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Experimental RTM Manufacturing Analysis of Carbon/Epoxy Composites for Aerospace Application: Non-crimp and Woven Fabric Differences

Thatiane Brocks*, Marcos Yutaka Shiino, Maria Odila Hilário Cioffi,

Herman Jacobus Cornelis Voorwald, Angelo Caporalli Filho

Fatigue and Aeronautic Materials Research Group, Universidade Estadual Paulista – UNESP, 333, Dr. Ariberto Pereira da Cunha Ave., CEP 12516-410, Guaratinguetá, SP, Brazil

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The success of manufacturing composite parts by liquid composite molding processes with RTM depends on tool designs, efficient heat system, a controlled injection pressure, a stabilized vacuum system, besides of a suitable study of the preform lay-up and the resin system choice. This paper reports how to assemble a RTM system in a laboratory scale by specifying heat, injection and vacuum system. The design and mold material were outlined by pointing out its advantages and disadvantages. Four different carbon fiber fabrics were used for testing the RTM system. The injection pressure was analyzed regarding fiber volume content, preform compression and permeability, showing how these factors can affect the process parameters. The glass transition temperature (Tg) around 203 °C matched with the aimed temperature of the mold which ensured good distribution of the heat throughout the upper and lower mold length. The void volume fraction in a range of 2% confirmed the appropriate RTM system and parameters choice.

Keywords: *Resin Transfer Moulding (RTM), woven fabric, non-crimp fabric, process monitoring*

1. Introduction

The RTM process is one in a range of liquid composite molding family (LCM) technology that succeeds in producing high quality laminates from dry preforms and usually injecting a low viscosity thermoset resin. RTM basically employs high pressure, low vacuum, and a rigid mold, which is required to hold the pressure, and consequently to guarantee the final geometry[1-4]. Different from the Vacuum Resin Transfer Molding (VARTM), a cheaper process, RTM has especially been employed when high fiber volume fraction is aimed and good quality of both sides of the parts are required[3].

Some new RTM mold concepts are available such as multiple injection ports, that can be switched on/off according to the manufacturing needs as large production demands or project complexity[5].

Regardless of the good mold project, the pressure control accuracy is a key requirement in RTM injection/vent gates success[5]. The effect of vacuum on vent gates had already been proven to enhance composite mechanical properties by removing all remained air from preform and assisting the resin flow front[6]. This is an important parameter to obtain high quality composites amongst more fifteen listed by Babu et al.[7]. Moreover, the author mentioned the decision of the parameter, which is extremely dependent on the materials to be processed, as well as the mold features. By employing Darcy's law, it is possible to observe a decreasing flow front speed due to the pressure gradient, which is also noticed the importance of correctly allocating the inlet injection ports[8].

The process is extremely dependent upon the resin characteristics whether it is preheated or not, and it is consequently affected by the mold temperature control[9]. Lebrun et al.[10] reported a significant variation in the through-to-thickness temperature profile, when resin is injected at a different temperature from the mold, thus changing the viscosity and the flow front path.

Regarding the resin viscosity, it has been reported different strategy to ease the wetting out of the fiber as increasing the mold temperature in relation to the resin, which has its viscosity decreased, and therefore its infiltration between the fibers tows improved. Hence a combination of transport equation, rheological model and chemical kinetics can aid in building an efficient injection cycle when a non-isothermal process is involved[11].

Localized resin flow front deviations, known as race-tracking, can be introduced by the preform placement, fiber nesting, tow waviness, fiber misalignment, among others, they are basically associated with local permeability variations[12].

A lot of advancement has been reported about flow front prediction, the majority of them based on Darcy's law, which correlates the pressure field with resin velocity within a porous medium. The fabric orientation may affect the permeability characteristics, which can be an isotropic or a non-isotropic form[10]. In regard to this feature, Shiino et al.[13] have already discussed the influence of the mechanical compaction of the preform on the flow behavior and the consequent mechanical properties of non-crimp fabric

*e-mail: thati_bd@yahoo.com.br

(NCF), being most deleterious, however comparisons with woven fabrics have not been done yet.

Authors have been reporting that a perfect match between preform and mold edge is a key issue to avoid errors in the flow front position, e.g. race-tracking, that in the pre-heated resin system can induce entrapped air[3,10]. Preform deformation related issues due to external loads and intrinsic fluid pressure have been the subject of study during the development of infusion process[8].

In relation to defects formation during the injection process such as voids, the mechanisms of generation can change that depends on the fabric type and orientation. One of them is the fingering process, which takes place due to the difference between the fiber tows and the tow gap permeabilities, leading to trapped air formation. Even after a long time of resin bleeding process, some of inconvenient entrapped voids remain in the laminate[14]. In the same work, the authors numerically described this process of void formation and confirmed it experimentally in woven laminate, however in a low fiber volume content ($K > 1 \times 10^{-11}$ m^2).

The RTM manufacturing process has been widely employed since the 70's, however an update of the state-of-art in the experimental procedure has hardly seen in research reports of specialized Journals. Thus this research work aimed to show the progression in this field so far by describing the assemblage of a complete laboratory RTM system, outlining the progression of events (e.g. injection temperature, applied pressure, and so forth) by the acquisition system connected to the mold. It was highlighted the mean differences in the flow front behavior as well as the laminate final characteristics, e.g. void volume fraction, between a woven and a NCF preform.

2. RTM system set up

For a better evaluation of the RTM process a flat laminate was aimed. For this purpose a linear resin flow front was conducted between an inlet chamber and an outlet gate, where a vacuum is usually applied. It was outlined the requirements to define material and mold project as well as heat and vacuum system as described below.

2.1. *Mold requirements*

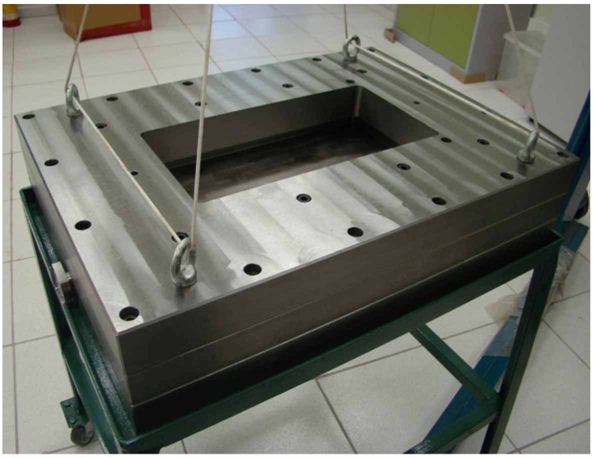
The material stiffness and its thermal expansion are the main issues to be taken into account for a precise laminate production. A low carbon steel was employed due to its low coefficient of thermal expansion (CTE = 1.2×10^{-5} °C^{-1}) compared to the aluminum alloy (CTE ≈ 2.3×10^{-5} °C^{-1}). This is an important project decision considering the high temperatures of the injection and cure cycles can reach that lead to a mismatch between carbon fibers (CTE ≈ 0.08×10^{-5} °C^{-1}) and a metal mold[15,16].

Figure 1a shows the complete mold. Each mold part has a thickness of approximately 50 mm, sufficient to hold a pressure of 10 bar without any deformation. This pressure is an estimated value accounted for fiber compaction and injection cycle step of a high fiber volume fraction composites.

Besides the concern about the sufficient stiffness of the mold cavity, the project was designed to accommodate a heat system on upper and lower mold parts. These will be described in another section.

The middle mold part is a frame that has the function of keeping a laminate thickness of 3 mm (Figure 1b), which fits into the indenter of the upper part, where the resistance is accommodated as well as in the lower part.

Figure 2 details the mold cavity that was rectified in order to produce a flat and polished surface. The chamber (Figure 2a) was projected to promote a linear flow front, so after filling this chamber, the resin runs through the preform (Figure 2b). The mold was also entirely sealed (Figure 2c) and machined in the upper and middle part with 10 degrees in relation to a vertical line for easing the laminate release. The mold parts were clamped with 3/8" screws distributed around the channel. A press with 10 kN capacity could replace the screws.


(a)

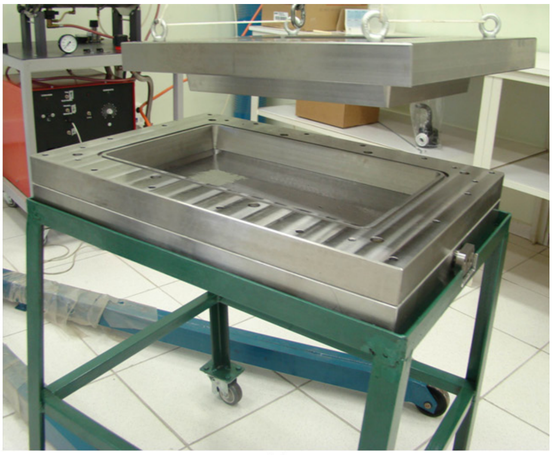

(b)

Figure 1. (a) Complete mold and (b) middle mold part detail.

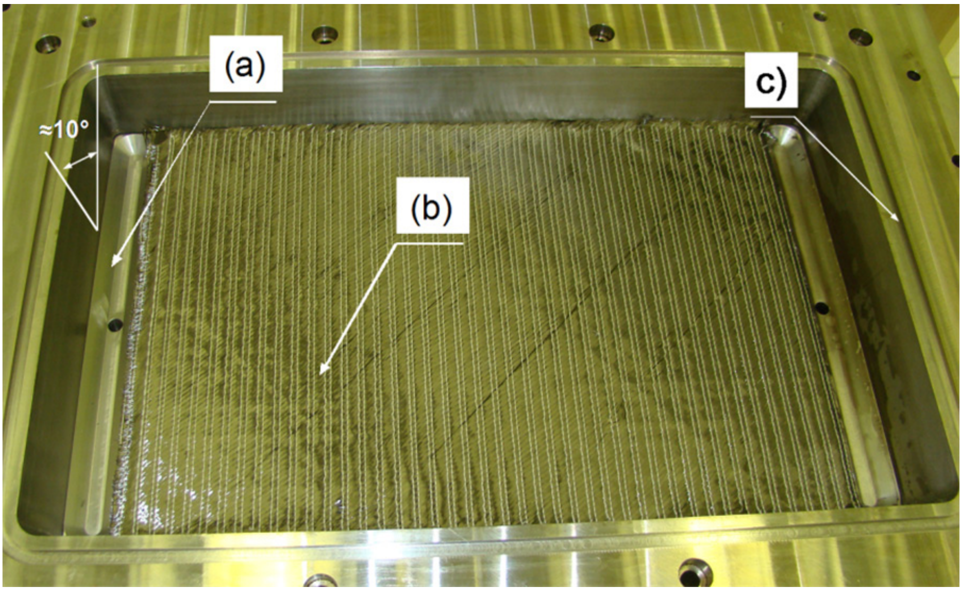


Figure 2. Female tool mold details: (a) line inlet gate; (b) carbon fiber preform; (c) sealing channel.

2.2. *Heated system for a isothermal process*

An important issue in the resin injection procedure is a controlled temperature, mainly when thermally activated resins are involved. In these cases, the resin viscosity is the first parameter influenced by temperature, justifying the need of a good control[17].

The resin viscosity affects the fabric impregnation, and injection pressure, that can lead to some defects like fiber wash-out, preform misalignment and race-tracking[18]. An inappropriate temperature control could contribute to induce undesirable defects as voids. If there is a temperature gradient, a viscosity gradient will take place too, which generates a difference on resin flow velocity along the preform and favors the air entrapment in along the thickness[10.]

Another advantage of the controlled heated system is the possibility of reducing the cure cycle through cure acceleration, which is a great industrial interest [10].

A closed system (Figure 3) was developed in order to provide heat. The continuous resistance with 5 mm diameter is placed between two thin aluminum plates, which distribute the temperature over the mold cavity. It can reach up to 700 °C, with electrical resistance power of about 4000 W and 220 V.

This design directs the heat exactly above the preform. A fiberglass blanket was used to isolate the heated system. An effective temperature control is provided by Type K thermocouples.

2.3. *Injection pressure and vacuum*

The importance of the injection pressure control is justified by its influence on the quality of injected products and the possibility of increasing the production. This can minimize problems as unsuccessful mold designs and poor control of injecting conditions. However, excessive pressures can cause deformations on preforms, as cited above[19,20].

Some mold design details, as corners, can affect the injection pressure. According to Bickerton[21], higher injection pressures are needed to mold filling with smaller corner radii.

The injection pressure increases as fiber volume fraction increases, showing that permeability is highly affected by the fiber volume fraction. Besides, the flow front resin can vary according to the fabric quantity and type[21]. Parameters as injection pressure, vacuum and temperature can be adjusted after each process to guarantee and improve the complete mold filling[19].

The vacuum pump, Figure 4, completes the injection system, removing the remained air within the preform and aiding the flow front direction. Processes without a controlled vacuum assistance induce higher void content in composites[6]. Besides, the cooled trap aids with the workplace healthy by collecting the resin surplus and monomer.

3. Experimental Work

3.1. *Materials*

Four types of intermediated modulus carbon fabric were employed: NCF (non-crimp fabric); 5HS (harness satin weave); PW (plain weave) and TW (twill weave), all impregnated with Cycom 890 RTM epoxy resin system. It is a one-part liquid epoxy resin system produced by Cytec Industries. The mechanical and thermal properties are suitable for aeronautic applications. It was employed a cure cycle of 2 hours at 180 °C and reaching a final *Tg* around 210 °C, considering the tanD peak of DMA analysis. The viscosity profile (time versus viscosity) of this resin system allows the injection of large parts, in which time is a limiting parameter.

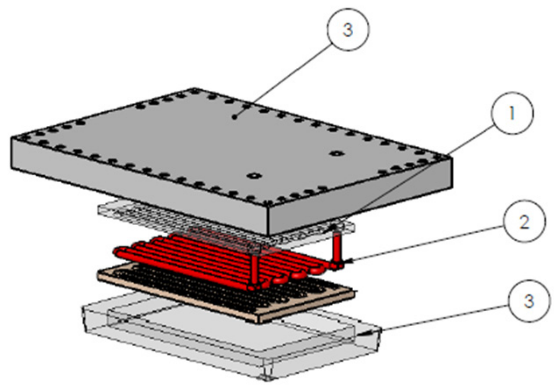


Figure 3. Heat system assembly on the mold: (1) aluminum plates, (2) resistance and (3) mold parts.

Figure 4. Controlled vacuum system: a - manometer; b - monomers cooled trap; c - resin trap.

3.2. *Sample preparation*

Four composite samples were prepared with different types of carbon fibers and one commercial epoxy resin system Cycom 890 RTM. The samples were named as NCF (non-crimp fabric), 5HS (satin weave), PW (plain weave) and TW (twill weave) that refers to each fabric type, according to Table 1. A total of 9 tests, as detailed in Table 1, were carried out with 4 types of fabric configuration. The identification T_i (i = 1,2,3...9) was used to identify the manufacturing and test condition of each sample.

The number of plies for each preform was defined in order to reach a fiber volume fraction over 50%. Considering that preform total thickness exceeded the 3 mm of the mold cavity thickness, in all cases the preform was compacted by the upper mold part, in order to reach 3 mm. The compression was higher for 5HS fabric, similar for PW and TW and lower for NCF fabric.

The theoretical fiber volume fraction was calculated considering the fabric real weight (gms = g/m^2), fibers density and the volume of the mold, according to Equation 1. In this research the thickness *t* was kept constant, as a consequence any extra ply would conduct to an increase in V_f.

$$V_f = \frac{gsm * n_p}{\rho_f * t} \quad (1)$$

where V_f is the fiber volume fraction, *gms* is the fabric real weight, n_p is the number of fabric layers, ρ_f is the fiber density and *t* is the mold cavity thickness.

3.3. *Viscosity analysis*

The Cycom 890 RTM resin viscosity profile was analyzed at 80 °C, 90 °C, 100 °C and 120 °C. It was carried out in a Brookfield DV-II + PRO viscometer. Each test was conducted for 60 minutes that is the time for the injection cycle.

3.4. *Acid digestion analysis*

By following the ASTM D 3171, the fiber and void volume fraction of the composites were determined. The composites samples were immersed in sulfuric acid at 150 °C for 4 hours and after hydrogen peroxide was added to the reaction. The resultant fibers were washed with acetone, then distillated water and dried at 100 °C for 12 hours.

3.5. *DMA analysis*

By following the ASTM D7028, the samples (50 × 7.2 × 3.0) mm T_g were determined using DMS 6100 model EXSTAR 6000 equipment (SII Nanotechnology Inc.). The analysis was performed in three-point bending mode with 10 µm of amplitude, at a frequency of 1 Hz and a 3 °C/min heating rate ranging from 30 °C to 300 °C.

4. Results and Discussion

Figure 5 illustrates different behaviors of the pressure development during the preform impregnation, and this case will be outlined to show the error and respective corrections employed. For the NCF-T1, the combination of high viscosity and a relative low fiber volume fraction (43%) yielded to a severe race-tracking at the corners and at the surfaces of the mold. Thus, this process took a few minutes to be completed, as the resin easily crosses through the preform at a low permeability locations.

Adding an extra ply to NCF-T2 and T3, the preform reached a fiber volume fraction of 54%, and adopting the same initial pressure and temperature of the NCF-T1 the development of pressure increased with time as a way to overcome the barrier of the fiber compaction. Due to a high pressure, the race-tracking phenomenon was developed at mold edges. The apparent solution was to decrease the resin viscosity by increasing the system temperature to 100 °C, which conducted a significant decrease in the injection pressure, as seen in NCF-T4. Despite the right choice in the mold tool, an increase of 20 °C created a large gap between the mold edges and the preform corners due to a

Table 1. Materials and manufacturing conditions.

Fabric type	Number of plies	Nominal thickness (NT)-uncompressed (mm)	Fiber volume fraction (%)	Preform compression (*MT/NT) (%)	Temperature (°C)
NCF-T1	4	3,2	43.44	6.67	80
NCF-T2	5	4	54.31	33.33	80
NCF-T3	5	4	54.31	33.33	80
NCF-T4	5	4	54.31	33.33	100
NCF-T5	5	4	54.31	33.33	100
NCF-T6	5	4	54.31	33.33	100
5HS-T7	10	5	52.43	66.70	100
PW-T8	14	6,2	51,56	48,39	100
Twill-T9	14	6,6	52,43	45,45	100

MT (mold thickness); NT (nominal thickness).

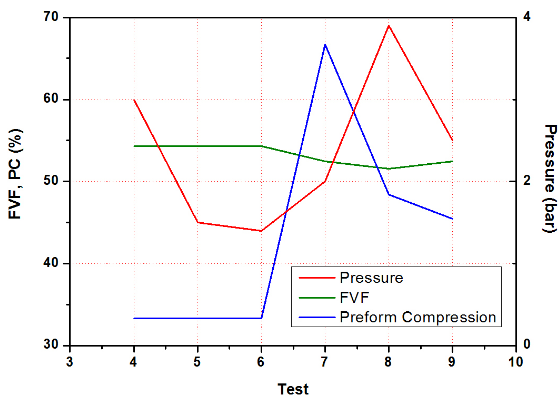


Figure 5. Comparison of the preform features with applied pressure.

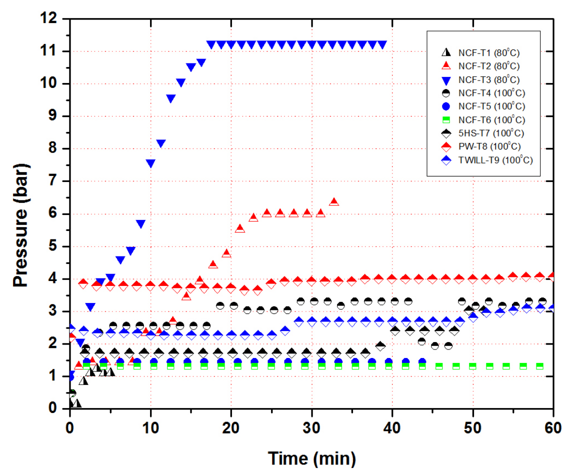


Figure 6. Pressure development of the 9 tests.

difference in the materials CTE. Adjustments in this gap was through the introduction of silicon gel. Other options could be chosen by rearranging the inlet gate positions to prevent or compensate the higher speed of the resin flow created in these regions. In the NCF-T5 test, the flow did not require any increase in the injection pressure, but required only a constant pressure of 1.5 bar. From this test, it was observed that as the pressure was kept constant, the flow front speed varied with time, as seen in Figure 6. This is regarded to the pressure gradient along the mold length.

For the PW-T8 and the TWILL-T9 tests, the injection pressure profile showed the same behavior. When the injection pressure was increased at about ≈25 min, it avoided a exceeding decrease in flow front progression, as seen in Figure 6. This increased of the pressure is necessary to overcome the lack of pressure as the flow front become closer to the outlet gate. In relation to the flow front behavior of the two woven fabrics: PW and TWILL, there are some differences in the applied pressure, as seen in Fig. 5, which can be considered as a matter of the ply interlacing characteristics. Other features like fiber volume fraction, fiber tow and number of plies kept the same. In the PW preform there is more crimp thus locking the neighboring plies, and as a consequence more pressure is required to overcome each increment of the flow front. For the TWILL weave the reduced crimp might avoid the locking characteristic.

According to Endruweit et al.[22] the number of layer affects the permeability in which the resin tends to run between them. However for the quadri-axial NCF the number of plies was less than the other laminates that would conduct to a low permeability that was not observed in the low pressure evolution in Figure 5.

The low pressure required for the last three NCF tests can be explained by the fabric construction in which gaps are inserted by the stitch thread format conducting to an enhanced intertow flow[23]. Another factor is the lowest preform compaction/compression (PC) as seen in Figure 5, despite the fact they presented higher theoretical FVF than other preforms due to its large tow size, as seen in Table 1, they required lower pressure. This permeability behavior has been already mentioned in work published by Liu et al.[23] but for a fiber glass NCF preform.

According to Jinlian et al.[24], the preform microstructure is a characteristic that can induce a different local permeability, reaching several orders of magnitude between insider and outsider fiber tow. This fact can lead to undesired micro voids formation in liquid process moldings.

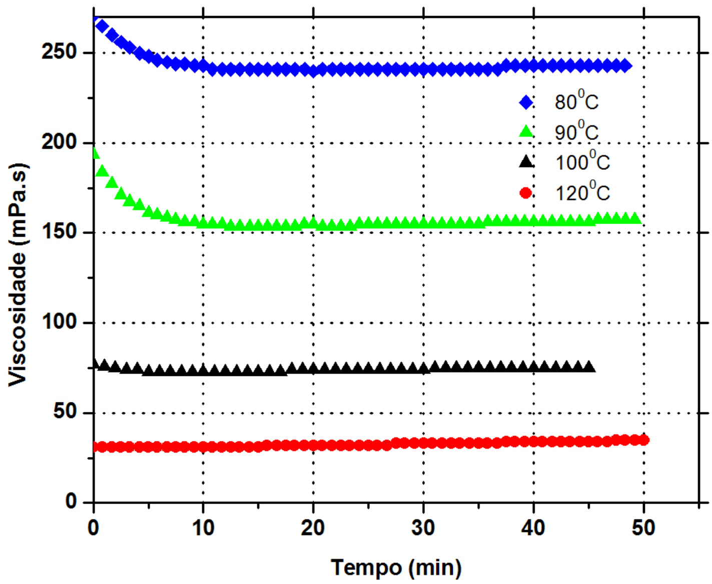


Figure 7. Epoxy resin system (Cycom 890 RTM) profile viscosity.

Table 2. Acid digestion and porosimetry composites data.

Composite	NCF-T6		PW-T8		TWILL-T9	
Values	Resin Inlet	Resin Outlet	Resin Inlet	Resin Outlet	Resin Inlet	Resin Outlet
V_f (%)	55.56 ± 2.12	52.85 ± 1.06	54.22 ± 1.17	56.33 ± 1.22	56.92 ± 0.28	58.19 ± 0.42
V_m (%)	40.49 ± 3.13	47.43 ± 1.57	45.04 ± 1.73	41.92 ± 1.81	42.03 ± 0.40	40.18 ± 0.61
V_v (%)*	6.37		2.94		3.27	

*Obtained through Hg porosimetry test - average value.

Table 3. Tg values by DMA (tan δ peak).

Composite	NCF	5HS	PW	TWILL
Tg (°C)	194	209	201	208

The vacuum assistance helps to minimize the voids, but the key to produce void free composite parts is by optimizing the process parameters as resin gel time and injection pressure[24,25], besides the mold characteristics as inlet gates, outlet vent and mold corners, since they can contribute to void formation[26].

Figure 7 shows the epoxy resin viscosity profile for four possible injection temperatures. A high thermal sensibility can be noticed, allowing advantageous diminishing viscosity by increasing the temperature. The gel time reaches after 60 minutes, within it the injection cycle last. This large temperature window allow the optimization for a variety of porous media in order to reduce the void content.

The NCF-T6, PW-T8 and TW-T9 void test results is showed on Table 2. Despite the fact that there is a tendency of void formation on warp that may increase the void volume fraction in woven fabrics as compared with NCF[24], here opposite trend was observed as the enhanced flow front in NCF may lead to void entrapment thus justifying high content of voids.

The void content of the test results showed that all the laminates are just above the limit recommended for aeronautical application, as beyond 2% of void volume fraction can substantially affect tensile, flexural and shear properties[26-28]. In this case other void content characterization techniques are recommended as the acid digestion indicated values accepted for such application.

The acid digestion results (Table 2) show similar values of fiber volume fraction (V_f) between all composites. According to these values, the time and pressure required to fill the mold is a function of fabric permeability or difficulties imposed by each textile. The NCF fiber volume fraction has an average of ≈2.21% less than the PW and TWILL preforms in which, combined with the permeability factor, have increased the injection time.

For PW-T8 and TW-T9 laminates, the low FVF on the inlet region means a better resin (V_m) impregnated region due to inlet gate proximity. This preform region remained for a long time in contact with the resin, thus favoring better tow fiber impregnation that can be named as saturation effect[29]. NCF fabrics did not present the same behavior, probably due to a better fiber alignment that facilitated the resin flow.

Table 3 shows the T_g temperatures obtained via Tanδ peak by DMA analysis, for a comparative purpose. By knowing that the cure temperature establishes the T_g temperature, both are associated with polymer cross-linking density formed during the cure cycle[30,31].

Similar T_g values are an indicative of an efficiently heat system. The difference among T_g values can be attributed to fiber/matrix boundary interface differences, since T_g

and degradation temperatures are signals of interfacial quality interaction, considering composites with the same matrix[32,33]. PW-T8 and TW-T9 have been submitted to interfacial adhesion analysis in a previous study, their dynamical-mechanical analysis results confirmed the TW-T9 higher adhesion, besides better thermogravimetric and SEM analysis features[34].

5. Conclusion

By comparing the processed composites characteristics, the RTM laboratory scale system can be considered adequate to produce high quality composites, since similar quality was found for different laminates.

The ideal temperature of the epoxy resin system was defined as 100 °C. In this temperature the flow front through the porous media was improved, and as consequence, the pressure injection was reduced. This temperature enabled to produce laminates with FVF over 50% due to its low viscosity.

The flow front velocity and injection pressure are also affected by viscosity, however the injection pressure increment required during the process was mainly attributed to low permeability because of the ply interlacing features. In regard to this, the NCF and woven fabrics showed different behavior as evaluated by the pressure evolution, despite the fact they have similar FVF.

By the analysis of the composite constituents, it can be observed that although the NCF has higher permeability, the woven fabrics have enhanced impregnation at the inlet gate that could be explained by the longer time of fiber tow exposure.

The differences in T_g and void volume fraction among the laminates was attributed to reinforcement characteristics. The similar T_g of the composites confirmed the efficiency of heat transfer through the mold walls, so the difference can be attributed to interface differences. The void volume fraction values is low enough for aeronautical applications, due to a good impregnation promoted by adequate combination from mold project, injection parameters and vacuum system. The void volume fraction differences were attributed to variations in the preform permeability of each type of lay up that has its interface locking features.

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