonitrile ethylene styrene (1.59%), and ABS–Polymethyl methacrylate (7.69%). The polymer waste was crushed in a granulator, and material properties testing was performed later in the laboratory. Material properties such as melt density, solid density, mechanical properties, and melt flow rate were calculated, and the values were added to the Moldflow[®] Insight material database. Thermal properties were taken from a generic ABS material present in the Moldflow[®] material database. The polymer data used in the simulation are shown in Table 1.

Table 1. ABS blend data table.

Description	Unit	Value
Melt density	Kg/m ³	960
Solid density	Kg/m ³	1050
Melt mass-flow rate	g/10 min	30.864
Transition temperature	°C	100
Elastic modulus	MPa	1780
Poisson ratio		0.39
Shear modulus	MPa	992.8
Specific heat	J/kg °C	2399
Thermal conductivity	W/m °C	0.18

The material properties for coniferous wood were already present in the database. Exact[®] elastomer and STRUKTOL[®] TPW 113 lubricant were added to improve the material flowability in the mold and ease of part removal from the mold, respectively. The properties of the elastomer and lubricant were available from the supplier and were also added to the database. The composite material composition is shown in Table 2.

Table 2. Simulation material composition.

Polymer (%)	Fiber (%)	Lubricant (%)	Plasticizer (%)
82	10	3	5

2.2. Test Model

In this work, an automotive car battery cover was used as an example for the optimization process. The battery cover was designed in-house as a replacement for the polymer-based battery cover for an existing automobile. The designed automotive cover is suitable for NFPC material consisting of recycled plastics and is a viable replacement for its virgin counterpart. The modeling of the battery cover was done in Solidworks 2017 and then it was imported to Autodesk Moldflow[®] Insight 2021 for meshing and analysis. The modeled and meshed part is shown in Figure 1. The dimensions are 340 mm × 170 mm. The thickness of the part varies from 2.5 to 4 mm. For meshing, Moldflow[®] has three mesh types, including Midplane, 3D, and Dual Domain. Due to the complex shape of the test model and its varying thickness, we selected 3D meshing, which in turn led to a greater meshing duration and in the end generated 1,179,779 mesh elements for the analysis.



Figure 1. Modeled and meshed part used in the simulation.

2.3. Taguchi Design of Experiment

The Taguchi approach has been widely used in engineering analysis to optimize the performance characteristics within the combination of design parameters [27]. The Taguchi approach employs three stages: system design, parameter design, and tolerance design. The main objective of Taguchi parameter design is to establish the optimal combination of design parameters and reduce variations in the product quality [27,28]. DOE is a scientific method used for multi-factor experiments, where some representative samples with uniform characteristics from a comprehensive test are selected to conduct experiments by means of probability, mathematical statistics, and orthogonal principle [21,24,26]. Taguchi DOE helps to study the effect of several variables on the desired quality characteristic and analyzes the effect of the degree of variables on the quality characteristics; using this approach, the best combination of variables can be determined [29]. Taguchi DOE minimizes the number of experiments for the optimization process and thus saves both cost and time [11,28,30]. In this study, the melt temperature, mold temperature, compression time, and pressure were selected as the process parameters (factors) for DOE analysis. The factors and their levels are given in Table 3.

Table 3. Level of factors affecting the compression molding process.

Symbol	Factor	Unit	Values		
			1	2	3
A	Mold temperature	°C	60	70	80
B	Compression time	s	30	40	50
C	Melt temperature	°C	210	220	230
D	Pressure	kN	400	500	600

Taguchi DOE employs a specially constructed OA, which helps to decrease the required number of experimental trials to study the entire parameter space [27,29]. The OA table is used as a tool to arrange the experiments reasonably and conduct an analysis of the results. Taguchi DOE with a L9 (3^4) orthogonal array was used in this study, consisting of four factors with three levels (Table 4).

Table 4. The LP orthogonal array (3^4).

Exp. No.	A	B	C	D
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

The warpage experimental results were transformed into S/N ratios. Taguchi employs the S/N ratio as one of the measurement indexes to measure the deviation in the quality characteristics from the desired value. The S/N ratio characteristics are further divided into three categories: the larger-the-better, nominal-the-better, and smaller-the-better [27–29,31]. Since the purpose of this study is to minimize warpage by optimizing the process parameters, the smaller-the-better quality characteristic was selected. The S/N ratio for each level of process parameters was analyzed based on S/N analysis, where higher S/N ratios correspond to better quality characteristics [29]. For the smaller-the-better quality characteristics,

$$S/N = -\log(\text{MSD}), \quad (1)$$

where MSD is the mean-square deviation for the output quality characteristic. For smaller-the-better quality characteristics, MSD can be expressed as:

$$\text{MSD} = \frac{1}{N} (\sum_{i=1}^n Y_i^2), \quad (2)$$

where Y_i is the value of warpage for the i th test, n is the number of tests, and N is the total number of data points.

ANOVA has been used to determine which parameters significantly affect quality characteristics [27,29]. Therefore, ANOVA was used in this study to evaluate the effect of the process parameters on warpage. The percentage contribution of the given process parameter was calculated using the following equations:

$$\text{SST} = \text{SSB} + \text{SSW}, \quad (3)$$

where SST is the total sum of squares, SSB is the sum of squares between the groups, and SSW is the sum of squares within the groups.

$$\text{SST} = \sum_{i=1}^n (x_i - \bar{x})^2, \quad (4)$$

where n is the number of experiments in the orthogonal array, x is the S/N ratio for the i th experiment, and \bar{x} is the mean S/N ratio. The percentage of contribution P can be calculated as follows:

$$P = \frac{\text{SSB}}{\text{SST}} \times 100\% \quad (5)$$

3. Results

3.1. Taguchi DOE

The results from the Taguchi DOE for the warpage response and product quality are shown in Table 5. According to Moldflow®, “good quality” means that the final product produced using the given combination of process parameters meets the production standards in terms of material properties and appearance, while “bad quality” means that

the final product quality was not sound due to a short shot. A “short shot” is defined as an incomplete filling of the mold, resulting in the production of an incomplete product.

Table 5. Quality and warpage results of the simulation.

Exp. No.	A	B	C	D	Results	
					Quality	Warpage (mm)
1	1	1	1	1	Bad	1.506
2	1	2	2	2	Good	1.496
3	1	3	3	3	Good	1.521
4	2	1	2	3	Good	1.512
5	2	2	3	1	Good	1.530
6	2	3	1	2	Good	1.517
7	3	1	3	2	Good	1.575
8	3	2	1	3	Good	1.502
9	3	3	2	1	Good	1.526

3.2. S/N Ratio

As stated previously, the experimental results for the warpage response were transformed into S/N ratios. The warpage responses and the corresponding S/N ratios are shown in Table 6. The smaller-the-better quality characteristic was used to obtain the S/N ratios.

Table 6. Warpage results and corresponding S/N ratios.

Exp. No.	A	B	C	D	Warpage (mm)	S/N
1	1	1	1	1	1.506	−3.556
2	1	2	2	2	1.496	−3.499
3	1	3	3	3	1.521	−3.643
4	2	1	2	3	1.512	−3.591
5	2	2	3	1	1.530	−3.694
6	2	3	1	2	1.517	−3.620
7	3	1	3	2	1.575	−3.946
8	3	2	1	3	1.502	−3.533
9	3	3	2	1	1.526	−3.671

The significance of each process parameter’s influence on the quality characteristic (warpage) was evaluated according to the maximum and minimum S/N ratio response of each parameter at different levels. The average responses were determined from the S/N ratios in Table 6 and are shown in Table 7. An example of the average response calculation is shown below, for the mold temperature:

$$\text{Level 1} = ((-3.556) + (-3.499) + (-3.643))/3 = -3.566$$

$$\text{Level 2} = ((-3.591) + (-3.694) + (-3.620))/3 = -3.635$$

$$\text{Level 3} = ((-3.946) + (-3.533) + (-3.671))/3 = -3.717$$

Table 7. Average responses of S/N ratio and delta.

Level	1	2	3	Delta	Rank
1	−3.566	−3.635	−3.717	0.151	2
2	−3.698	−3.575	−3.644	0.122	3
3	−3.570	−3.587	−3.761	0.191	1
4	−3.640	−3.688	−3.589	0.099	4

The delta value for any process parameter is calculated by subtracting the lowest value from the highest value. Based on the delta value, from highest to lowest, a significance ranking was given to each parameter. Rank 1 indicates the most significant parameter while rank 4 indicates the least significant parameter.

Figure 2 shows the S/N ratio response diagram for warpage. The optimum parameter settings to minimize warpage can be easily identified based on the response diagram. The highest value from each parameter represents the best level. Based on Figure 2, the optimum process parameters were obtained by a combination of A1, B2, C1, and D3, as shown in Table 8. Thus, the optimum process parameters are mold temperature 60 °C, compression time 40 s, melt temperature 210 °C, and pressure 600 kN.

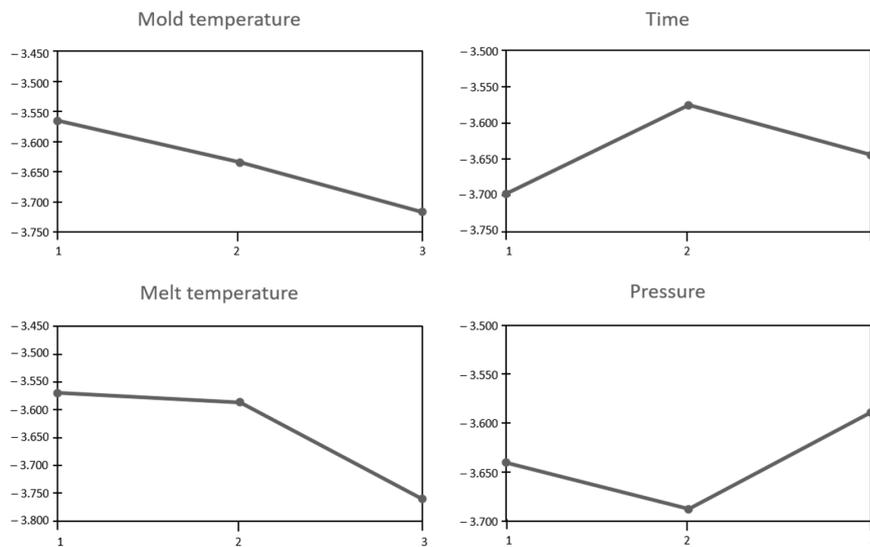


Figure 2. S/N ratio response graph.

Table 8. Average responses of S/N ratio and delta.

Parameter	Value
Mold temperature (A1)	60 °C
Compression time (B2)	40 s
Melt temperature (C1)	210 °C
Pressure (D3)	600 kN

3.3. Confirmation Test

Based on the optimum parameters obtained from the Taguchi DOE and S/N ratio, a confirmation test simulation was run to check the product quality, fill time, and warpage for the optimum parameters. Fill, pack, and warp analyses were performed in Moldflow[®] for the optimum parameters. Figure 3 shows the fill time and warpage values of the component produced using optimum parameters.

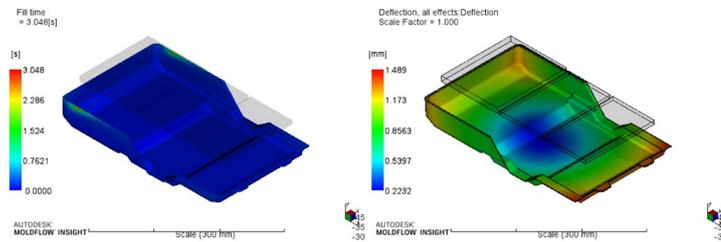


Figure 3. Result of fill time and warpage value for optimal process parameters.

3.4. ANOVA

ANOVA was used here to evaluate the percentage significance of the process parameters on the warpage value. ANOVA results include the sum of squares (SS), degrees of freedom (DOFs), variance (V), F-ratio, and the percentage contribution of each parameter (P%). They are summarized in Table 9. The F-ratio and P% are critical for interpretation. If the F-ratio is large, then the p -value is small, which means that the results are statistically significant. A high value of P% indicates a highly influential parameter. From Table 9, it can be observed that melt temperature (C) had the highest contribution among all factors, with a 48.12% contribution to the warpage value. This was followed by mold temperature (A, 24.73%), compression time (B, 16.33%), and pressure (D, 10.82%) as the factors influencing warpage. Similar observations were found by Hussain et al. [31] in their work optimizing the injection molding parameters by minimizing warpage using the Taguchi method and process simulation software. They also found that the melt temperature had the most significant impact on the warpage value.

Table 9. ANOVA results for warpage.

Parameter	DOF	SS	V	F-Ratio	P%
A	2	0.00107	0.000535	0.985472	24.73
B	2	0.00071	0.000353	0.585604	16.33
C	2	0.00208	0.001041	2.782957	48.12
D	2	0.00047	0.000234	0.363903	10.82

4. Conclusions

This study was focused on optimizing the compression molding processing parameters for the manufacture of an NFPC part by utilizing Taguchi DOE and Moldflow[®] simulation. In finding the optimal parameters using an orthogonal L9 array, the S/N ratio and ANOVA were implemented in an integrated manner. Moldflow[®] simulation generated the warpage values for the set of parameters described in the L9 orthogonal array. Then, the S/N ratio response for each parameter was generated from the corresponding warpage values. Based on the S/N ratio response, the optimum process parameters were found to be a mold temperature of 60 °C, a compression time of 40 s, a melt temperature of 210 °C, and a pressure of 600 kN. Using the optimal parameters, a confirmation test was performed in Moldflow[®], and the resulting warpage and fill time were obtained for the composite part. Then, the influence of each processing parameter on the warpage value was calculated using ANOVA. Based on the ANOVA results, it was observed that the melt temperature was the most influential parameter, with a percent contribution of 48.12%; this was followed by the mold temperature (24.73%), compression time (16.33%), and finally pressure (10.82%).

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