v. 4, n. 18, 2024

Journal of
Engineering
Research

RESIN INFUSION TECHNIQUE APPLIED TO THE MANUFACTURE OF POLYESTER MATRIX COMPOSITES REINFORCED WITH NATURAL FIBERS – A REVIEW

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DOI https://doi.org/10.22533/at.ed.3174182421065

Abstract: The evolution in the resin infusion process is due to cost reduction, environmental regulations and the high quality of composites manufactured using these techniques. The resin infusion process essentially consists of using vacuum pressure to flow the resin through layers of dry materials (placed in a mold and covered by a vacuum bag) through carefully placed pipes. Once an infusion has started, little can be done to correct any errors; which normally lead to the total loss of the piece. For this reason, experience is acquired through a process of trial and error. Therefore, here we present a review of the available knowledge, techniques and matrices used, as well as a review of the state of the art on what has been done regarding the techniques used and properties obtained in composites with the inclusion of natural fibers.

Keywords: Resin infusion, composite materials, natural fibers.

ABBREVIATIONS

The following abbreviations are used in this manuscript:

RTM	-	Resin Transfer Molding
VARTM	-	Vacuum-Assisted Resin Transfer Molding
SCRIMP	-	Seemann Composite Resin Infusion Molding Process
VIP	-	Vacuum Infusion Process
UPR	-	Unsaturated Polyester Resin
MEKP	-	Methyl Ethyl Ketone Peroxide
V_f	-	Volumetric Fraction of Fibers
V_m	-	Matrix Volume Fraction
V_v	-	Void Volume Fraction (porosity)
K	-	Unsaturated Permeability in the Plane

INTRODUCTORY REMARKS

Technological development related to consumer demands continues to increase demand on global resources, leading to major issues of material availability and environmental sustainability (FARUK et al., 2012). In this scenario, several industrial sectors have sought to mitigate the environmental impacts arising from production processes, as well as the use of manufactured products and their disposal. Among these sectors, the automotive sector, often emphasized as a villain in effects such as global warming, has stood out in the search for environmentally viable alternatives.

The production chain of materials used in the manufacture of vehicles is more harmful to the environment than the smoke expelled from the exhaust pipes. There are emissions from the petrochemical, plastic processing and manufacturing industries (CASAS, 2010). Within this logic, governments seek alternatives, sometimes with public policies, sometimes with incentives for production diversity. In Brazil, the flex car (Ethanol-Gasoline and Gas) dominates the market. In other parts of the planet, for example, in the United States, Canada, Japan, China, France, Germany, Israel and Australia, the government and the private sector work together to develop efficient electric cars (COSTA, 2010).

Inherent in the development of innovative and “green” technological products, the use of lighter and more resistant materials can make projects in the transport sector viable as the weight/resistance ratio becomes more favorable. The use of composites in structural, semi-structural and/or functional components are among the options traditionally proposed for use in this segment.

The global composite materials industry reached a total value of 17.7 billion dollars in 2010 and, despite a still slow global economy, its growth rate was 10.3%. Of the global composite materials market revenue, resins accounted for US$8.8 billion and fibers accounted for US$7.7 billion, with auxiliary materials covering the remainder. Analyzing the penetration of composites in different market segments, LUCINTEL Consulting found that in 2010 in the transport sector, based on monetary value, composites represented 3.6% of the sector; in the naval sector, the share was 68%; in the Construction sector, composites represented 7.0% of the market; in the aerospace segments 10% and in wind energy 38%. These numbers clearly indicate that there is enormous growth potential for composites.

The potential of fiber-reinforced polymer composites was recognized more than 50 years ago, but only now can they find their applications in almost every sector, including construction, aerospace, automotive and electronics. Composite materials are increasingly used for dielectric applications, that is, applications that make use of electrically insulating or semi-insulating behavior (DHAL and MISHRA, 2013).

Since with the increase in the geometric complexity of parts associated with high structural performance in composite materials, the aeronautical industry is increasingly seeking to develop manufacturing processes that meet these needs associated with cost reduction, weight and integration of components without structural compromise. Thus, infusion processes such as Resin Transfer Molding and Vacuum Assisted Resin Transfer Molding have been gaining prominence for the manufacture of parts for aeronautical use. The terms RTM (Resin Transfer Molding) and VARTM (Vacuum-Assisted Resin Transfer Molding) are established terms in the aeronautical industry (PRADO, 2009).

More traditional techniques, such as autoclave-assisted manual lamination, do not meet the requirements for less polluting and more affordable processing currently regulated by environmental laws. Thus, new processes, such as resin transfer molding, or RTM, are constantly developing. The RTM processing technique basically involves the movement of the resin in a preform, so that it is completely filled, forming a composite material (ALVES, 2006).

One of the variations of the RTM process is the technology patented by Seemann and known as the Seemann resin infusion composite molding process, or SCRIMP. This technique allows the processing of parts with repeatability, without voids, with a high volumetric fraction of fibers, high specific resistance and low volatile emissions, which makes the process less harmful to the environment (ALVES, 2006).

In recent years, government regulations on carbon dioxide emissions and recyclability of materials have produced an increase in the use of natural fiber composite materials in both the automotive and construction industries. But one of the keys to its success is the possibility of using well-studied fiberglass composite processing techniques, such as RTM, VARTM or SCRIMP. Therefore, it is crucial to understand how key processing variables are affected when glass fibers are replaced with natural fibers, which have different structure, different fabric architecture, and different chemical interactions with resins. One of these variables is the permeability of the fabric, which is the key parameter that regulates the flow in the fiber bed, along with the viscosity of the fluid. Tissue permeability is especially important in low-pressure injection techniques such as VARTM or Vacuum Infusion Process (VIP), where void formation and injection time can be increased dramatically as permeability decreases (FRANCUCCI et al., 2010).

One of the main advantages of using processes such as RTM, VARTM and VIP is the absence of the need to use an autoclave, which is essential in the production of high-performance composites, but requires a high investment to acquire. Despite this, these processes can generate products with a high fiber content and low porosity, essential parameters for the good performance of composites.

The vacuum infusion process (VIP) is a molding process used to manufacture large composite structures. Its popularity is due in part to the low cost of the tooling set and environmental safety (the process eliminates more than 90% of volatile organic compounds emitted by unsaturated polyester resins). Furthermore, the low operator involvement increases the reproducibility of the process compared to open mold techniques such as hand-lay-up or spray-up and the components have a relatively high fiber content, up to 60% by volume (RAGONDET, 2005).

Composites reinforced with natural fibers have undergone an extraordinary transformation. These materials have become more and more sufficient as new compositions and processes have been intensely researched, developed and consequently applied. The oil crisis has made biocomposites significantly important and they have become engineering materials with a very wide range of properties. However, like all materials, they are constantly under competitive pressure from the global market, which in turn requires continuous development. The times of simply mixing plastics with loads of natural waste and characterizing their main properties are over (FARUK et al., 2012).

The new vehicle manufacturing proposal aims to replace polluting elements with natural products, so-called bioproducts, in their internal and external structure, in addition to the option for ecological fuels. Natural fibers replace plastic or metal parts. With each component of a vehicle that is replaced, there is a reduction in crude oil consumption at the end of the production chain (CASAS, 2010). The most common natural fibers used in composite applications are stem and leaf fibers (hard fibers) with fibers such as hemp, jute, flax, kenaf or sisal. These materials have already been adopted by European automotive manufacturers (ROUISON et al., 2004).

Considering this scenario, the permanent search for natural fibers with good mechanical resistance and the adaptation of composite processing parameters to these materials aligns with the logic of sustainable development necessary to maintain humanity's demands. As products from renewable sources are included in the various production sectors, with compromised properties and performance, environmentally favorable, economically viable and socially fair products are increasingly included in industrial activity, since the inclusion in the production chain of products with technical and economic viability that are accessible to traditional rural communities, for example, generates income and diversifies production in the field.

In this way, the infusion process aligns with the environmental, economic and social gains resulting from the application of natural fibers in composites and adds all the advantages of a closed manufacturing process, environmentally more correct than open mold processes, healthier for the worker and with the potential to manufacture composites with high finishing quality, low void content, high fiber inclusion values and with lower costs than performance systems similar. The successful application of the process for the manufacture of composites with reinforcement of natural fibers requires a thorough study of the techniques and parameters developed for synthetic fibers and their adaptation to natural fibers, taking into account the inherent properties of each type of fiber and the great abundance of species available in the natural environment with potential for application in composites.

LITERATURE REVIEW

COMPOSITE MATERIALS

The evolution of composites has allowed the manufacture of parts and structures previously reserved for metals. The use of the material in structural components increases its reliability and guarantees more and more applications. Projects such as the innovative Boeing 787 and Airbus A-350 reaffirm the potential of composites and their ability to reduce components and mass without compromising mechanical behavior (MARINUCCI, 2011).

The class of composite materials is quite broad and comprehensive, ranging from fiber-reinforced polymers, metal/composite hybrid materials and structural concretes, and other composites that incorporate a metallic matrix or ceramic matrix. Therefore, the basic characteristic of composites is to combine, at a macroscopic level, at least two distinct phases called matrix and reinforcement (LEVY NETO and PARDINI, 2006).

A composite consists of a reinforcement and a matrix. The reinforcement, of high strength and rigidity, is impregnated by the matrix, which maintains the fibers in their geometric position and transmits tension through the component, at the same time guaranteeing chemical resistance. Composite materials are used in a wide range of applications in the automotive, aeronautical and sports sectors and for construction and architecture. There are different manufacturing processes depending on the type of application, production rate and size of components (RAGONDET, 2005).

The properties of fiber-reinforced composites are directly associated with the properties of the material's constituent elements, such as fiber and matrix properties, concentration or volumetric fractions, interface and fiber/matrix adhesion, layer arrangement and orientation, as well as filament geometry, such as shape and size. Composites can be formed by particulate or fibrous reinforcement elements. Particulate reinforcements can be spherical, cubic, tetragonal or any other regular or irregular shape, but are almost always equiaxed. Fibrous reinforcements have fiber as a reinforcing element, which has the characteristic of having a length much greater than the dimensions of the cross-section (MARINUCCI, 2011).

Another determining factor in the performance of a composite against the most diverse types of requests are the volumetric fractions of fiber (V_f), matrix (V_m) and voids (V_v). These parameters are used to quantify the percentage volumes of each component (fibers, matrix and voids, respectively), in relation to the total volume of the composite. The values of V_f and V_m in any type of composite are determined by the manufacturing process adopted (LEVY NETO and PARDINI, 2006).

POLYESTER RESIN

Unsaturated polyesters are esters formed by the reaction of a dialcohol (glycol) and an anhydride or dibasic acid (diacid), releasing a water molecule. They are produced when one of the reactants contains unsaturation's, usually by maleic acid or anhydride. The main types of unsaturated polyester resins (UPR – Unsaturated Polyester Resin) are: orthophthalic, terephthalic, isophthalic and bisphenolic resins. As UPR is solid at room temperature, appropriate monomers are normally used, such as styrene, which is used to reduce viscosity and facilitate resin processability, in addition to participating in the resin polymerization process (TARGA et al., 2009).

Polyester resins are supplied to the molder in the form of a viscous liquid and transform into a rigid infusible (thermoset) solid through an exothermic chemical polymerization or curing reaction. However, the curing of polyester resins would take place very slowly, because the molecules that constitute it have low mobility. Consequently, the probability that two unsaturation's come close enough to give rise to intermolecular bonds is very small. This problem is solved by adding unsaturated monomeric units of low molar mass to the polyester resin, making the probability that intermolecular interconnections occur considerably greater. At the beginning of the reaction, the double bonds (unsaturation's) must be broken for interconnections to occur. This problem can be solved by heating the resin, applying electromagnetic radiation, or adding catalysts and reaction accelerators. The catalyst's free radicals attack the unsaturation's in the polyester or in the low molar mass monomer, styrene, for example, to initiate the chain polymerization reaction, which gives rise to a styrene-polyester copolymer, thus forming a thermorigid three-dimensional network (LEVY NETO and PARDINI, 2006).

The formulation of a polymeric matrix containing polyester resin is made by adding initiators and curing promoters to the resin. Curing initiators, generically called organic peroxides, can be hydroperoxides, such as cumenon hydroperoxide (CHP); and peroxides, such as methyl ethyl ketone peroxide (MEKP), benzoyl peroxide (BPO) and acetyl acetone (AAP). For systems formulated for cold curing, the most recommended peroxides are MEKP, BPO, AAP and CHP. For heat curing systems, BPO, tertium butyl peroctoate (TBPO) and tertium butyl perbenzoate (TBPB) are suggested. The activation of curing initiators is carried out by curing promoters (accelerators), the most common of which are tertiary amines or salts of cobalt, vanadium, copper or manganese, the first two being the most used. In everyday life, healing initiators are usually called catalysts. This term is not technically correct to express the phenomenon that occurs in healing. A catalyst is a substance that increases the speed of a chemical reaction without being actually consumed in the process, a fact that does not happen in the curing of polymeric matrices where the curing initiator is consumed in the reaction (MARINUCCI, 2011).

As there are a large number of acids and glycols available, it is possible to obtain a large number of resin variations. However, factors such as raw material cost and ease of processing reduce this number. On the other hand, if only unsaturated biaacids were used in the manufacture of polyester resins, the spacing between double bonds would be short, resulting in a brittle and brittle material. Therefore, it is necessary that the basic polyester formulation has saturated biacids in its composition that act as chain extenders. The greater the quantity and the greater the proportion of saturated acid, the more tenacious the polyester resin will be after polymerization, and the less shrinkage upon curing will be observed. The most commonly used saturated acids in the synthesis of polyester resins are orthophthalic acid (in the form of anhydride) and its isomer, isophthalic acid (LEVY NETO and PARDINI, 2006).

Thermosetting resins can be modified to provide greater ease of processing or to change properties. These modifiers can be either in the form of solid particles or in liquid form, such as diluents and elastomers. In general, the addition of diluents, whether reactive or non-reactive, is a standard procedure when high viscosity resins (>5 Pa.s) or even solid resins are used for reinforcing fiber impregnation processes. In the case of unsaturated polyester resins, the addition of a reactive diluent occurs at a proportion of 35 to 45% by mass, allowing viscosity control, cost reduction and providing greater wettability to the reinforcement fibers. The most commonly used reactive diluents for unsaturated polyester resins are styrene and diallyl phthalate (DAP). These diluents copolymerize with the unsaturation points present in the polyester molecular chain, forming cross-links with it (LEVY NETO and PARDINI, 2006).

NATURAL FIBERS

A greater awareness has emerged that non-renewable resources are becoming scarce and our inevitable dependence on renewable resources. This century could be called the cellulosic century, because more and more renewable plant resources for products have been discovered. Plants that produce natural fibers are classified as primary and secondary depending on their use. Primary plants are those grown for their fiber content, while secondary plants are plants in which fiber is produced as a byproduct. Jute, hemp, kenaf and sisal are examples of primary plants. Pineapple, palm and coir are examples of secondary plants (FARUK et al., 2012).

Vegetable fibers can be considered as composites of cellulose fibrils held together by a matrix consisting of lignin and hemicellulose, whose function is to act as a natural barrier to microbial degradation and serve as mechanical protection. Its structural characteristics are related to the nature of cellulose and its crystallinity. The most common organization of a plant fiber is represented in Figure 1. Each lignocellulosic fiber has a complex layered structure; consisting of a thin primary wall, initially deposited during cell growth, which surrounds a secondary wall. The secondary wall is made up of three layers (S1, S2 and S3), where the intermediate layer (S2) determines the mechanical properties of the fiber and consists of a series of microfibrils, helically formed by long cellulose chains and organized in the direction of the fiber. Such microfibrils have a diameter of 10 to 30 nm and are the result of the packaging of 30 to 100 extended cellulose chains (SILVA et al., 2009).

Climatic conditions, age and degradation processes influence not only the fiber structure, but also the chemical composition. The main chemical component of a living plant is water. However, on a dry basis, all plant cell walls consist mainly of sugar-based polymers (cellulose, hemicellulose), which are combined with lignin and with smaller amounts of extractable products, protein, starch and inorganic substances. The chemical components are distributed throughout the cell wall, which is made up of primary and secondary wall layers. The chemical composition varies from plant to plant, and within different parts of the same plant (FARUK et al., 2012). Table 1 shows the range of average chemical constituents for a wide variety of plant types.

Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Waxes (%)
Sugarcane bagasse	55.2	16.8	25.3	-
Bamboo	26-43	30	21-31	-
Flax	71	18.6-20.6	2.2	1.5
Kenaf	72	20.3	9	-
Jute	61-71	14-20	12-13	0.5
Hemp	68	15	10	0.8
Ramie	68.6-76.2	13-16	0.6-0.7	0.3
Banana	56-63	20-25	7-9	3
Sisal	65	12	9.9	2
Coir	32-43	0.15-0.25	40-45	-
Pineapple	81	-	12.7	-
Curaua	73.6	9.9	7.5	-
Rice husk	35-45	19-25	20	14-17

Table 1: Chemical composition of natural fibers. Source: FARUK et al., (2012).

The development of textile technologies such as weaving, knitting and braiding have resulted in the formation of composites with superior mechanical properties, with the

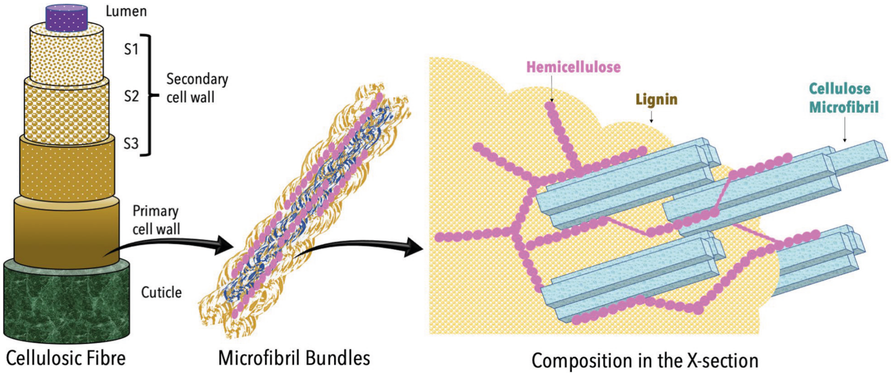


Figure 1: Structure of a plant fiber. Source: Shuvo (2020).

continuous orientation of the fiber not being restricted to one direction. In applications where more than one fiber orientation is required, a fabric combining 0° and 90° fiber orientations is useful. Fabrics are produced by interlacing warp fibers (0°) and weft fibers (90°) in a regular pattern or weaving style. The integrity of the fabric is maintained by mechanically blocking the fibers. Drape (the ability of a fabric to conform to a complex surface), surface texture and stability of a fabric are controlled primarily by the weave style (JOHN and THOMAS, 2008).

Figure 2 shows some typical styles of fabrics used in making composites.

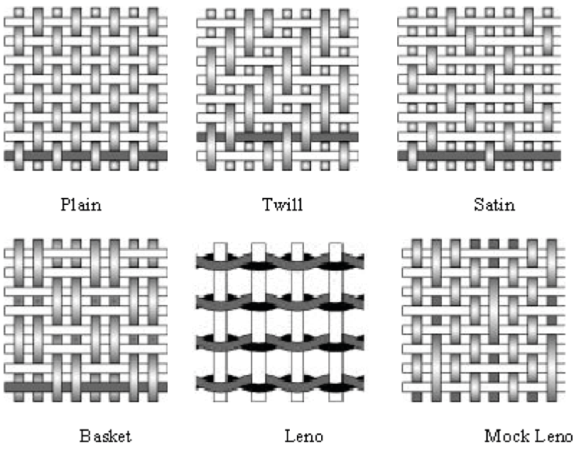

Plain
Twill
Satin
Basket
Leno
Mock Leno

Figure 2: Some typical fabric styles used as reinforcements in making composites. Source: LI et al., (2009).

POLYMER COMPOSITES REINFORCED WITH VEGETABLE FIBERS

Composites reinforced by natural fibers have attracted the attention of the scientific community mainly because they are becoming an alternative solution to depleting petroleum sources. The production of 100% natural fiber-based materials as a substitute for petroleum-based products is not an economical solution. The most viable solution would be to combine petroleum and bio-based resources to develop a low-cost product with diverse applications. The application of composites reinforced by natural fibers has been extended to almost all fields (JOHN and ANANDJIWALA, 2008).

Biocomposites are emerging as a viable alternative to glass fiber reinforced composites. Natural fibers have advantages over synthetic or artificial fibers (e.g. glass and carbon) in areas such as low cost, low density, competitive specific mechanical properties, reduced energy consumption, carbon dioxide sequestration and biodegradability. The combination of biofibers such as kenaf, hemp, linen, henequen and sisal with polymer matrices from non-renewable and renewable sources, for the production of composite materials that are competitive with synthetic composites, requires special attention to the biofiber-matrix interface, and its resulting adhesion, as well as to the processing methods used to produce these materials (DRZAL et al., 2003).

There are many parameters that affect the performance of a natural fiber reinforced composite. The degree and type of adhesion cannot be calculated quantitatively, although its importance is well recognized. Aspect ratio has a considerable effect on the properties of the composite, so it is important to conserve fiber length as much as possible during processing operations. The fiber aspect ratio should be in the range of 100-200 for maximum effectiveness. Fiber orientation has a significant effect on the properties of the composite. During processing, fibers tend to orient along the flow direction causing mechanical properties to vary in different directions (JOHN and ANANDJIWALA, 2008).

COMPOSITE MANUFACTURING PROCESSES

The final properties of materials are not only determined depending on their individual characteristics, but also on the way the materials are produced and the way they are inserted into this composite. In all existing composite manufacturing processes, from those that incorporate the simplest to the most advanced technologies, there is a set of limitations, namely, with regard to the production capacity of the parts, size, geometric shape, structural quality, homogeneity in production, possibility of automation and additional cost (LOPES, 2009).

The first consideration in the construction of composites is cost. The maritime industry generally opts for manual processes or other open mold techniques. However, Environmental Protection Agencies began to ban construction in open molds and began to require the manufacture of composites in closed molds, due to gas emissions that are harmful to the environment and the workers involved in these processes. Due to this fact, research and development of new techniques in closed molds to reduce costs and emissions has been greatly expanded. Traditionally, the RTM method has been chosen for manufacturing composites. This method offers many advantages, including better thickness tolerances, a less rough laminate surface, and a reduction in volatile gas emissions. However, the material costs required for this process are high for larger pieces. To reduce these costs, new techniques appeared, consisting of a variation of the RTM method, in which the closed mold is replaced by a bag, greatly reducing process costs. This is how Infusion techniques began to appear (FRANCO, 2008).

Manufacturing processes correspond to 50 – 60% of the total cost of a composite, and for this reason it is a subject that demands significant attention from the industrial and scientific community, in view of the interest in reducing the representation of this item in the final cost of the product. Composite processing can be classified in two ways: by the type of matrix to be used (polymeric, ceramic or metallic), or by the type of process used to obtain the composite, that is, processing via the liquid phase, via the gas phase or via solid particle phase. The selection of the manufacturing process for producing a composite component must mainly consider the size and geometry of the part, the desired microstructure, including the type of reinforcement and matrix, performance and market evaluation (LEVY NETO and PARDINI, 2006).

Another way to classify composite manufacturing processes is based on the tooling (mold) used. Conceptually, the manufacturing processes of composite material parts, components and structures can be divided into open mold processes and closed mold processes. The distinction lies in the quality of the finish achieved on the surface of the manufactured element and the quality of the molds required to form the part. Processes that use an open mold provide a smooth and uniform finish on only one side of the part, requiring only one mold, while in processes that use a closed mold, a smooth finish is achieved on both sides and a pair of molds, called male and female or plug-in molds, is necessary. Examples of processes that use an open mold are contact lamination (hand lay-up), projection lamination (spray up) and filament winding. The most commonly used closed mold processes are resin transfer molding (RTM/RTM light) and press molding (MARINUCCI, 2011).

As the levels of demand for composites became more stringent, manual manufacturing processes proved to be unsatisfactory in relation to controlling the orientation and volumetric fraction of fibers, which basically define the mechanical resistance of a composite. The orientation of the fibers did not imply changes in the process, but controlling the volumetric fraction of the fibers was a fundamental requirement in optimizing the process, so that the transfer of the mechanical resistance of the fibers to the composite was maximized. From the need to develop a process to impregnate the fibers in a more precise and homogeneous way, before the molding process itself, a semi-manufactured product emerged, known generically as pre-preg. Pre-preg is, therefore, an intermediate product, ready for the molding of composites, consisting of a mixture of reinforcing fibers impregnated with a specific polymer, formulated thermoset or thermoplastic, in a particular mass fraction, forming fabrics or tapes. unidirectional that form a single blade (LEVY NETO and PARDINI, 2006). Pre-pregs are formed from fibers impregnated with resins where their curing stage has already begun. This stage of curing is called stage B, where about 30% of possible cross-links are present. To prevent the curing process from advancing, and thus compromising the best conditions for molding the composite, pre-pregs must be stored at low temperatures (around -12ºC) (PRADO, 2009).

Table 2 illustrates a comparison between some of the main composite manufacturing methods, hand lay-up, autoclave and RTM/VARTM, depending on the process productivity and parameters achieved by the manufactured composites such as fiber volume fraction and reinforcement arrangement.

THE RESIN INFUSION PROCESS

The rapid growth of the composites industry and the ongoing effort to produce rigid, lightweight, and rapidly manufactured components has promoted the use of pressure techniques to consolidate and form materials with excellent mechanical properties. One of the main obstacles to the insertion of the composites area in the market is the high cost associated with some manufacturing technologies for these materials, such as autoclave processing. In response to these challenges, liquid composite molding processes have stood out, such as RTM (Resin Transfer Molding) and VIP (Vacuum Infusion Process). However, despite its popularity, the RTM process continues to require large investments, as it uses very heavy and complex molds capable of withstanding injection pressure in large parts, which leads to high production costs. VIP promotes the obtaining of a robust, high-quality laminate with a reduced number of imperfections. As in any other composite manufacturing process, planning its development and care during manufacturing are critical factors for the success of the process (LOPES, 2009).

According to the GURIT Guide to Infusion, resin infusion offers the advantages of using

Process	R.S[a]	Prod.[b]	V_f^c	Cost	A/R[d]	Form
Hand lay-up	Low	Low	Medium	Low	2D	Simple to complex
Autoclave	High	Medium	High	Medium to high	2D	Simple to complex
RTM/VARTM	Medium	High	Medium to high	Low	2D, 3D	Simple to complex

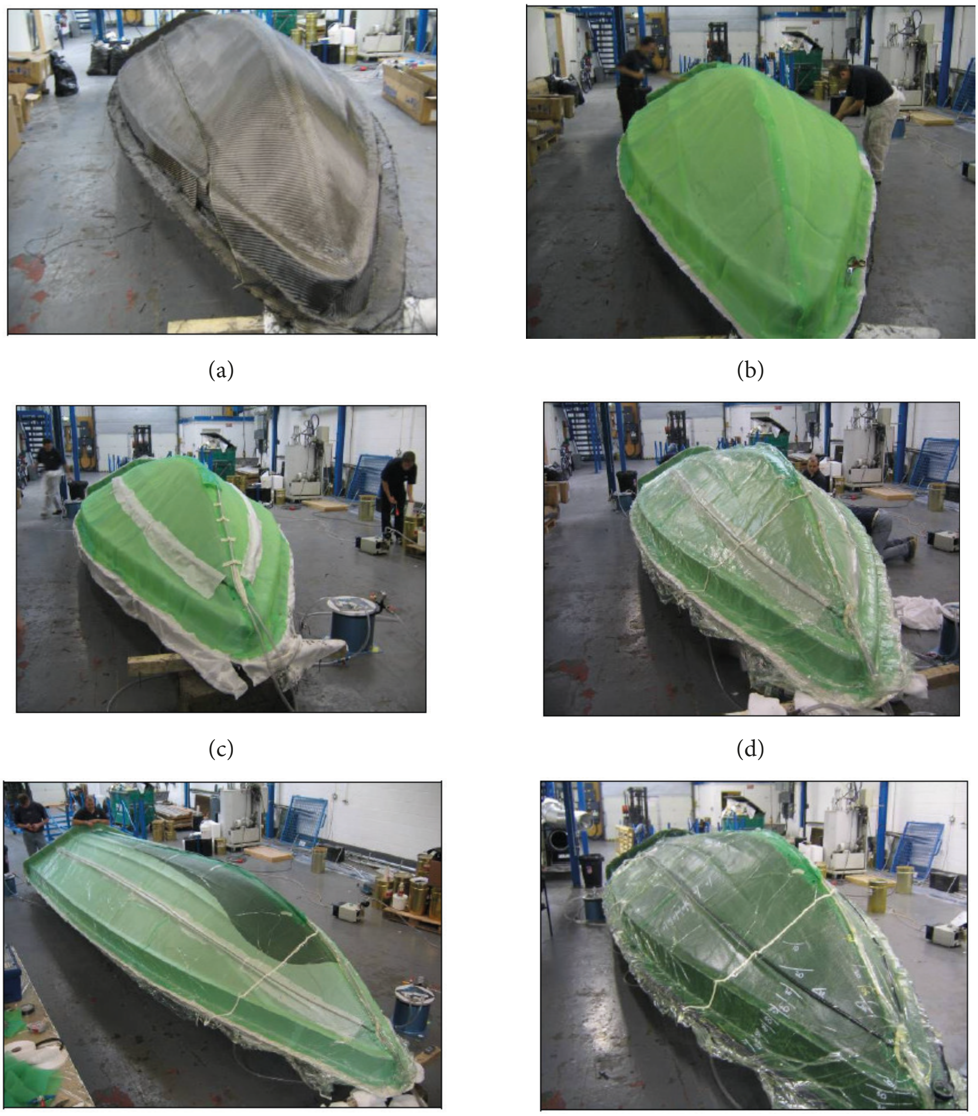
R.S[a] - Required Skill.

Prod.[b] - Productivity.

V_f^c - Fiber volume fraction.

A/R[d] - Arrangement/Reinforcement.

Table 2: Comparison of different composite manufacturing processes (NAIK et al., 2013).



(a) (b)

(c) (d)

(e) (f)

Figure 4: Boat construction process by VIP. (a) placing the reinforcements on the mold; (b) positioning of the infusion screen; (c) installation of resin inlet channels and vacuum lines; (d) sealing the system using the vacuum bag and sealing adhesive; (e) system pressurization and resin infusion; (f) end of resin infusion and beginning of composite curing. Source: Gurit Guide to Infusion.

conventional reinforcing materials and slightly modified resin systems to create components that provide low void content, with optimal fiber volume fraction values at a much lower cost than similar performance systems such as pre-pregs. This is generally made possible by the low cost of the tooling required for the composite, which can be cured at room temperature or at low temperatures.

The infusion process is characterized by a flow of resin, coming from a container, from the entry channels in the laminate to the exit channels, which lead to an intermediate container. The main difficulty encountered during planning the infusion process refers to the size, the shape and type of laminate to be manufactured, which requires a different arrangement of channels for each part shape, to allow complete flow throughout the laminate, without delay in fluid progression or solidification of the injected resin before the infusion is complete (LOPES, 2009).

VIP is part of a family of molding techniques called liquid composite molding (LCM). It involves the infusion of a low viscosity resin into a dry, fibrous preform placed in a rigid mold and covered by a flexible membrane. The process is based on a pressure gradient to drive the resin into the mold and impregnate the preform. The mold enters at atmospheric pressure and exits under vacuum (RAGONDET, 2005). The typical configuration for VIP is illustrated in Figure 3.

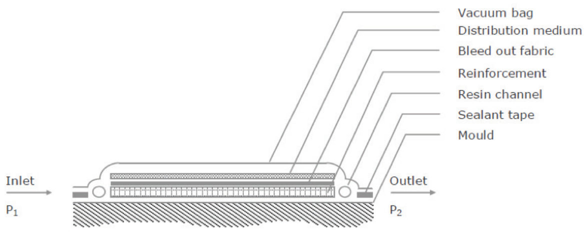


Figure 3: Schematic drawing of the infusion process. Source: RAGONDET (2005).

The infusion process has been used in several industrial applications, notably the naval, automotive, wind turbine and civil aviation industries. Figure 4 shows an example of application of the process in an industrial environment.

Resin infusion processes have several variables to guarantee the quality of the component to be manufactured, from characteristics of the materials used to determining the injection and resin exit points. Among the various variables we can highlight the viscosity of the resin system and the permeability of the dry reinforcement. Some other variables are directly or indirectly related to one of these two variables, such as temperature (ALVES, 2006).

INFUSION FLOW CHARACTERISTICS

The impregnation of the preform by the resin occurs in two ways: macro and micro-flow. Macro-flow consists of impregnation between wires. While microflow consists of impregnation inside the wires. Yarn is considered to be a set of filaments or fibers. The preform can contain wires oriented in various directions, and flow can occur in different directions relative to the wires (ALVES, 2006).

It is assumed that as the advancing flow front encounters a fiber bundle, it flows around it, trapping an air pocket as it does so. After the front surrounds and contours a bundle of fibers, it is slowly impregnated by the fluid. The basis of the assumed retention mechanism is that the interstitial space within the fiber bundles is much smaller than the spaces between the fiber bundles that constitute the preform. Thus, it is expected that the permeability of a fiber bundle for resin impregnation is much lower than the permeability of the preform for resin flow (PARNAS et al., 1994). This mechanism is schematized in Figure 5.

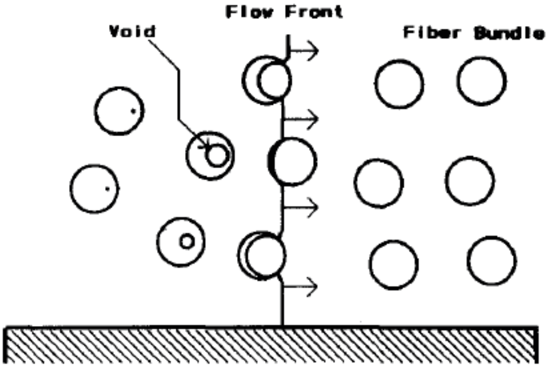


Figure 5: Process of introducing air into a flow through a preform with the fibers oriented perpendicular to the flow. Source: PARNAS et al., (1994).

The complementary process of flow perpendicular to the direction of the wire is the case of liquid flowing parallel to the direction of the wire. In this case, the geometry that must be considered is shown in Figure 6, which describes a low permeability thread embedded in a higher permeability medium, which is made up of other threads and more open regions between the threads. The shape of the flow front illustrated indicates the case in which the flow front would be delayed in the wire due to lower permeability inside the wire than outside it (PARNAS et al., 1994).

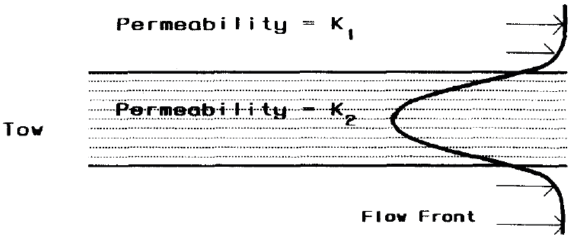


Figure 6: Flow model parallel to a low permeability wire embedded in a higher permeability medium. Source: PARNAS et al., (1994).

There are four important aspects to consider when modeling resin flow in infusion processing: (i) the influence of resin pressure on the fiber volume fraction of the preform, because resin pressure reduces compaction; (ii) the effect that changing the volumetric fraction has on the permeability of the preform, since permeability is a function of the volumetric fraction of the fibers; (iii) the influence of compaction change on fluid movement and preform thickness; (iv) the effect of the fiber preform that behaves as a double-scale porous medium (ACHESON et al., 2004).

Most preforms used in infusion consist of bundles of fibers. The resin generally flows around it into these beams. This makes the analysis difficult, as one must continue to account for the mass loss to the beams even after the flow front has passed and is far from them. This phenomenon is often referred to as dual-scale porous media because there is macroscale flow around the beams and microscale flow within them (PARSEVAL et al., 1997), as schematically shown in Figure 7.

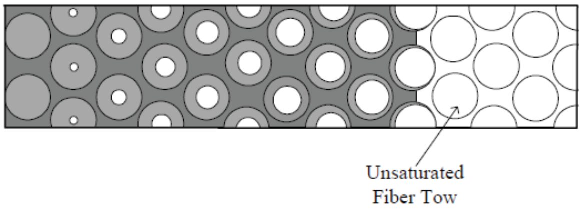


Figure 7: Illustration of dual scale flow. Source: MASTBERGEN (2004).

Typical preforms used in RTM and infusion consist of regularly or randomly textured fiber bundles. The microstructure of the fiber preform is highly non-uniform. Between beams there are channels, through which the resin can flow very easily and consequently the effective permeability is high. The local permeability within the beam, on the other hand, is low because of the presence of fibers. However, the capillary pressure within the bundle is much higher than that between the bundles. The effects of local permeability and capillary pressure compete with each other and the local velocity of the resulting resin depends on both factors. In the case of a high resin velocity, voids will form within the fiber bundles because the flow in the channel is faster than within the bundle due to the high permeability of the channel (Figure 8 (a)). For a low resin velocity, however, capillary flow will dominate within the bundles and therefore voids will form in the channels between bundles (Figure 8 (b)) (KANG et al., 2000).

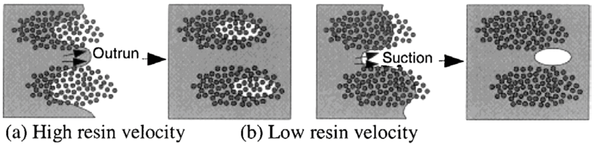


Figure 8: Formation of voids within and between fiber bundles. Source: KANG et al., (2000).

The fluid percolating through the porous structure of the preform can generate double-scale impregnation, generating a delay in the impregnation of the interior of the fiber bundles (microflow) in relation to the position of the flow front. A good strategy must be used to complete the impregnation. As mentioned previously, a pressure gradient is present in the cavity, therefore, the thickness and fiber volume fraction of the preform being impregnated are not uniform. In order to optimize the quality of the final component, the fiber content must maintain the design specifications, in other words, the thickness of the laminate must be controlled. This can be achieved by subjecting the entire mold cavity to the lowest absolute pressure present in the cavity, that is, the pressure in the exit channel. One solution, called bleeding, consists of closing the inlet and letting excess resin flow out of the mold until pressure balance is reached in the cavity. However, this technique presents some risks since the resin formulation has to be adjusted so that gelation does not occur before a uniform thickness distribution is obtained. Furthermore, it is not possible to define the best time to stop the bleeding process. One risk is allowing too much resin to flow out, which would result in a lack of resin in the laminate (RAGONDET, 2005).

Conventionally used manufacturing methods have converged to adopt the practice of resin bleeding to reduce or eliminate microvoids. Generally, when the resin reaches the exit channel, its injection is interrupted. The practice of resin bleeding continues the injection even after the resin reaches the exit channel. This excess resin is allowed to flow out of the mold cavity into a resin trap. This practice allows the fiber bundles, which have a much lower permeability than fabric, to continue to saturate with resin. This bleeding concept is definitely warranted, but it remains difficult to determine how much resin bleeding should be allowed to ensure that all regions are saturated (KUENTZER et al., 2007).

An alternative processing practice is to add a flow resistance at the outlet channel position to reduce permeability. This practice reduces the amount of resin wasted due to bleeding. The additional resistance, positioned as an attachment to the bleed channel, slows the rate of resin bleeding and also provides higher pressure and longer resin residence time in the mold to saturate the fiber bundles. Tubing with a smaller diameter or additional layers that are porous but have a lower permeability than the preform can be connected to the outlet channel to create additional resistance. Again, it would be useful to quantitatively determine the level of strength that must be added to ensure that all areas are fully saturated with resin, as well as the relationship between the level of resistance and the amount of resin wasted due to bleeding (KUENTZER et al., 2007).

COMPRESSIBILITY OF REINFORCEMENT FIBERS

Reinforcement compressibility is very important in all resin transfer processes, and affects both the material and the processing properties of the part. As the fabric is compressed by fluid pressure or the mold surface, the fibers become compacted and the fiber volume fraction increases. This decreases the thickness of the part, decreases permeability, and decreases porosity. It is possibly more important to understand compressibility in single-sided molding processes than in dual-mold processes (male and female). In a double mold process, the permeability and thickness of the fabric are fixed at a certain value that is equivalent to the internal thickness of the mold. Throughout the process, permeability is constant and independent of injection pressure. In single-side molding processes, fabric compaction can lead to several important phenomena. In processes where the flow is in the plane of the fabric, such as VARTM and VIP, a part with a non-uniform thickness can be created as the liquid compaction pressure varies throughout the mold (MASTBERGEN, 2004).

In processes where the resin is forced through the thickness, the pressure applied to the fluid is also the compression pressure of the fabric. Therefore, the thickness and permeability of the fabric can change throughout the process and depends on the pressure arising in the process. This can create an interesting competition mechanism in these types of processes. According to Darcy's law, an increase in pressure increases the velocity of fluid through tissue. However, increasing fluid pressure will increase compaction pressure and decrease permeability. It could be possible, in certain cases, for an increase in pressure to increase the injection time, although this is not common. For most fabrics, the decrease in thickness tends to compensate for the decrease in permeability in flow through the thickness. The effect of compaction on permeability is very dependent on tissue architecture, meaning that some tissues are affected more than others. The compression of the fabric also affects its porosity, which will affect the saturation time of the fabric fibers by the fluid. This fact adds yet another complication to the problem. Although permeability decreases with compaction, decreasing porosity can increase fluid velocity through the preform. Decreasing porosity also increases capillary pressure. However, in most cases these effects are minor (MASTBERGEN, 2004).

In the infusion process, the vacuum is used to promote the flow of the resin through a flexible mold, which does not restrict the expansion of the preform created when the resin penetrates the mold and reduces the pressure supported by the tissues. Therefore, preform compaction through thickness will change as resin fills the mold. This physical change translates into changes in mass balance and permeability during flow. In infusion, the pressure distribution between the preform and the resin changes dynamically because the total pressure must be equal to the compaction air pressure during resin flow. The essential question is, therefore, the importance of this behavior in the overall model of the infusion process (ACHESON et al., 2004).

Experimental compaction studies of fibrous preforms were carried out by CHEN et al., (2001). The researchers identified three distinct regimes when compressing the preform fabric: an initial linear regime, an intermediate nonlinear regime, and a final linear regime, as illustrated in Figure 9. The first linear regime begins when the fabric layers are lightly compacted with a minimum amount of pressure and the cross sections of the fibers are brought into contact. The frictional resistance at the contact points between the layers of yarn prevents them from slipping. However, increased pressure can cause slippage to occur around large voids where the stacking structure is not stable. During this phase, the apparent deformation of the preform comes almost entirely from the apparent compressibility of the intertial spaces caused by the yarns filling the voids, not from the compressibility of the solid fiber itself. In the next stage, the nonlinear regime, the larger voids were filled and in the remaining interstitial space there is a more stable structure. At this stage, the compression of solids and the deformation of voids contribute to deformation. When additional pressure is exerted, the solids are subjected to large deformations, reaching the third regime. At this stage, it is assumed that the geometry of the cross-section has achieved self-similarity such that the porosity approaches a constant. The compression that occurs in this regime is due only to the compression of the solid material.

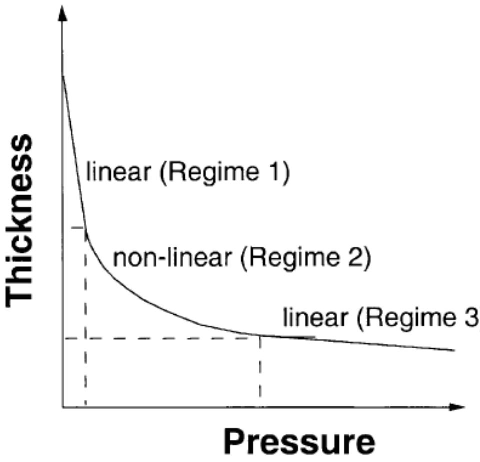


Figure 9: Thickness versus pressure curve of preform fabrics upon compaction. Source: CHEN et al., (2001).

REINFORCEMENT PERMEABILITY

Permeability indicates the relative ease with which the resin moves through the pore space in the porous medium and is a fundamental property used in the study of RTM/VARTM and infusion. Permeability depends on reinforcement architecture, porosity, resin properties, processing conditions, mold design and part geometry. Resin properties that affect permeability are viscosity, surface tension and contact angle. Processing conditions such as injection pressure, flow rate and temperature affect permeability. Permeability can be unsaturated (transient, occurring at the beginning of the process when the fluid at the flow front is surrounding and saturating the fibers) or saturated (steady state). When the flow condition is stabilized, the flow behavior would be independent of time and distance. Such a condition is considered an equilibrium state and the flow behavior is characterized using the steady state or saturated permeability. Under such conditions, there is a linear relationship between flow rate and pressure difference (NAIK et al., 2013).

The type of reinforcement directly influences permeability, being able to increase or reduce it, depending on the way the fibers are designed/arranged. A fabric with greater intertwining of the threads, such as a plain weave, will have more channels between the fiber bundles through which the resin can flow more easily. When the fabric fibers are not intertwined, as is the case with non-woven fabrics, there will be fewer channels between the fiber bundles, making it difficult for the resin to flow, reducing permeability. In general, the more interwoven the reinforcement, the greater the permeability to resin flow and the better it will be for the resin infusion process. However, a high degree of yarn intertwining will limit the maximum volume of the fiber fraction because there will be more open space that cannot be easily compressed. In thin-walled parts, such as those obtained in the infusion process, it can be considered that the resin flow occurs in the plane. However, parts obtained through the RTM process can reach 80 mm in thickness or more. In these cases, when the thickness is greater than 10 mm (approximately), permeability in three directions must be considered (PRADO, 2009).

In order to better understand the dynamics of impregnation in the infusion, this is compared to a simpler process, such as RTM. The latter involves a preform placed in the cavity of a mold with constant thickness. The infusion is conducted under positive pressure or flow rate. Since the thickness of the cavity is constant, the permeability of the preform remains constant during infusion. In this example, the impregnation dynamics are equivalent to a straight one-dimensional planar flow. The latter is assumed to correspond to the flow of an incompressible fluid through a porous medium, as described by the equation known as Darcy's Law:

$$v = \frac{K}{\varepsilon.\mu} \cdot \frac{dP}{dx} \tag{1}$$

where **v** is the interstitial velocity, **K** the permeability of the preform, **ε** the porosity, **μ** the viscosity of the fluid and **dP/dx** the pressure gradient along the flow distance.

The permeability of the medium is a property of the reinforcing material that characterizes the ease with which the resin flows along the material. This parameter is generally considered a constant in Darcy's Law, but it can be experimentally determined. Permeability is a characteristic strongly influenced by the volumetric fraction of fiber existing in the composite, that is, the level of compression during vacuum infusion. In short, we can infer that the permeability of the reinforcement has a great impact on the filling time of the component and the higher its value, the faster the mold filling velocity (LOPES, 2009).

A form of integration of Darcy's law shows time as a function of the squared distance to the flow front, as described in Equation 2.

$$t = \frac{\mu\,.\,\varepsilon}{2\,.\,K} \cdot \frac{x^2}{\Delta P} \tag{2}$$

In the infusion process, the upper face of the mold is made of a flexible membrane. The pressure difference is applied between the mold inlet, connected to a resin container, under atmospheric pressure, and the mold outlet, connected to a vacuum pump. The preform is ready to be infused when the air has been extracted from the cavity, the airtightness of the mold has been checked and the pump has been adjusted to the required level of vacuum. Then, the inlet is opened and the resin impregnates the preform. During infusion, preform compaction evolves locally with the pressure gradient and the latter develops with the position of the flow front as shown in Figure 10. The impregnated part of the preform is subjected to a non-uniform pressure distribution, with atmospheric pressure at the entrance to the resin channel and vacuum at the flow front. The compaction and permeability of the impregnated area vary with the position and progression of the flow front. The thickness of the impregnated part is not uniform, the preform is typically thicker at the inlet where the pressure level is highest. The dry part of the preform is under uniform pressure equivalent to the vacuum established in the exit channel. However, Darcy's law remains valid for infusion flow if combined with the continuity equation and if the thickness and porosity distribution are known. The flow in the cavity develops only in the plane and is straight (at this stage of the infusion description it is assumed that the lay-up has homogeneous permeability, that is, without different permeability layers) (RAGONDET, 2005).

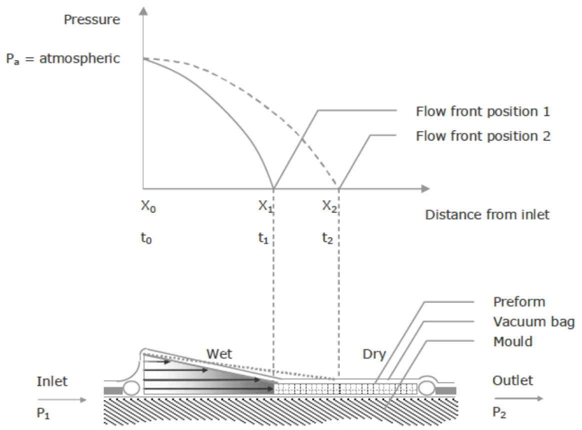
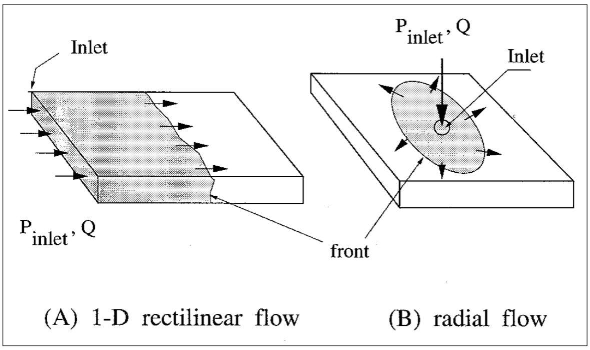


Figure 10: Evolution of pressure in the mold cavity with the progression of the flow front during vacuum infusion. Source: RAGONDET (2005).

Tissue permeability can be calculated if the pressure and flow rate required in the infusion process is determined. This can be done through straight or radial flow front tests (Figure 11). For straight tests, permeability can be easily calculated, however two different measurements are necessary to obtain permeability in the X and Y axes. For radial tests, permeability in X and Y is determined in a single test, however the analysis of the data obtained is more complex. When measuring permeability by radial infusion, it is also possible to eliminate end effects (edges), which can negatively influence the determination of permeability using the straight-line method (PRADO, 2009).



(A) 1-D rectilinear flow

(B) radial flow

Figure 11: Schematic representation of infusion with straight and radial flow. Source: PARSEVAL et al., (1997).

The flexibility of the vacuum bag introduces a new aspect that is not present in RTM: the fluid pressure field that induces flow also modifies the state of local compaction of the reinforcement and, ultimately, changes the permeability. As changes in fluid pressure are due to the movement of the flow front, the same happens with the compaction pressure on the reinforcement, which leads to variations in porosity and permeability. This is illustrated in Figure 12 (CORREIA et al., 2004).

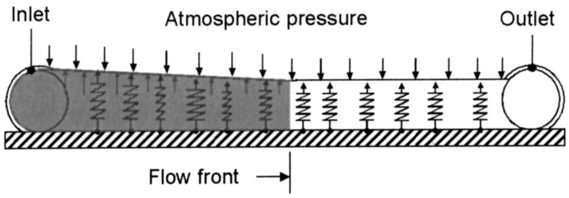


Figure 12: Effect of fluid pressure on compaction in VIP. Source: CORREIA et al., (2004).

Although some permeability values have been reported for natural reinforcements in the literature, a detailed insight into their flow behavior is still needed. Furthermore, results obtained for one fiber type and fabric architecture are difficult to compare with values obtained for other fibers. Therefore, it is very important to identify the main mechanisms present in the impregnation of natural fibers. A fundamental aspect that has been studied by several authors in glass fibers is the difference between saturated and unsaturated permeability (FRANCUCCI et al., 2010).

Unlike synthetic fibers, natural fibers absorb fluid, acting as a dissipator. Liquid absorption consumes fluid from the main stream, which travels through the reinforcement, increasing resistance to flow during unsaturated flow. Furthermore, saturation of natural fibers can cause swelling, which can reduce porosity and increase resistance to flow during saturated flow (FRANCUCCI et al., 2010).

INFUSION ARCHITECTURE

In infusion time is a critical factor in the process. Infusion and demolding of the manufactured part are generally slow processes, especially when producing large structures. One solution to reducing manufacturing time is to decrease infusion time by adding a delivery medium to the preform. This, generally a polymeric polyamide or polyethylene mesh, can also be a high permeability reinforcing layer made from the same fiber as the preform. The delivery medium is normally placed on the top face of the preform, as it must be released after infusion (not if the delivery medium is a reinforcing layer). When the distribution medium is a polymeric mesh, a layer of release fabric is placed between the preform and the distribution medium. This fabric, a thin layer generally made of polyamide and with low surface density, helps remove the distribution medium. Due to its high permeability, the distribution medium increases the resin flow on the upper surface of the preform, thus reducing impregnation time by up to 86% (RAGONDET, 2005).

Several researchers [FRANCO (2008); MASTBERGEN (2004); ALVES (2006); PRADO (2009); LOPES (2009)], have dedicated themselves to studying different infusion architectures that guarantee complete wetting of the reinforcements by the resin during the process, allowing total saturation of the preform and preventing thickness variation throughout the manufactured part. Flow promoters (such as delivery media), flow retardants (generally lower permeability fabrics), flexible membranes with directional channels for flow, diameters and positioning of the resin inlet and outlet tubes are some of the mechanisms used to ensure the success of the infusion.

PROPERTIES OF NATURALLY REINFORCED COMPOSITES

As the applications of composites reinforced with natural fibers multiply, the responsibility for resistance and predictability of properties becomes more demanding and is no longer sufficient, as reported by FARUK et al., (2012) the simple loading of natural waste into thermoplastic and thermosetting polymeric matrices. Several works have been developed in order to establish repeatability and predictability of the properties of composites reinforced with natural fibers, always using the most common synthetic fibers such as fiberglass as a mirror.

ALVES et al., (2010) compared the performance of composites reinforced with jute fibers and glass fibers applied to the automotive industry. Figure 13 shows a semi-quantitative comparison of all aspects of an automotive front hood reinforced with each of these fibers, based on the sustainability of the project and technical aspects, since the ecological design of materials needs to meet technical requirements. The best results were assumed to be 100%, while the other values are a fraction of them. Related to the technical parameters, the results were based on tests carried out on the composites tested according to the ASTM standards (D-3039, tensile test on polymer composites, D-790, flexural test on reinforced and unreinforced plastics and D-256, Izod impact test on plastics).

The social parameter is a qualitative aspect, which was based on the social effects of fiber reinforcement at all stages of the production chain. Jute fibers showed better aspects in all of them, with glass fibers only showing some advantages in increasing industrial employment.

The environmental aspect was established based on life cycle assessment, which takes into account aspects such as raw material logistics, fiber production chain, vehicle production chain and disposability, concluding that jute fiber adds an increase of around 15% in the performance of composites, while in economic aspects it was taken into account that jute fibers cost around seven times less than glass fibers. In this sense, it is possible to observe that jute fibers have many advantages in replacing glass fibers to reinforce composite materials.

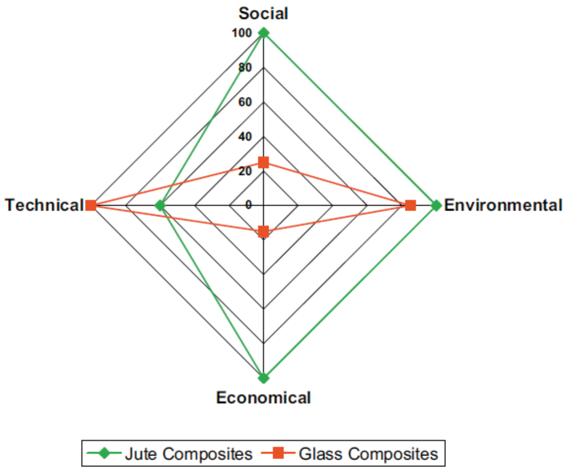


Figure 13: Performance comparison of auto parts made from composite reinforced with glass and jute fibers. Source: ALVES et al., (2010).

Due to the great variability of mechanical and physical properties of plant fibers, there is great difficulty in determining an adequate cross-sectional area, which is necessary to calculate the average tensile strength of the fibers. This occurs because materials from natural sources have large variations in their microstructure and mechanical properties, due to different climatic conditions during their cultivation, such as humidity, rainfall, solar irradiation, soil composition, among others (TARGA et al., 2009). This inherent variability in reinforcement causes a certain level of dispersion in the mechanical properties of composites reinforced with natural fibers.

The consolidation and expansion of the market for composites with natural reinforcement necessarily involves the repeatability and predictability of their properties. Research has converged on the effort to develop analytical and semi-empirical mathematical models that can incorporate the various variables that govern the properties of these composites, normally starting from already consolidated models developed for synthetic composites.

Micromechanical models are widely used to calculate the mechanical properties of composites. These models are analytical equations developed to simulate simplified geometric and mechanical representations of the real world, but, nevertheless, they usually show a good fit with the measured properties. The equations contain a number of material parameters that, in principle, are directly measurable or can be estimated (MADSEN et al., 2009).

The Rule of Mixtures is a widely accepted micromechanical model for calculating the stiffness and tensile strength () of composites with unidirectional continuous fibers, loaded in the longitudinal direction of the fibers. According to CALLISTER JÚNIOR (2002), the limit of longitudinal tensile strength of composites reinforced with aligned continuous fibers subjected to loading in the direction of the fibers can be calculated by Equation 3 (Rule of Mixtures):

$$\sigma_c = (V_f \sigma_f + V_m \sigma_{m'}) \tag{3}$$

where V_f is the volumetric fraction of fibers; σ_f the tensile strength of the fiber; $\sigma_{m'}$, the tension in the matrix at the moment fiber failure occurs and V_m the volumetric fraction of the matrix.

AL-QURESHI and STAEL (1997) proposed the Modified Mixture Rule for natural fiber composites, shown in Equation 4, which takes into account, in addition to parameters related to the properties of the fiber, matrix and the proportion between them, important aspects that directly influence the mechanical properties of the composite, such as the arrangement of reinforcement and the fiber-matrix interface.

$$\sigma_{c\_theoretical} = (\beta)(\sigma_f)(V_f) + (\gamma)(\sigma_m)(V_m) \tag{4}$$

where, $\boldsymbol{\sigma_{c\_theoretical}}$ the tensile strength of the composite; $\boldsymbol{\beta}$ the fiber alignment efficiency factor; $\boldsymbol{\sigma_f}$ the tensile strength of the fiber; $\boldsymbol{V_f}$ the volumetric fraction of fibers; $\boldsymbol{\gamma}$ the fiber matrix adhesion factor; $\boldsymbol{\sigma_m}$ the tensile strength of the matrix and $\boldsymbol{V_m}$ the volumetric fraction of the matrix.

The value of β can be defined according to Table 3.

According to CASARIL et al., (2007), the factor refers to the efficiency of covering the phases present by the matrix and varies from 0 to 1 depending on the ratio between the shear stress at the matrix/fiber interface and the shear stress of the matrix.

TARGA et al., (2009) manufactured orthophthalic polyester matrix composites reinforced with jute fiber fabric by the processes of manual lamination, resin infusion and lamination followed by compression. The values obtained for the three manufacturing processes tested are shown in Table 4. Based on these results, the authors concluded that the compression lamination technique presented the best fiber-matrix adhesion, due to the greater wettability of the resin in the fiber, possibly caused by the compression force.

Porosity is an inevitable part of all composite materials, due to the mixing and fusion of two distinct material phases, the fibers and the matrix. In the case of plant fiber composites, the porosity phase typically makes a significant contribution to the overall volume of the composite, caused by factors such as: (i) the existence of cavities in the lumen of plant fibers; (ii) the complex surface chemistry of plant fibers that makes fiber/matrix adhesion difficult; (iii) the irregular shape and dimensions of the plant fibers, which restrict impregnation by the matrix; (iv) the low packaging capacity of plant fibers, which limits the maximum volumetric fraction of fiber obtained, and (v) the processing techniques applied; which are normally directly adopted from work on synthetic fiber composites, still needing to be adapted for plant fiber composites. Altogether, porosity is a bulk phase of plant fiber composites that cannot be neglected, and needs to be routinely integrated into the assessment of composite performance (MADSEN et al., 2009).

Researchers such as MADSEN (2004), SHAH et al., (2013), have proposed models to predict the mechanical properties of composites reinforced with natural fibers considering the effect of porosity and its interactions with the other phases of the composite. MADSEN et al., (2009) developed an equation derived from the Rule of Mixtures that includes porosity and simulates its effect on the tensile strength of plant reinforcement composites:

$$\sigma_c = (\eta_0 \eta_1 V_f \sigma_f + V_m \sigma_{m*})(1 - V_v)^2 \tag{5}$$

where $\boldsymbol{\eta_0}$ is the fiber orientation efficiency factor; $\boldsymbol{\eta_1}$ the fiber length efficiency factor; $\boldsymbol{V_f}$ and $\boldsymbol{\sigma_f}$ the volume fraction and tensile strength of the fiber; $\boldsymbol{V_m}$ and $\boldsymbol{\sigma_{m*}}$ the volume fraction and tension of the deformed matrix at the point of failure of the composite; $\boldsymbol{V_v}$ the porosity.

In terms of mechanical performance, lignocellulosic fibers have proven to be attractive. However, due to the short length of technical plant fiber, manufacturing composites with unidirectional reinforcement may require the reinforcement to be in the form of threads. The spinning process results in a twisted structure, where twist is the primary binding mechanism. The addition of twist to the yarn affects the transfer of tension between the fibers within the yarn and

Fiber Orientation	**Load Application Direction**	**Reinforcement Efficiency (β)**
All fibers parallel	Parallel to the fibers	1
	Perpendicular to the fibers	0
Bidirectional (Tissues)	Half of the fibers are parallel and half are perpendicular	1/2
Fibers distributed uniformly and randomly in a specific plane	Any direction in the plane of the fibers	3/8
Fibers distributed uniformly and randomly in a three-dimensional space	Any direction	1/5

Table 3: Efficiency of fiber reinforcement in the composite for some fiber orientation angles in relation to the loading direction (CASARIL et al., 2007).

Manufacturing Method	$\sigma_{c\_experimental}$	$\sigma_{c\_theoretical}$	$(\beta)\ (\sigma_f)\ (V_f) + (\gamma)\ (\sigma_m)\ (V_m)$
Manual	23.5 MPa	23.5 MPa	(0.5)(149.8)(0.20) + (**0.30**)(35.7)(0.80)
Infusion	34.6 MPa	34.6 MPa	(0.5)(149.8)(0.25) + (**0.59**)(35.7)(0.75)
Compression	46.2 MPa	46.2 MPa	(0.5)(149.8)(0.38) + (**0.80**)(35.7)(0.62)

Table 4: values obtained depending on the manufacturing process adopted (TARGA et al., 2009).

therefore influences the strength and fracture mechanism of the yarn (SHAH et al., 2013). The angle formed between the direction of the twisted fiber and the longitudinal axis of the yarn is defined as the surface twist angle of the yarn (α) and is used as a control parameter in the textile industry. Figure 14 illustrates the surface twist angle of yarn), formed at a distance R from the longitudinal axis of the yarn.

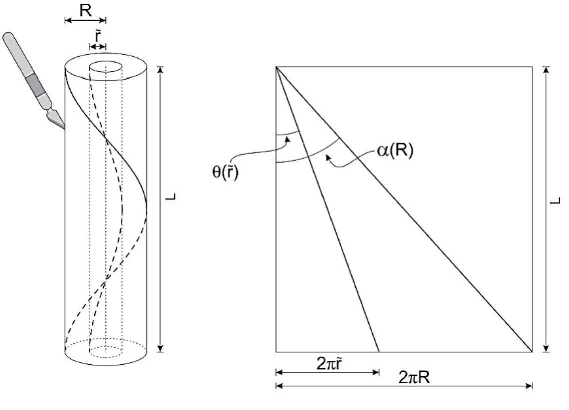


Figure 14: Definition of the surface twist angle of a yarn (RASK, 2012).

The surface twist angle of the yarn represents, for composites, a misalignment of the fibers in relation to the direction of stress, with direct effects on the tensile properties. SHAH et al., (2013) proposed a model to predict the tensile strength of composites reinforced with unidirectional threads, developed from the Modified Mixture Rule, incorporating the angle :

$$\sigma_c = (cos^2\alpha.\eta_l.\eta_d.V_f.\sigma_f + V_m.\sigma_{m*})(1 - V_v)^2 \qquad (6)$$

where $\boldsymbol{\alpha}$ is the surface twist angle of the yarn, $\boldsymbol{\eta_l}$ the efficiency factor of fiber/matrix length and interface; $\boldsymbol{\eta_d}$ the efficiency factor of reinforcement distribution and orientation; $\boldsymbol{V_f}$ and $\boldsymbol{\sigma_f}$ the volume fraction and tensile strength of the fiber; $\boldsymbol{V_m}$ and $\boldsymbol{\sigma_{m*}}$ the volume fraction and tension of the deformed matrix at the point of failure of the composite; $\boldsymbol{V_v}$ the porosity.

FIBER VOLUME FRACTION AND VOID VOLUME

The volumetric fraction of fibers is one of the main parameters in determining the mechanical properties of composites with natural fibers (MESSIRY, 2013). Disregarding the existence of voids, the volumetric fraction of the fibers can be calculated according to the following equation:

$$V_f = \frac{\rho_m . m_f}{(\rho_m . m_f + \rho_f . m_m)} \tag{7}$$

where $\boldsymbol{V_f}$ is the volumetric fraction of the fibers; $\boldsymbol{\rho_m}$ the specific mass of the matrix; $\boldsymbol{m_f}$ the mass of the fibers; $\boldsymbol{\rho_f}$ the specific mass of the fibers and $\boldsymbol{m_m}$ the mass of the matrix.

The tensile properties of aligned fiber composites are predominantly affected by the volume fraction of the fibers (V_f) and, therefore, this parameter has to be determined precisely in order to evaluate the relatively minor effect of other parameters. V_f can be determined directly by image analysis of polished composite cross-sections. However, this requires a number of assumptions to be made to be able to optically distinguish between the fiber, matrix and porosities in the images. The volume fraction of fibers in composites is generally estimated indirectly from gravimetric and volumetric measurements of the composite laminate and its constituents. In a commonly applied method, V_f is calculated from the laminate volume (V_c), the mass of the laminate (m_c), the fiber mass (m_f), and the specific mass of the matrix () (MADSEN, 2004):

$$V_f = \frac{v_c - \frac{m_c - m_f}{\rho_m}}{v_c} \tag{8}$$

The method is quick and simple, as it only requires knowledge of V_c, m_c and m_f, which are easily determined, and ρ_m which is normally determined by the supplier. However, it is based on determining the volume of the matrix, and therefore the estimated V_f includes the porosity content in the laminated composite. Thus, for composites with low V_f (and low V_v) equation (8) is probably acceptable, but for composites with V_f approaching $V_{f\ (max)}$, equation severely overestimates V_f. A more accurate method for estimating the volume fraction of fibers in composites is based on determining the fiber volume:

$$V_f = \frac{\frac{m_f}{\rho_f}}{v_c} \tag{9}$$

This method requires that ρ_f be measured accurately, and it must be assessed whether absolute or apparent fiber density should be applied. The method is suitable if the manufactured laminate is perfectly quadrangular, but if it deviates in this way the determination of V_c becomes inaccurate. Instead, equation (9) can be modified to apply to small laminate samples where the laminate specific gravity (ρ_c) can be determined by the flotation method (MADSEN, 2004):

$$V_f = \frac{\frac{m_f}{\rho_f}}{v_c} = \frac{\frac{m_f}{\rho_f m_c}}{\frac{v_c}{m_c}} \rightarrow V_f = \frac{\rho_c}{\rho_f} W_f \tag{10}$$

Rearranging equation (10), the volumetric fraction of the matrix (V_m) can be calculated and, subsequently, the void fraction (V_v) is determined:

$$V_m = \frac{\rho_c}{\rho_m}(1 - W_f)\ ;\ V_v = 1 - (V_f + V_m) \tag{11}$$

Void spaces and porosity can act as stress concentrators that lead to failure of composite material samples. As void content in natural fiber composites becomes increasingly important, manufacturing techniques, not yet fully developed, and the natural origin of the fibers necessarily induce variations in the mechanical properties of the composites. Both factors contribute to the creation of voids that affect the overall properties of the composites. The void content of composites can be calculated using the following standard formula (DHAKAL et al., 2007):

$$V_v = 1 - \rho_c \left(\frac{W_f}{\rho_f} + \frac{w_m}{\rho_m} \right) \tag{12}$$

where $\boldsymbol{V_v}$ is the volumetric fraction of voids; $\boldsymbol{\rho_c}$ the specific mass of the composite; $\boldsymbol{w_f}$ the fiber mass fraction (%); $\boldsymbol{w_m}$ the mass fraction of the matrix (%); $\boldsymbol{\rho_f}$ the specific mass of the fiber (g/cm^3) and $\boldsymbol{\rho_m}$ the specific mass of the matrix (g/cm^3).

MADSEN et al., (2007) developed a model that quantitatively describes the constituent volumes in a system composed of discrete natural fibers embedded in a continuous matrix. The model is based on the mass and volumetric fractions of the composite constituents. The volume percentages of fibers and matrix are related to the total volume of the composite; however, these variables cannot be established without knowing the quantity of the third part based on volume in the composites, the porosity. Thus, the volume percentages of matrix fibers and porosity are mutually dependent variables, and the fiber weight fraction is used as the independent variable. The authors defined the total pore volume as the total volumetric quantity of air-filled cavities within the composite, dividing this volume into a series of smaller porosity components, assuming that the total pore volume (V_p) can be separated into three main components, as exemplified in Figure 15:

- fiber-correlated porosity, which was assumed to be linearly correlated with absolute fiber volume;
- matrix-correlated porosity, which was assumed to be linearly correlated with absolute matrix volume;
- structural porosity, which is related to the situation in which the available matrix volume is insufficient to fill the free space of a set of fully compacted fibers.

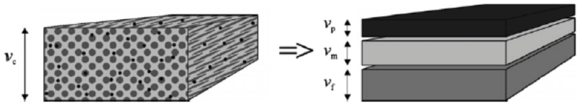


Figure 15: Schematic drawing of the separation of the absolute volume of a composite (V_c) into the absolute volumes of fibers (V_f), matrix (V_m) and porosity (V_p). Source: MADSEN et al., (2007).

An example of a subcomponent of fiber-correlated porosity is the air-filled cavities within fibers (e.g., the lumen in plant fibers). Likewise, an example of a matrix-correlated porosity subcomponent is air bubbles trapped in the matrix. Figure 16 shows a schematic illustration of the absolute volumes constituting a composite with variable volumetric composition. Volumes of material are represented as plates with relative thicknesses in proportion to their volumetric dimensions. The plates corresponding to the fibers (V_f) represent solid volumes of pure fiber. Likewise, the plates corresponding to the matrix (V_m) represent solid volumes of pure matrix. Fiber-correlated porosity (V_{pf}), matrix-correlated porosity (V_{pm}), and structural porosity (V_{ps}) plates represent porosity volumes. The figure exemplifies a typical approach applied to increase the fiber volume fraction in composites, with the fiber volume being kept constant and the matrix volume being continuously reduced. The "x" axis represents the increase in the volume fraction of the fiber. The discrete nature of the fibers means, however, that the fiber assembly can only be compacted to a given minimum volume (under the conditions of the operating process), which, therefore, determines the volume of the minimum composite that can be obtained ($V_{c\ min}$). Therefore, a maximum fiber volume fraction ($V_{f\ max}$) can be defined:

$$V_{f\,max} = \frac{v_f}{v_{c\,min}} \tag{13}$$

Thus, there is a transition process where the set of fibers is fully compacted to its minimum volume, and the amount of matrix is just enough to fill the free space between the fibers. More precisely, this transition case is defined as the situation in which the sum of V_f, V_m, V_{pf} and V_{pm} is equal to $V_{c\ min}$. In Figure 16, the transition case is denoted Case A/B, and specifies the transition between the two cases of volumetric interaction of the composites: Case A and B. In case A, the fiber assembly is not fully compacted, and the amount of matrix is sufficient to fill the free space between the fibers. Thus, in process A, V_c is greater than $V_{c\ min}$. In Case B, the set of fibers is completely compacted, and the amount of matrix is insufficient to fill the free space between the fibers. Consequently, the structural porosity volume (V_{ps}) is created, which represents the remaining free space between the fibers. In case B, V_c is equal to $V_{c\ min}$ (MADSEN et al., 2007).

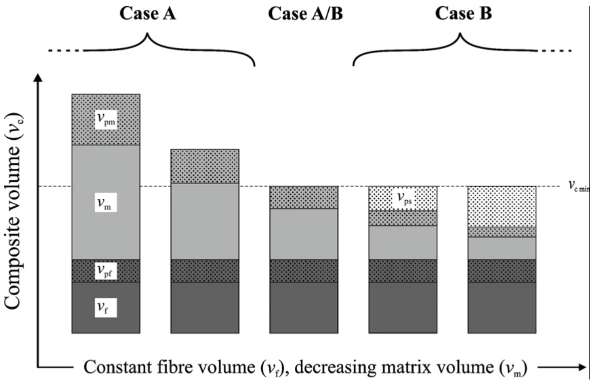


Figure 16: Schematic illustration of the absolute volume of the constituents of a composite with variable volumetric composition. Source: MADSEN et al., (2007).

STATE OF ART

BABU et al., (2019) they carried out a comparative study on the mechanical properties of polyester composites reinforced with Kenaf fiber prepared by VARI, RTM and CM techniques. This study showed that the tensile strength, Young's modulus, flexural strength and flexural modulus of the VARI samples are superior to the RTM and CM samples. The void content and water absorption property of composites prepared by VARI is lower as compared to RTM and CM due to good fiber/matrix interaction. According to the authors, mechanical properties increase with increasing fiber content. In the VARI process, the fiber content can reach up to 70%, but in the RTM process and compression molding it can only reach 40%. In the VARI process, thickness is controlled by compacting the fiber under vacuum pressure.

GOMES et al., (2007) produced biodegradable thermoplastic matrix composites derived from corn starch reinforced by continuous aligned curaua fibers, manufactured by three different manufacturing methods. Initially, the authors produced composites by adding aligned fibers directly into a metallic mold and then introducing the resin, afterwards the mold was closed and the composite was heated to 150°C for one hour with 3.27 MPa of compression, generating composites with a fiber volume fraction of 69.3%. Composites were then produced using preforms of aligned long fibers, previously agglomerated using a small portion of the matrix and dried at 30° C for 24 hours, the manufactured preforms being inserted into the mold together with the resin and the composite compressed under 6.54 MPa of pressure at a temperature of 150° C for one hour, fiber volume fraction of 67%. Finally, the researchers produced test specimens using prepreg technology, manufacturing five sheets of prepregs that were then stacked and compressed at 16.9 MPa at a temperature of

150° C for 1 hour, with a fiber volume fraction of 69.9%. The authors found a 51.4 and 27.3% higher performance in the tensile strength limit of composites manufactured from prepregs and preform, respectively, in relation to those manufactured using the direct method of inserting fibers into the mold. In relation to Young's Modulus, the influence of the fiber configuration when added to the mold was even more sensitive, with gains of 176.9 and 123% of prepregs and preform, respectively, being observed in relation to the direct method.

MEMON and NAKAI (2013) manufactured braided tubular composites made of PLA matrix and jute and fiberglass yarns by pultrusion. The authors explored the various processing variables, such as the braiding angle, arrangement of the wires in the feed, space between the braiding wires, filling ratio, pultrusion temperature, speed and pulling force of the wires, among others, correlating these variables with the quality of the product generated and with the mechanical properties under four-point bending of the composites produced. The results demonstrated the feasibility of producing composites by pultrusion using natural fibers, representing, according to the researchers, an important step towards the economically viable production of high-performance biocomposite products with a uniform cross section.

TARGA et al., (2009) used flat jute fabric weighing 280 g/m² to produce orthophthalic polyester matrix composites, studying the effect of the type of manufacturing process on the properties of the composite. The authors produced composites by manual lamination, lamination followed by compression of 0.5 MPa for 4 hours and vacuum infusion, subjecting all specimens to post-curing at 60° C for 60 minutes. The results showed that the composites manufactured by manual lamination had tensile performance 34% lower than that of the full polyester matrix; composites manufactured by infusion achieved practically the same performance as the matrix in tension; those that underwent compression of 0.5 MPa were 29.4% higher in traction than the polyester matrix.

RODRIGUES et al., (2015) manufactured polymer composites reinforced with natural fibers from the Amazon using the vacuum infusion technique, according to the authors the infusion process proved to be a good method for manufacturing composites reinforced with natural fibers, as, in addition to being more environmentally favorable due to being a closed mold process, it enabled the manufacture of products with a high fiber content and good mechanical properties. The use of the infusion process allowed, in the best case, the production of curaua-reinforced composites with up to 38% fiber volume fraction with a porosity level below 3%; jute-reinforced composites with up to 32% fiber volume fraction with a porosity level below 5% and raffia-reinforced composites with up to 45% fiber volume fraction with a porosity level just above 2%. Furthermore, the tensile mechanical performance of composites reinforced with aligned curaua fibers increased by 22.2% and the longitudinal elastic modulus by 48.6% with the increase in the vacuum level from 53.3 to 101.3 kPa mainly due to the increase the efficiency of the fiber/matrix interface. For the highest vacuum levels, composites reinforced with aligned jute threads, due to the high diameter of the threads, and mainly the surface twist of the fibers, showed a reduction in their tensile strength and stiffness limit, due to the difficulty in impregnating the yarn with polyester, thus reducing the mechanical anchoring responsible for transferring load from the matrix to the reinforcement. Finally, the composites reinforced with raffia fibers presented the lowest performance among all those manufactured, due to the lower fiber resistance in relation to the others and the weak fiber-matrix interface observed.

CORRÊA et al., (2021) analyzed the performance of the vacuum infusion process (VIP) in the manufacture of jute yarn and polyester resin composites in comparison to the manual lamination process (hand lay-up), considering multiple attributes with support from the Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS). According to the authors, the results indicated that the vacuum infusion process is the best alternative for the production of composites with jute yarn when compared to hand lay-up, taking into account the attributes and weights used. TOPSIS demonstrated to be simple and capable of easily considering a good number of quantitative and qualitative attributes, using basic computational resources; therefore, constituting a promising support tool in the selection of material processes. Finally, the use of the Saaty scale was effective in generating a score when qualitative data collected from experts' opinions were used, enabling the integration of quantitative and qualitative variables in the objective assessment with multiple attributes of competing processes.

RIBEIRO et al., (2022) made a comparison of the Young's modulus of polyester composites reinforced with continuous and aligned lignocellulosic fibers of jute and mallow determined experimentally and from theoretical prediction models, this research showed that using the vacuum-assisted hand lay-up/vacuum-bagging method is a highly efficient way to produce composites with a low void volume fraction. The Madsen's model was the most accurate in predicting the experimental results; the Young's modulus varied by 7, 13, and 19% in jute fiber composites with 5, 15, and 25 vol%, and by 4, 3, and 28% in mallow fiber composites. The ROM, Al-Quresh, Halpin-Tsai, and Nielsen prediction models showed similar results for Young's modulus.

In another study, RODRIGUES et al., (2023) investigated the unsaturated permeability of amazon natural fibers fabrics, according to the authors the permeability of curaua, jute, and raffia fibers is greatly affected by how the fibers are arranged and the level of vacuum applied, as well as changes in fiber compaction and resulting fabric and yarn porosity. The curaua fabric had a slightly lower permeability compared to the aligned fibers at the same compaction level. Curaua fibers treated with a 5%, 20%, and 30% NaOH solution exhibited permeability levels 16%, 36%, and 48% higher than the natural fibers. As the compaction pressure of the fibers increased, the permeability of the jute fiber in aligned yarns and flat weave decreased, as anticipated. Aligned fibers experienced a 38% increase in permeability when exposed to a vacuum of 53.3 kPa compared to 101.3 kPa. Fibers exposed to a vacuum of 13.3 kPa (1.65×10^{-8} m^2) had over four times the permeability of fibers exposed to 101.3 kPa (3.88×10^{-9} m^2), with porosities of 0.82 and 0.77 respectively. Finally, the decrease in permeability of raffia fiber was observed as the vacuum level increased during the permeability test. This change was more noticeable for aligned fibers compared to fibers in a flat fabric when the vacuum level rose from 53.3 kPa to 101.3 kPa.

CONCLUSIONS

The study of plant fibers is the branch of science dedicated to investigating and analyzing lignocellulosic structures. These natural materials constitute a source of renewable, abundant resources and relatively low-cost production. Its characteristics and properties such as biodegradability, low abrasiveness, weather resistance, among others, make this material a technologically promising alternative while at the same time showing care and respect for the environment.

It can therefore be concluded that the resin infusion process, in addition to being an environmentally friendly process, is a very attractive process with regard to most of the properties of the composites obtained by this process. The resin infusion process can also be improved to obtain parts with greater thickness and even better quality. In this development of the technique, new experiments can be carried out, such as: inclusion of profiles, sandwich manufacturing, manufacturing of components with more complex shapes, etc.

At last, vacuum infusion is able to build faster, with more strength and less weight than manual lamination. At first, it may seem like a complex process with many variables, but after mastering the process, the amateur or professional builder will be able to manufacture much more efficiently.

ACKNOWLEDGEMENTS

The authors thank support to this investigation by the Brazilian agencies: CNPq, CAPES and FAPERJ. This study was financed in part by the Coordination of Superior Level Staff Improvement – Brasil (CAPES) – Number code: 88887.672196/2022-00.

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