

WHITE PAPER



LIQUID MOULDING PROCESSES IN COMPOSITES MANUFACTURING

The use of lightweight components stands or falls on the choice of materials. Product value, product costs, production costs, development costs and risks are, however, difficult to estimate when dealing with lesser known materials such as composites. Moreover, the wide range of materials and processes makes the selection even more difficult.

This is why SLC-Lab, as well as its partners in the **CompositeBoost** project want to pass on essential tools and methodologies to help designers and OEMs to make the right choices. We want to give support by providing more clarity, and this by publishing various white papers that deal with current issues. This eighth white paper focusses on liquid moulding.

Many thermoset composite parts are produced by a type of liquid moulding (LM) process. In general terms, liquid moulding involves placing a fibrous reinforcement into the cavity of a mould, after which it is impregnated with resin under the influence of a pressure difference between the mould cavity and the resin supply. The resin is then left to harden (cure) before the part can be removed from the mould.

Many variants of LM processes exist. A distinction can be made between closed and open-mould processes. In open mould processes, most of the material in the mould is exposed to the environment. Examples of open mould processes are hand-layup and spray-layup. In closed-mould processes the material is sealed off. Vacuum infusion and RTM are examples of closed-mould processes.

Processes that are somewhere between closed and open-mould processes also exist. In compression RTM, for example, the resin is poured over the reinforcement in the mould before closing the mould, or is injected in a partially closed mould. The closing force on the mould then provides the necessary pressure difference for impregnation.

Closed-mould processes offer better and more consistent material properties for the final product and provide a cleaner/less hazardous working environment. As such, closed-mould processes increasingly replace open-mould processes. This whitepaper focusses on closed-mould processes.

Traditional LM processes inject the liquid resin into the mould through one or several injection points. Air from the cavity can escape through vents in the mould or is actively removed by applying a vacuum. Depending on the magnitude of the injection pressures, different names are attributed to these processes, as will be explained later.

This paper provides an overview of the most used LM processes for thermoset composites and discusses the main parameters that are of importance in this type of process.

OVERVIEW OF LM PROCESSES

Although all LM processes are based on the same principle, there are many variants with each their own characteristics and applications. The differences between the various processes are mainly based on these factors:

- Rigid/flexible/semi-flexible tool
- High/low/no pressure
- Vacuum/no vacuum
- Injection strategy: location and type of injection points

Other differences can be linked to the use of different consumables, distribution media, degree of automation etc.

The rigidity of the top mould has an important effect on the resin injection process. In this whitepaper we will consider the family of RTM processes, with a rigid top mould, and the family of VARI processes, with a flexible top mould, consisting of only a plastic film. Intermediate processes have also been developed, for example the light-RTM process (LRTM), where the top mould consists of a thin, semi-rigid shell.

RTM processes

RTM, or Resin Transfer Moulding (figure 1), is an LM process in which a dry reinforcement is placed in a rigid bottom mould (figure 2) . The (also rigid) top mould is then closed. Resin is injected under a mild pressure (typically less than 10 bar) in the mould cavity through one or more injection points. Air is removed from the cavity by the incorporation of strategically placed 'vents' in the mould. After proper curing, the product can be released from the mould. (figure 3)

A well-known RTM variant is VARTM, or Vacuum Assisted RTM. In this process, vacuum is applied on the mould cavity which speeds up the infusion process and allows for the removal of trapped air in the resin.

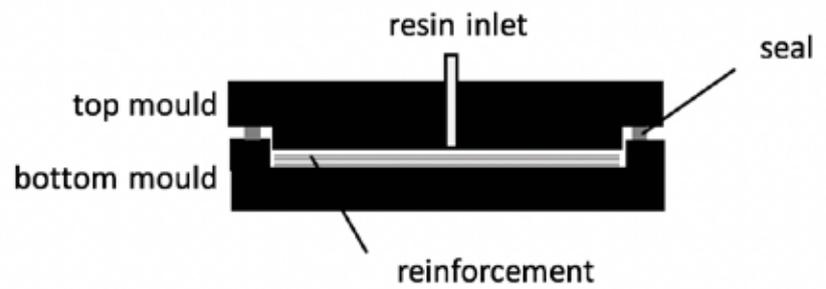


Figure 1: Illustration of the RTM process.

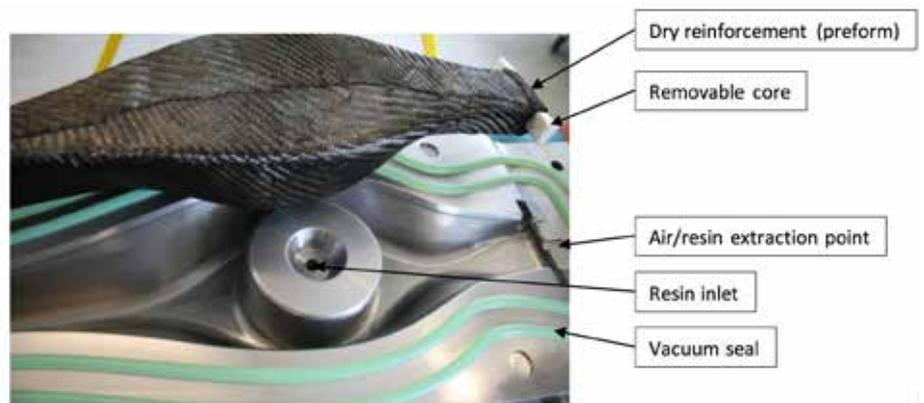


Figure 2: The dry reinforcement for a carbon fibre part with the bottom half of the RTM mould.



Figure 3: Separating the two mould halves after injection, exposing the semi-finished product. Note the tubing used for resin transport.

An advantage of using two rigid moulds is that the thickness and thus the fibre volume fraction in the composite can be controlled quite well, and that there is little variation in this property throughout the part. As the permeability of technical textiles is often relatively low and the viscosity of the thermoset can sometimes be high (for instance for fire resistance purposes), injection times can be long, resulting in even longer cycle times.

For short cycle times in the range of minutes, high injection pressures (over 100 bar) are needed, and these processes are often referred to as Structural Resin Injection Moulding (SRIM) or High-Pressure RTM (HPRTM). HPRTM process equipment is quite heavy and thick steel moulds are usually used, as part of a fully automated process. More information on tooling can be found in our whitepaper “Tooling methods and materials for composite tooling”¹.

Several other variants of the RTM process have been developed over the years, including Same Qualified Resin Transfer Moulding (SQRTM) and Compression Resin Transfer Moulding (CRTM). In the former process, liquid resin is injected over a prepreg lay-up. In the latter, liquid resin is injected into a (partially) open mould, followed by a compression stroke.



In Light RTM, one rigid and one semi-rigid mould are used. The two mould halves are typically held together by a vacuum. Resin infiltration is also done by applying vacuum. Sometimes a very low additional injection pressure (less than 1 bar) is used. Because of the lower pressures involved in LRTM, the requirements for the moulds rigidity are less, and thus other materials such as wood or composite moulds may be used. The semi-rigid top mould is usually a relatively thin composite shell. Because of the lower pressure gradient, the cost of the tooling is dramatically lower than for RTM or VARTM, but the LRTM

1. Accessible from: <https://www.sirris.be/sites/default/files/content/composites-whitepaper-7-tooling-methods.pdf>

process is slower. To decrease the injection time, different injection strategies are used for LRTM.

VARI processes

In Vacuum Assisted Resin Infusion (figure 4), the top mould is replaced by a flexible vacuum bag. Of course no pressure can be used in the injection, because this would inflate the vacuum bag. In VARI, the driving force for infusion is only vacuum. Therefore the tooling cost can be kept very low, yet at the expense of consumables (seals, vacuum bag ...). To keep infusion times within reasonable limits, the flow distance should be short, and a large cross-section for flow is advisable. For very large structures, resin distribution gates must be used to shorten the flow distance. This can for example be done by using spiral tubes (figure 5). Flow distribution media can also be used. These open-structure textiles allow quick and easy distribution of the resin over the surface of the reinforcement, after which the resin can infiltrate the reinforcement in the through-thickness direction, thereby greatly shortening the flow distance. A VARI process which relies on such transverse flow is the Seemann Composites Resin Infusion Moulding Process (SCRIMP). Other variants of VARI exist, for example the Vacuum Induced Preform Relaxation process (VIPR). In this process, local vacuum suction is applied on the vacuum bag, to locally increase the cross-section and as such facilitate the flow.

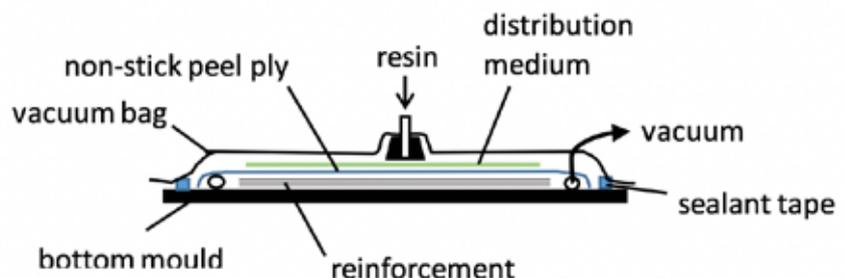


Figure 4: Illustration of a VARI process with the use of a distribution medium

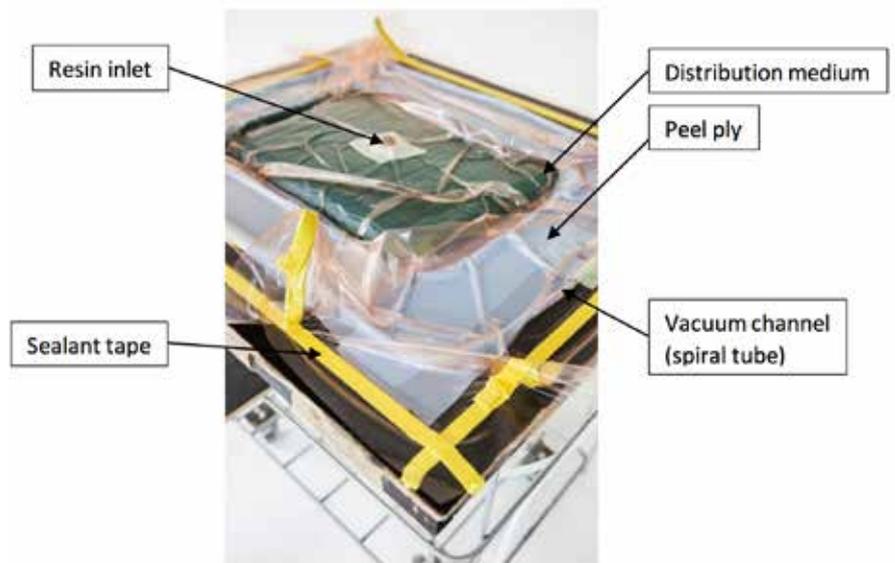


Figure 5: A mould prepared for a VARI process.



Figure 6: Vacuum infusion of a (part of a) ship's hull. The resin "front" is visible as the line between the dark and light green of the resin distribution medium.

Because only vacuum is used in VARI processes, the cost of the tooling can be kept very low. This makes these processes ideally suited for small series or prototypes. On the other hand, because of the flexibility of the vacuum bag, it is very difficult to control the fibre

volume fraction, and local variation in volume fraction and wrinkles can occur. Also, the usage of consumables is high: seals and distribution media can be used only once. Both consumable and reusable vacuum bags are available. Closed Cavity Bag Moulding (CCBM) and Silicone Bag Moulding, for example, use reusable, flexible silicone top moulds. Table 1 shows a comparison of a number of different LM processes in terms of some important process characteristics. In general, it can be said that the higher the process pressure, the higher the investment cost, part accuracy, fibre volume fraction and surface finish can be, and the lower the infusion time and achievable part size.

PROPERTY	RTM	HPRTM	LRTM	VARI
Investment cost	● ● ●	● ● ● ● ●	● ● ●	●
Labour cost	● ●	● ● ● ● ●	● ●	●
Part accuracy	● ● ●	● ● ● ● ●	● ●	●
Part size	●	●	● ●	● ● ●
Infusion time	●	● ● ● ● ●	● ● ●	● ● ●
Surface finish	● ● ●	● ● ● ● ●	● ●	●
Achievable fibre volume fraction	● ● ●	● ● ● ● ●	● ●	● ●

Table 1: comparison between RTM, HPRTM, LRTM and VARI. A higher number of markers indicates a higher value for the considered property.

PARAMETERS IN LIQUID MOULDING



Liquid moulding of composites involves infiltration of a liquid resin into a fabric reinforcement. As stated before, this whitepaper focusses on thermoset resins. Thermoset resins undergo an irreversible chemical reaction (curing reaction) in which polymers are cross-linked to form a hard product. This is an exothermal process, in which energy is produced as heat. The rise in temperature speeds up the cross-linking process until no more components are available to react. After this, the reaction slows down considerably, again lowering the temperature.

During the reaction, some stages can be distinguished that are of particular interest for the LM process. As an illustration, figure 7 shows the temperature as a function of time for a vinyl-ester resin (without post-curing).

1. Constant temperature, no change in liquid properties. This time can be extended using additives (inhibitors);
2. Initial rise in temperature of the liquid reducing the resin viscosity, leading to an increase in flow speed;
3. Gel-time at which the liquid turns in a (non-flowing) gel. At this time, the resin flow stops. The resin injection must be complete before the gel-time;
4. Further rise in temperature up to peak-exotherm (max. temperature);
5. Cooling down until the polymerisation reaction stops;
6. (If required) post-curing at elevated temperature (e.g. in an oven) allowing further cross-linking to improve the mechanical and chemical properties.

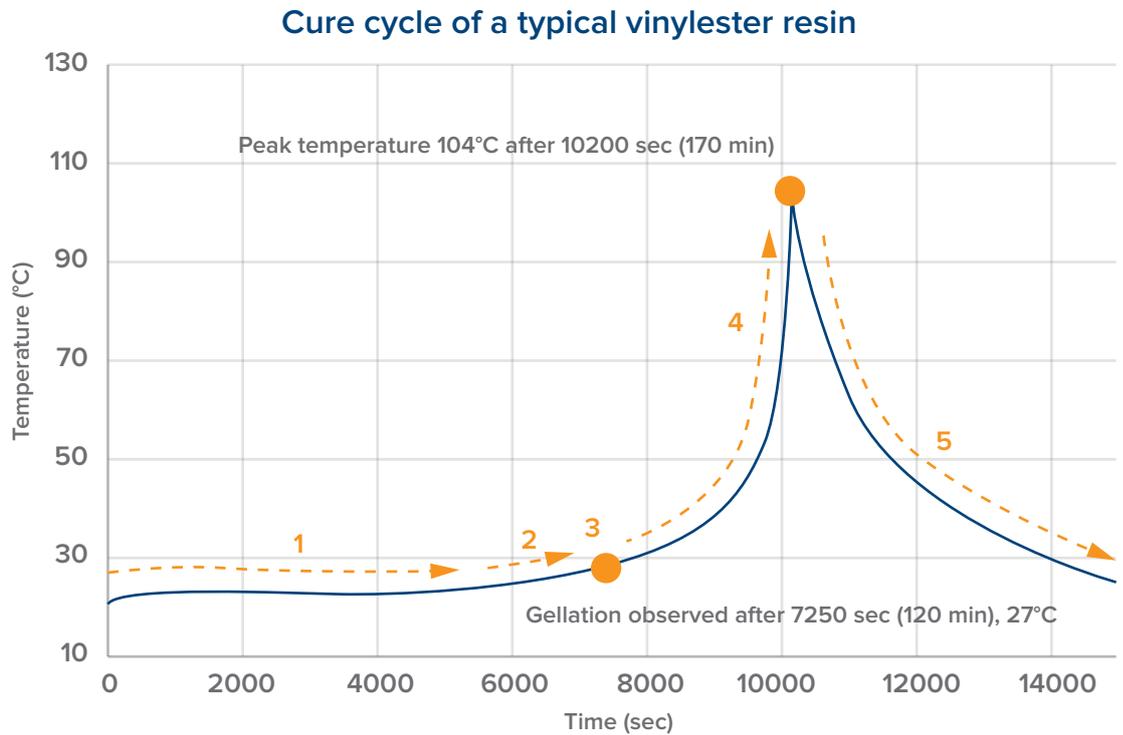


Figure 7: Cure cycle of a vinyl-ester resin, without post-curing.

Although there is a large variety in thermoset resins, polyesters, vinyl esters and epoxies are most commonly used. Polyesters and vinyl-ester resins contain all components involved in the reaction (a polymer dissolved in a monomer), and will cure over time if left on their own. To control the curing, in most cases a catalyst (typically a peroxide) is added just before the LM process. By varying the amount of catalyst and/or other additives, the reaction can be controlled (e.g. gel-time, peak exotherm). Epoxies on the other hand, require a separately stored hardener which is mixed with the polymer to start the cure. The resin/hardener ratio is fixed and must be closely controlled in order to obtain full curing. Different types of hardeners provide the possibility to control reaction speed and exotherm, but once mixed, there is little to no room to control the reaction chemically.

Darcy's law

Resin injections in a fibrous preform can mathematically be described² by Darcy's law, which is – for a one-dimensional case – expressed by the following equation:

$$u = \frac{Q}{A} = -\frac{K}{\mu} \frac{dP}{dx}$$

Where:

u: fluid velocity [m/s]

Q: flow rate [m³/s]

A: cross sectional area of the cavity [m²]

K: permeability of the porous medium [m²]

μ: resin viscosity [Pa s]

dP/dx: pressure gradient [Pa/m]

For two- and three-dimensional flow, the permeability cannot be described by a single parameter because of the anisotropic nature of most fibrous preforms. However, having a good understanding of Darcy's law in the one-dimensional case will be beneficial in optimising the process parameters for LM. Specialised software such as RTM-Worx and PAM-RTM can be used to simulate complex LM processes. The Darcy equation indicates the important material and equipment parameters that govern LM processes. They are briefly discussed below. There are many books available explaining the matter in more detail.³

2. For more accurate descriptions of the impregnation process (for the sake of process modelling and simulation), more complex models are needed.

3. For example: Manufacturing techniques for polymer matrix composites (PMCs), ed. S. Advani and K. Hsiao, Woodhead publishing limited, 2012

Permeability

Permeability can be understood as the openness of a porous medium, related to its porosity and the way these pores are interconnected so a fluid can pass through it. The unit of permeability is m^2 . In liquid moulding of composites, this medium is usually a fibrous reinforcement (a textile). The type and parameters of the reinforcement will determine the textile's permeability.

A higher permeability is beneficial from a processing point of view, because it will allow for a higher flow rate (i.e. faster mould filling and curing) and/or a lower pressure (i.e. lighter moulds, cheaper injection pumps). However, the permeability is strongly dependent of the fibre volume fraction of the preform. A higher fibre volume fraction implies a more compact preform and hence a lower permeability. Therefore, a compromise must be sought between the mechanical performance of the composite (directly proportional to the fibre volume fraction) and the ease with which the component is manufactured.

Most fabrics will have an anisotropic permeability, i.e. the permeability depends on the direction of the flow with respect to the fabric main axes (figure 8). In some fabrics, for example in woven fabrics and non-crimp fabrics, continuous 'channels' exist between the bundles of fibres. Resin can flow readily through those channels and from there spread out to impregnate the fibre bundles. Resin flow perpendicular to the fibre bundles is much slower, as the resin constantly has to deviate from the straight path. Hence, the transverse permeability is often lower than the longitudinal permeability. Consequently, the presence of oriented fibre bundles shortens the impregnation length and as such, the presence of these channels greatly improves the textile's permeability.

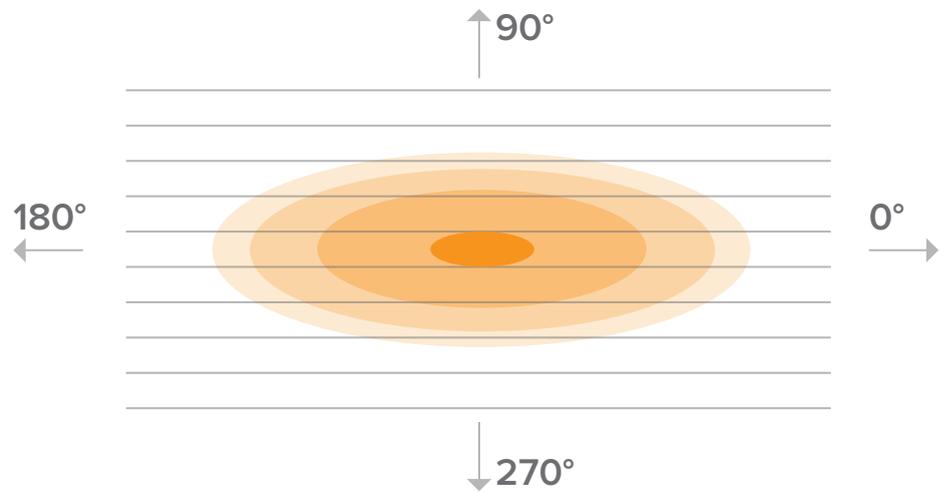


Figure 8: Resin flow visualised as function of fibre orientation for a unidirectional continuous fibre preform. The permeability is highest in the direction of the fibre bundles (0°).

The permeability is a characteristic of the fibrous preform. It can only be changed by changing the preform, i.e. by changing fibre orientations or the fibre volume fraction. Since the fibrous preform composition is defined by the part requirements, such changes inevitably affect part performance. If the permeability needs to be increased, this requires a reduction in fibre volume fraction, which means that for the same load bearing capacity, the part thickness has to be increased, resulting in a weight and cost increase of the part. For VARI processes, this means lowering the pressure gradient and thus the compaction force on the preform.

An alternative can be offered by using flow media, either internally in the part, or externally on the surface. External flow media are highly permeable non-stick meshes that can be placed on top of the dry reinforcement. During injection, the resin will spread easily through the channels in the mesh and from there will impregnate the reinforcement through-the-thickness. After curing, the flow medium can be removed from the laminate. Internal flow media, for example Rovicore™ or

Multimat[®], are placed inside of the preform. Resin can flow easily in this material and from there it can impregnate the rest of the preform through-the-thickness. Internal flow media cannot be removed after infusion and will remain an integral part of the composite. If core materials are used (foam, balsa ...), flow can be promoted by grooves in the core material. Many suppliers offer grooved variants of their foam core materials.

The use of flow media can reduce the impregnation time significantly and offer a solution for highly dense, difficult to impregnate textiles.

Viscosity

Viscosity, or the ease of flow of the resin, is of primordial importance in liquid moulding processes. If the viscosity of the resin is high, Darcy's law shows that excessive pressure gradients may be needed to achieve complete mould filling.

As a rule of thumb, the viscosity of a resin should be below about 1 Pa.s to make liquid moulding feasible without excessively long impregnation times or excessively high pressure gradients. For most thermoset polymers in uncured condition, this rule is satisfied. The limitation in viscosity is a challenge when fire resistance is required of the composite. The restrictions on the use of halogenated fire retardants necessitates the use of solid inorganic fillers such as ATH (aluminium trihydroxide). Not only will these fillers significantly increase the resin viscosity, if the filler particles are too large, they are likely to be filtered out by the fibrous preform, resulting in a drop in fire retardant properties as a function of the distance to the injection point.

Pressure gradient

The pressure gradient is the driving force in the liquid moulding process: under the influence of this gradient, resin will flow from the high pressure region (the inlet) to the low pressure region (the outlet). In a very simplified approach, the pressure gradient can be represented as the pressure difference between inlet and outlet, over the distance between inlet and flow front.

For the simplified, one-dimensional case of a mould with a constant-thickness cavity and constant permeability, the pressure drops linearly between resin inlet and resin front. When the resin front progresses, the pressure gradient decreases, slowing down the resin flow (see figure 3). For this idealised case, the travel time is quadratic to the travel distance.

The travel distance or the distance the resin needs to flow through the mould cavity depends on the part size, but also on the injection strategy (location and number of injection ports). If the pressure difference is limited (for instance because of mould restrictions) or if the resin viscosity is too high, the travel time can become too high, causing the resin to cure before the mould cavity is fully filled. In these cases, an injection strategy must be chosen that minimises the flow distance.

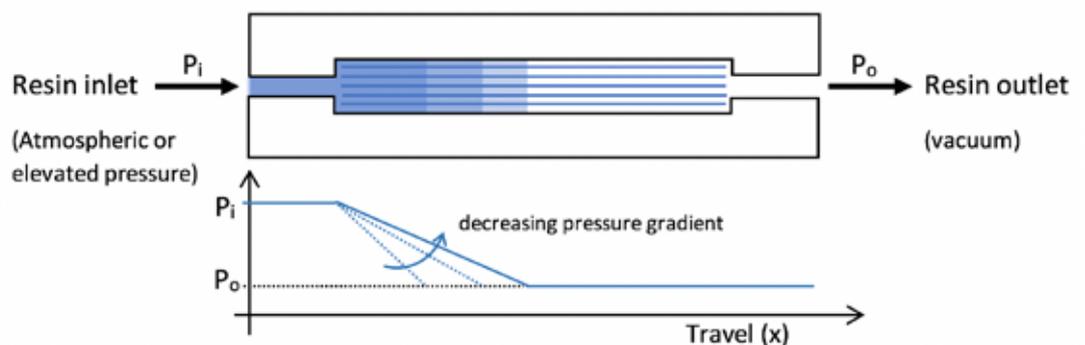


Figure 9: evolution of the pressure gradient during flow for a one-dimensional case of a mould with a constant-thickness cavity and constant permeability. The resin inlet and inlet channels are assumed frictionless, without pressure loss.

Typical pressures in LM can vary greatly from 1 bar or less in RTM light or VARI processes to about 10 bar in standard RTM up to over 100 bar in HPRTM.

Increasing the inlet pressure to above the atmospheric pressure means that rigid moulds and clamping equipment are needed, for example to hold the mould closed and to avoid the mould from bending under the injection pressure (be aware that 1 mm bending of the mould implies an increase of the part thickness of 33% for a 3 mm thick part, significantly increasing resin consumption, part weight and induces the risk of thermal distortions and even thermal cracking). If the pressure becomes too high, there is also a risk of distorting the reinforcement inside the mould cavity.

Non-rigid moulds will deform due to the pressure difference between both sides of the mould. This is especially true for VARI processes utilizing a flexible vacuum bag. In this case, the inlet pressure is usually almost equal to the atmospheric pressure (if the resin reservoir level is assumed to be at the same height as the mould cavity). In this case, no compacting force is exerted on the fibrous preform at the inlet. The compacting force will increase towards the resin flow front. A higher compacting force increases the fibre volume fraction of the preform and thus the thickness. The pressure over the resin must therefore be equalized before curing if a constant thickness is required.

For LM processes with rigid moulds, such as (HP)RTM, the thickness of the preform and thus the fibre volume fraction, is constrained by the mould.

Temperature

An important parameter in LM, not explicitly visible in Darcy's law, is the temperature. A higher mould and/or inlet resin temperature will enable an easier flow, since it will lower the viscosity of the resin. On the other hand, if the temperature becomes too high, the resin will

cure faster and there is a risk of not filling the mould completely before the resin reaches its gel-point. In some cases, active cooling can be used to slow down the curing reaction.

As a rule of thumb, the reaction can be assumed to go twice as fast with each 10 degrees Celsius increase of temperature (Arrhenius' law).

An important point to keep in mind is the ambient temperature in the workshop: if it is not controlled, this may lead to significantly different filling times between summer and winter. Therefore, a temperature control for the mould and the resin is highly recommended in order to have a robust process.

Another aspect is the influence of material thickness. Thicker laminates or areas of resin build-up generate more heat, potentially resulting in an inhomogeneous temperature throughout the mould. This may result in areas where the resin gels prematurely, preventing some areas to be fully impregnated with resin.

The materials (moulds, cores, etc.) surrounding the resin can have a large effect on the possibility to transfer heat.

CONCLUSION

Liquid moulding of composites encompasses a wide range of composite production techniques involving the closed-mould infusion of a dry reinforcement with a resin. Important parameters governing these processes are pressure, viscosity of the resin, permeability of the reinforcement, and temperature. Depending on the series size and cost considerations, different techniques are opportune, ranging from vacuum bag moulding in the VARI process for small series, up to the use of heavy metal moulds in the RTM process.

This white paper was written within the scope of the project “CompositeBoost”.

COMPOSITEBOOST

‘CompositeBoost’ is a collaborative project involving the Sirris SLC-Lab, UGent and KU Leuven. Based on six highly relevant issues, the project partners want to use these essential tools and methodologies to allow designers and OEMs to make the right choices. Masterclasses, demonstrations and exploratory case studies will help transform the composites processor into a reliable production company and partner. This means that our companies will retain their competitiveness over foreign competitors.



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