Chapter 6
Critical Concerns on Manufacturing Processes of Natural Fibre Reinforced Polymer Composites

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Abstract For the last one decade, natural fibre reinforced polymer composites have attracted a considerable attention around the globe due to their inherent performances such as biodegradability, recyclability and accessibility. A suitable manufacturing process is required to develop a promising reinforced polymer composite accordingly. It has been observed that the matrices, materials and chemical, physical, mechanical and thermal properties of both the hosting polymer and the employed natural fibres play the most important role in choosing an appropriate processing technique. Recently, valuable studies have been carried out in this field, as engineers have tried different processing methods and conditions. In this chapter and later a brief introduction to biomass materials, a comprehensive review has been done over the latest biocomposite processing techniques. Beneficial solutions and treatment methods have been addressed. Furthermore, a combination of both traditional and modern processing technique has been recommended for future studies.

Keywords Natural fibre • Biocomposites • Manufacturing • Mechanical properties

6.1 Introduction

Since the last two decades, manufacturers have been progressively looking for higher strength, lower weight, cheaper and more eco-friendly material to produce novel products in order to create entirely new products by taking advantage of these characteristics. Natural fibres as an alternative reinforcement agent in biocomposites present numerous advantages over conventional glass and carbon

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fibres, low cost, comparable specific tensile properties, low density, nonabrasive to the equipment, nonirritating to the skin, reduced energy intake, fewer health risk, renewability, recyclability and biodegradability.

Since the 1980s, natural fibres are experiencing increased demand as fillers in polymer matrices. The application of natural materials in polymer composites in a broad range of production lines has resulted in decreasing the production of carbon-based products and the employment of petrochemical products. Meanwhile, following the fibre complex structure, various mechanical performances have been observed through the application of the same fibre in different hosting matrices. The product strength can be affected and reduced due to a number of issues, wettability of the natural fibre, presence of degradation at the fibre/matrix interface (following the fibre attractive or repulsive response to water) and fibre destruction during the manufacturing process. A pretreatment process of fibre can prevent this reduction in the product strength and moreover enhance the processing procedure. In this chapter, following a brief introduction to agricultural residues (as a rich source for natural fibres) and the suitability of known manufacturing processes, based on the fibres and matrices materials, mechanical and thermal properties are discussed in detail.

6.2 Biomass Materials

At present, agricultural residue materials are supplying 14 % of the world's energy, while yet a huge amount of bypassed biomass has no particular application (Saxena et al. 2009). Generally this large quantity of biomass is burnt or dumped, producing undesirable contaminants in the air, SO_X and NO_X. Studies have shown that this carbon renewable resource can be transformed to solid, liquid and gaseous product over numerous treatment processes. Natural fibres are derived from lignocellulose materials, and therefore they are hydrophilic. From Fig. 6.1, lignocellulosic biomass contains several important noteworthy natural polymers, cellulose $[C_6H_{10}O_5]_n$ (35–83 % dry weight basis), hemicelluloses $[C_5H_8O_4]_m$ (0–30 % dry weight basis) and lignin $[C_9H_{10}O_3 \cdot (OCH_3)_{0.9-1.7}]_x$ (1–43 % dry weight basis) (Balat et al. 2008).

6.3 Manufacturing Techniques and Concerns

6.3.1 Manufacturing Principles

Appropriate manufacturing processes are essential to be employed to convert the raw materials to the desired product. For manufacturers, ideal application, performance, size, structure, processing characteristic, speed of production and costs of processing are the major topics to discuss about while designing a production line. In the meantime for initial scheming, through the mentioned principles, the composite finishing size is highlighted and a leading feature. Injection moulding and compression moulding are simple and swift processing techniques that are both

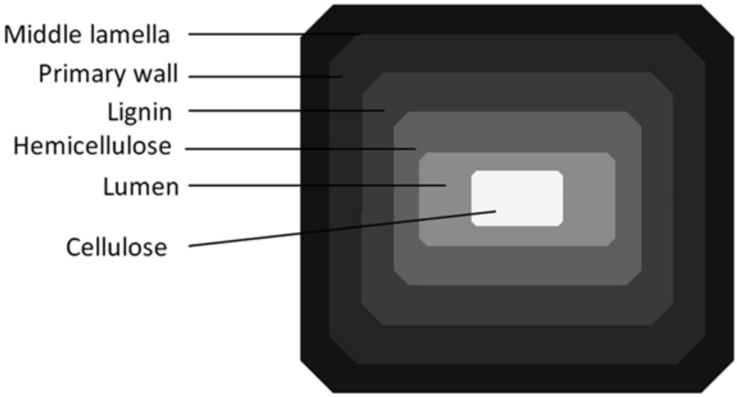

Fig. 6.1 Schematic diagram of lignocellulosic fibre's physical structure

endorsed for small/medium-sized feeding materials. Generally, for a simple form of composite product, compression moulding is recommended. Open moulding and autoclave process are addressed for bulk productions correspondingly. Furthermore, the product shape complexity has a direct impact on the processing technique to be used likewise. Recently a comprehensive review on the effect of different processing conditions and different surface modification techniques on the cellulose fibres and their composites has been carried out (Thakur and Thakur 2014).

For short fibre and nanomaterial-based processing, injection moulding and compression moulding are employed, while pultrusion is mainly used to produce stretched uniform products (Memon and Nakai 2013a, b). Pultrusion is a continuous composite processing method that combines reinforcement impregnation with composite consolidation. In this method, individual prepreg layers are flattened out and a bond is formed between them. The die structure is carefully chosen according to the desired product shape. Continuous controlled steps of heating are required to cure the composite product. Following an appropriate fabrication technique, certain properties of the raw materials require further attention, thermoplastics/thermosets, low/high viscosity and manufacturing temperature. The elementary principle of pultrusion process is the continuous melt impregnation of fibre ropes (roving) in a pultrusion tool (shown in Fig. 6.2). In terms of reducing the energy consumption, the location of the implanted heaters can be more influential than the number or the power of the heaters (Silva et al. 2014). In addition to stretched uniform products, studies have employed pultrusion process to manufacture phenolic foam composites (Yun and Lee 2008).

As mentioned earlier, the aspects in selecting an appropriate processing technique for reinforced polymer composites are totally different with the neat commercial polymers. The performance of a reinforced composite is significantly reliant on the alignment, length, content and diameter of the employed fibre. Additionally, the interface between the fibre and the matrix can be directly influenced by the natural fibre surface properties. To enhance this interface, mercerization is an eminent treatment

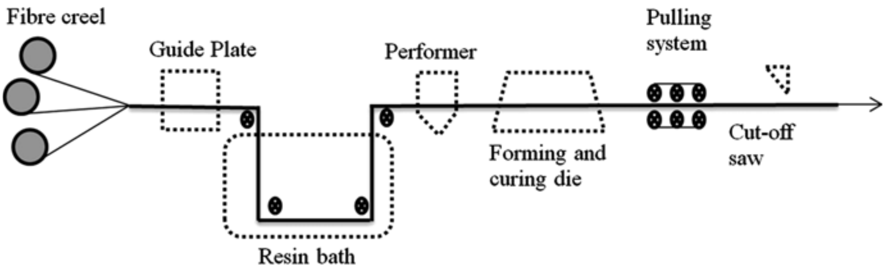

Fig. 6.2 Schematic diagram of pultrusion process

process suitable for surface treatment of fibres. Fibre surface coating removal or pretreatment by using chemical treatment is required to ensure good bonding properties. Fibre treatment enhances the matrix–fibre interface; thus it allows the stress to transfer from the matrix to the fibre which holds the higher flexural modulus of the composites. A heterogeneous system is assembled during the introduction of natural fibres to polymer composites following a weak adhesion at the matrix–fibre border (Matuana et al. 2001; Keener et al. 2004).

Natural fibre pretreatment can play an important role in enhancing the adhesion properties. Chemical and mechanical modifications are employed to increase the fibre resistance to moisture adsorption as well as the surface roughness. Several well-known fibre mechanical and chemical treatment processes are as follows:

- Grinding of fibres
- Mercerization of fibres
- Acetylation treatment
- Electron beam irradiation modification of fibre. (*This method is known economical for mass-produced frameworks*)
- Silanization of fibres
- Steam explosion of fibres
- Graft copolymerization of a monomer/polymer onto fibres. (*This method can be introduced to the applications where degradability of the hosting polymer composite is in priority*)

Following the reviewed studies on various natural fibre treatment processes, fibres with a particle size less than 500 μm are recommended for reinforcing purposes. Moreover, since 2012, to enhance the performance of the polymer composites, while using smaller amount of material at the same time, studies have shifted from using micron-sized particle to nanosized particle. A temperature of about 80–100 °C is initiated to be suitable for drying the fibres. Sodium hydroxide is known to be the most compatible and efficient chemical for an alkali treatment.

In addition to alkali treatment (mercerization), silanization of fibres is also considered to be an appropriate treatment for interface modification; notable improvements in specific tensile and flexural strength properties of fibres are reported following this type of treatment. Also, acetylation is found to bring changes in fibre surface morphology and results in the surface to become much smoother. Acetylation in vapour phase is reported to decrease the hydrophilic properties of the fibres and enhance the thermal stability.

6.3.2 Moulding Processes

6.3.2.1 Injection Moulding Process

For thermoplastic-based processing, injection moulding and compression moulding processes are recommended. Rheological measurements (shear viscosity measurements and extensional viscosity) have been carried for injection moulding machine (Kelly et al. 2009; Bariani et al. 2007) and extruder (Vera-Sorroche et al. 2014). During the injection, as an advantage of using an injection moulding machine, following the screw speed, the volumetric flow rate can be calculated accordingly. Through this determination, minor leakage flows from the valve are negligible, and the speed of the screw is considered constant through the process. The installed screw inside the electric injection moulding machine is empowered and accurately controlled using a servomotor (shown in Fig. 6.3). The precision of rheological measurements is based on the accuracy of this servomotor.

During the injection moulding process, a molten blend of both polymer and fibre is forced into the voids of the mould. Various researches have been carried out to determine the opportunities of reinforcing polymer composites through injection moulding (Serizawa et al. 2006; Huda et al. 2005, 2006a, b). Generally, plastic pellets of the neat thermoplastic polymer are employed for this particular type of processing. At the feeding stage, both plastic pellets and ground fibres are fed, respectively, through a funnel into a preheated compression barrel with installed screw/screws shown in Fig. 6.3. The heat of the barrel melts the plastic pellets and forms them into a viscous liquid. The phase transition of plastic pellets enhances the

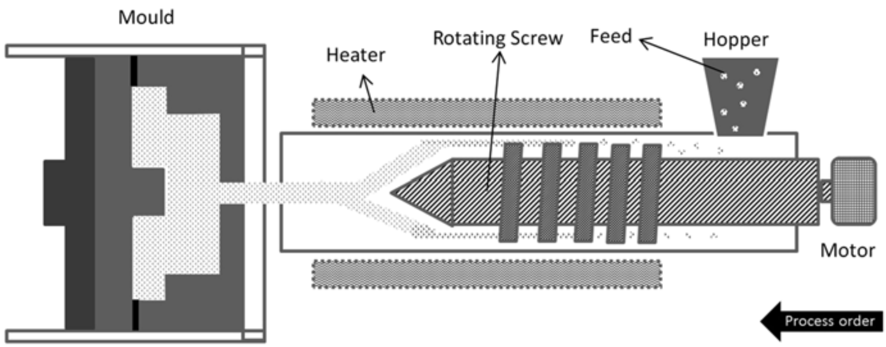

Fig. 6.3 Schematic diagram of an injection moulding machine

injection performance to the designed moulds and later to the sprue nozzle. The heat is applied to melt the plastic pellets fed in between the barrel and screw. Also, the purpose of heating the barrel is to transform the solid pellets into viscous liquid which can be drove through the screw and sprue nozzle and lastly forced into the matched-metal closed mould cavities. Pressurizing is required to drive the blend through sprue nozzle. Following the changes in the process pressure, the moulds are required to be clamped securely. After a proper cooling and solidification stage, the shaped reinforced composite is collected from the moulds.

The screws are employed due to several purposes and benefits:

– To change the phase of the plastic pellets into viscous liquid
– To generate a shear force to enhance the blending and dispersion properties
– To drive the fibre-melted plastic pellet blend through the barrel, the sprue nozzle and into the moulds

For biodegradable polymers such as poly(lactic acid) (PLA), an easier and smoother flow of the polymer is observed following a drop in the shear viscosity which is obtained following an increase in process temperature. Further to an increase in process temperature, an increase in shear rate can decrease the molten polymer viscosity significantly. Generally, this drop in polymer viscosity is caused by following a break in molecule chains influenced by an increase in temperature or shear force.

For a reinforced polymer composite, in the presence of force, the stress is expected to transfer from the polymer matrix to the natural fibre. Furthermore, during the reinforcement, it is desired to load the fibre to its full capacity while having a strong interfacial bonding. One of the common techniques used to reach these valuable goals is chopping the fibre short accordingly. Further studies on fibre size are required while employing the common injection moulding process; in this process, following the great shear rates in the barrel as well as the sprue nuzzle, the application of too small fibres causes notable attrition. Hence, in practice, a strong attrition can result in a shorter fibre length than expected. As a consequence, the fibres in the product are shorter than the critical length; this limits the fibres to carry their determined load effectively and efficiently.

Meanwhile, the fibre can relatively act as a defect in the polymer matrix due to its length and surface properties. It needs to be noted that the fibre could fracture prior to the failure of the matrix formation in case the fibre length drives beyond the expected critical length. Hence, determination of fibre critical length prior to injection moulding is essential. Commonly, the strength as well as the toughness of the finishing product improves following an increase in fibre content. However, this advantage is limited while using injection moulding machine, due to a slim feeding gate, fibre filling, and blend viscosity. Also, there are several concerns that influence the modulus distribution, fibre volume extension and later to the mixing stage with the molten polymer, residual stress and also orientation of the fibres with respect to the depth.

Fast cooling process of the molten composite apart from any external force results in an internal stress called residual stress. The tensile stresses at the surface and the core areas are expressed by the residual stress distribution, and compressive stresses at the intermediate areas are known as the characteristic residual stress distribution in injection-moulded parts (Kim et al. 2009). Since the 1990s, three levels of stress in laminated structure have been recognized: the 'micro-stresses' present among fibres within each ply, the 'macro-stresses' developing in multiaxial laminates at the ply-to-ply scale and a third more dominant level of stress resulting from different thermal histories of different parts of a laminate during the cooling sage (Shokrieh 2014).

Residual stress causes an early fracture in the composites which directly affects the quality of the finishing product (a product based either on a neat thermoplastic polymer or fibre reinforced composites).

The physical and mechanical pressures that the molten polymer goes through from the beginning of the injection moulding process until the end (filling up the mould cavity) have a direct influence on the residual stress distribution along the flow path (White 1985). The residual stress results in shrinkage of the final product. Hence it can be said that the dimensional accuracy of the finishing product is strongly linked to the residual stress distribution in the moulded part.

Furthermore, due to the high process temperature, change in moisture absorption characteristics, in addition to undesired layers and internal void formations, could take place. Therefore studies on residual stress can play a critical role in parameter selection. To avoid any progress of residual stress causing warpage, stress cracking or ageing, optimizations on several parameters can play an important role, processing technique, employed material and linear parameters. In formation of the residual stress, the linear parameters (such as the mould cavity shape and size, the locations of injection gates and the vents that allow the air to escape) play the major role (Batra et al. 2012). This prevention involves controlling the temperature of the molten blend as well as the mould and also the speed of the screw and injection during the process. A notable decrease in stress level and later to a compressive shift of stress onto the surface can be obtained following an increase in mould temperature (Carpenter et al. 2014; Kim et al. 2007). The processing properties and the finished product performance are both influenced by the rheology of the molten polymer as well as the fibre source and content (Park et al. 2013; Garancher and Fernyhough 2014).

Natural fibres are only subjected to a low thermal stress (Nechwatal et al. 2003). The matrix orientation is directly linked to the fibre content adjustments (Folkes and Russell 1980). Following the heat generated during the injection moulding, fibres are therefore oriented. During the solidification of the matrix, the orientation of the fibres is fixed. At low flow rates, the rotating screws affect the parallel alignment of the flow direction. Fibre alignment follows the shearing as well as the injection flow stretching direction; this takes place near the walls of the mould called the skin shown in Fig. 6.4. Beneath the top layer, the liquid viscose mixture remains undergoing the applied shear, and during this time, fibres align along the shear directions accordingly. After the formation of the top layer (skin), the core layer is formed as the fibres are swayed by the bulk deformation of the flow in the mould. The flow structure shown in Fig. 6.4 presents a general 'microstructural' observation.

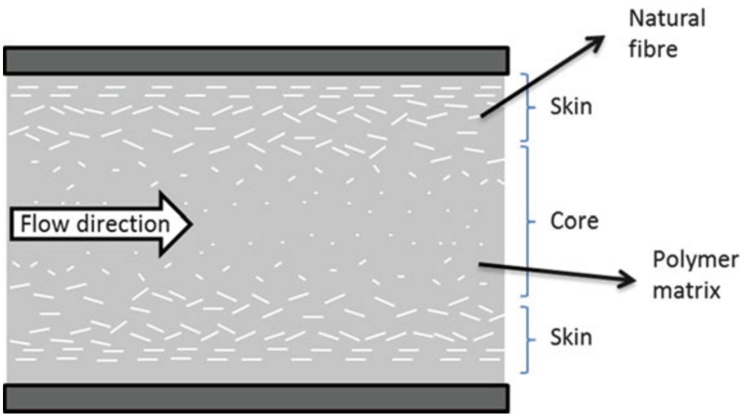

Fig. 6.4 Influence of flow on fibre orientation during injection moulding

The technique to observe the skin–core arrangement in lab-scale preparations with low fibre content is challenging. During 2001, dumbbell-shaped composites have been suggested to enhance the observation (Lee et al. 2001). Recently, studies have suggested the application of computational tomography (CT scan) to identify the amount, dispersion and orientation of components in the hosting matrix (Pujadas et al. 2014).

6.3.2.2 Compression Moulding Process

This process is a high-pressure, high-volume, plastic moulding technique that is appropriate for moulding high-strength products. Many production lines have chosen compression moulding to produce parts due to many advantages of this process, low cost, short cycle time, high-volume production, dimensional accuracy, improved impact strength, uniform shrinkage due to uniform flow and uniform density. To reinforce polymer composites with natural fibres, the application of compression moulding (a mixture of autoclaving and hot-pressing process) has been studied by researchers (Serizawa et al. 2006; Memon and Nakai 2013a, b; Dhakal et al. 2013).

It is mainly followed by two steps: preheating and pressurizing. As shown in Fig. 6.5, the composite is placed in a cavity of the matched mould, and after closing the mould (by bringing the two halves together), pressure and heat are applied in order to squeeze the composite filling the mould cavity. The mould is opened and the part is ejected subsequently. Generally this process operates at a pressure in a range from 14 to 20 MPa and at a temperature of 150–190 °C. There are two traditional types of compression moulding: 'sheet moulding compound' (SMC) and 'bulk moulding compound' (BMC). Composite density, strength and fibre orientation are the material properties affected by the process.

Applying extreme pressure may result in the fracture of fibres. To minimize the presence of fracture and damage on fibres, the fibres are recommended to be

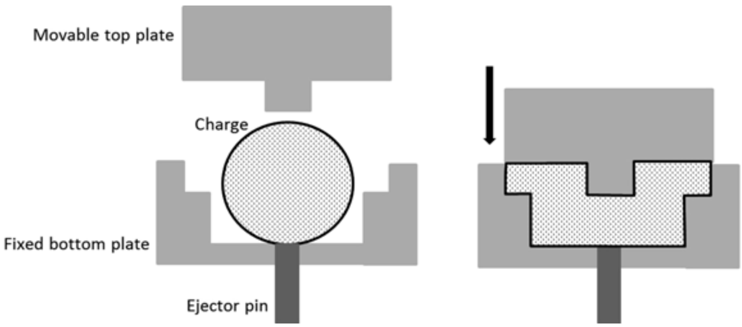

Fig. 6.5 Schematic diagram of compression moulding process

smoothly located inside the mould divest of any shear stress. Additionally, a premix of the natural fibre with the polymer compound is advised. Long fibres could be employed to produce biocomposites with higher volume fracture accordingly.

6.3.2.3 Hot-Pressing Moulding Process

Previously, it was understood that hot-pressing process is a part of compression moulding process. During the hot-pressing stage, two plates are required for compressing and heating the fibre–polymer matrix subsequently. Through this process, the main challenge is to control the matrix viscosity, mainly for thin samples. Minor faults such as warpage, minor voids, fibre breakage, sink marks and residual stresses are able to decrease the mechanical properties notability.

The viscosity level needs to be optimal enough to allow the polymer matrix to fill in the space between fibres, at the same time to maintain the matrix inside the mould, and generally to minimize possible defects to appear. Optimization of viscosity is essential to push out the trapped air in the mould and charge. Formation of voids which cause undesirable effects on the flexural and tensile properties emerges (Chambers et al. 2006; Hagstrand et al. 2005). More recently studies have been able to study the void morphology with 3D μCT images (Lambert et al. 2012). Following the filament structure of fibres, in order to produce quality composites, the wetting procedure requires additional attention (Yan et al. 2014). To enhance the wetting procedure, process parameters need to be controlled adequately, the initial charge shape, process viscosity, temperature, holding time and the composite thickness. The control of process parameters such as temperature becomes much more essential as natural polymers become the hosting matrix (Zhang et al. 2014; Shabanian et al. 2013). Through a study on the influence of various parameters on the mould heating rate, at a mould plate area of 60 mm by 60 mm, it is found that a change in mould material from stainless steel 420 to stainless steel N700 can raise the heating rate from 5.7 to 7.6 °C/s (Chen et al. 2012). Furthermore a range of temperature within 6–8 °C/s has been recommended for practical applications.

Meanwhile, it should be reminded that fibre quantity and quality both play an important role in having a smooth and effective biocomposite process. Optimized pretreatment of natural fibres can avoid many processing damages and additional costs. Pretreatment of fibres involves washing out any undesirable excess component, bringing the fibre into a suitable shape, and increasing the thermal stability of the fibre. Recently through novel treatments and procedures, researchers have observed and characterized the correlation between temperature and the porosity of the materials which could be followed to enhance the fibre–matrix interface (Brewer et al. 2014; Shaaban et al. 2013). Furthermore, the mould configuration itself has a direct influence on the flow behaviour, and appropriate mould shapes may lead to enhance the final product anisotropy. Generally, in the mould, fibre content inside sharp curves is lower than that of other smooth sections.

6.3.2.4 Resin Transfer Moulding Process

For thermo-based processes, resin transfer moulding (RTM) process is advised. RTM is a low-pressure, low-speed (vacuum assisted) moulding process suitable to many well-known applications of biocomposite reinforcements (Laurenzi et al. 2014). During this process a blend of resin-hardener catalyst is added to the mould containing the preform fibre shown in Fig. 6.6. To avoid any resin leakage during injection, the two matching mould are compressed tightly. During this process, a former curing stage is recommended assuring that the resin is completely cured. Normally for polyester or vinyl ester resins where load bearing is not a critical filler, colourants and other resin modifiers are added into the resin to reduce costs, prevent problems such as shrinkage, improve flame resistance properties and also enhance the mechanical properties.

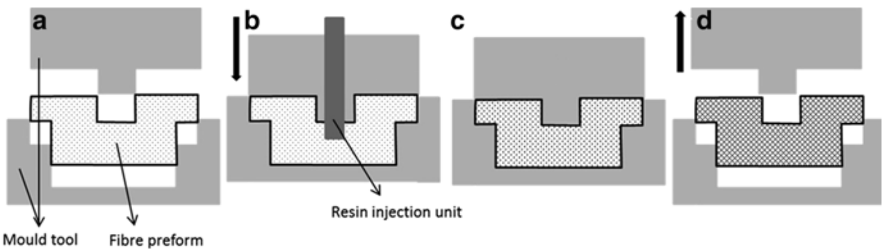

Fig. 6.6 Schematic diagram of resin transfer moulding process stages: (**a**) tools, (**b**) injection, (**c**) curing, (**d**) demould stage

RTM process involves notable advantages and disadvantages as follows:

Advantages	Disadvantages
– Very large and complex shapes can be made efficiently	– Professional tooling design and construction are required
– Dimensional tolerances	– Reinforcement loading may be difficult with complex parts
– Faster production	– Employed tools are costly
– Labour saving	– Mould design is complex
– Surface finish	
– Lower material wastage	
– Design flexibility	
– Significant strength to weight characteristics	

A rich application of natural fibres is in Lamborghini Aventador LP 700-4 Roadster 2014. To fabricate lightweight reinforced composites for this luxury model, the RTM technology has been employed. RTM technology has been applied in many common applications of natural fibre reinforced composites such as automotive, aircraft and construction.

During the RTM process, numerous parameters and variables are required to be controlled and optimized, permeability of the fibre mat, temperature of the mould, the resin injection pressure, the viscosity of the resin, the location and configuration of the gate, vent control and placement techniques of the preform and the preform architecture and permeability (Bickerton and Advani 1997). Following the resin's low viscosity, higher mould temperature and injection pressure can shorten the duration of the process cycle. Interestingly scientists have concluded that neither a pre-drying process nor any adjustments in the mould temperature has any noteworthy influence on the performance of kenaf fibre reinforced polyester laminates (Rassmann et al. 2010). Nevertheless, there are chances of deformation and damages in the preform fibre at high temperatures and pressures. Hence practical optimizations are required following the correlation between the parameters and variables. Optimizations have a direct influence on the fibre wetting properties, resin injection unit selection and void formations during the process (Park et al. 2011).

Various architectures of preform fibre are being used for RTM process, biaxial weave, triaxial weave, knit, multiaxial multilayer warp knit, 3D cylindrical construction, 3D braiding, 3D orthogonal fabric and angle interlock construction. The design of the preform fibre plays a significant role during the RTM process. It is challenging to assure full wetting inside the preform due to the inconsistent geometry of natural fibre and inhomogeneous fibre architecture. Furthermore, during the filling stage, incomplete wetting process and interruption in uniformity of the flow may occur due to the poor fitting size of the preform. Poor permeability of the preform may worsen this scenario. The inhomogeneity of preform fibre leads to non-uniform permeability.

During the process, the velocity differences progressively decrease due to the gradual increase in flow resistance. Through the flow from point to point, an uneven local velocity is observed due to the non-uniform microstructure of the preform fibre. At the same time, a vacuity between the preform and the mould edge can result in early arrival of edge flow causing an interruption in flow uniformity. Surprisingly, fibre concentration plays much more important role in the propagation of the edge flow compared to the injection pressure. Studies have suggested the application of larger preforms than the mould size to prevent the effect of edge flow. The edge flow results in dry spots and spillage. Furthermore, through different techniques, fast resin flow has been found applicable; however, inappropriate methods may result in the formation of unwanted bubbles affecting the finishing product properties. To prevent any void formation, vacuuming of the mould and injection pot has been recommended prior to the start of the injection process. Simulation studies have been carried out to minimize the void formation during RTM process (Lee et al. 2006). More recently, studies have been able to overcome the challenge of producing a uniform biocomposite by applying ultrasound technology during the RTM process (Planellas et al. 2014).

6.4 Conclusion and Future Perspective

Over the past one decade, much effort has been devoted to the use of natural fibre reinforced polymer composites due to their unique properties, biodegradability, environmental friendliness, notable accessibility, flexibility, easy processing and impressive physicomechanical properties. The material as well as the mechanical characteristics of these biocomposites is unlike the old carbon and glass fibre composite; hence research is still ongoing following the presence of an uncertainty in applying suitable manufacturing process for producing these composites. A combination of both traditional and modern processing techniques has been employed to produce novel reinforced polymer composites. This chapter has briefly addressed critical concerns on various processing techniques.

RTM process is found to be suitable for thermoset-based processes, while injection moulding and compression moulding process are found to be more appropriate for simple thermoplastic-based processing. Compression moulding is found to be a very productive and economical technique for producing reinforced composites. During the production of such composites, increase in yield ratio and decrease in cycle time have been recommended to be cost-effective. Following the limited natural fibre length, processes such as pultrusion are not applicable as high-tensile-strength fibres are necessary during the pultrusion process. Conventional dosing in an extruder is challenging in case of poor flow ability of natural fibres. A homogeneous dispersion of fibres into the polymer matrix is found to have a high impact on the product mechanical performance. Furthermore, the wettability of fibres was found to play an essential role during the process. Pretreatment of fibres is suggested to enhance the performance natural fibres through all processing stages.

Three fields of investigations are recommended for future studies: the influence of polymer viscosity on the processing quality, speed and cycle time; the application of ultrasonic technology during compression moulding process; and finding a suitable processing technique to reinforce polymer composites with recycled old newspaper.

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