Simulation and Experimental Validation of the Effect of Material and Processing Parameters on the Injection Stage of Compression Resin Transfer Molding

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The effects of gap thickness, injection flow rate, overflow percentage, initial fiber content, and resin viscosity on the resin distribution at the end of the injection stage of compression resin transfer molding have been studied. Experimental results have shown that in all the cases, a certain amount of resin penetrates the preform, contrary to the extended hypothesis that the resin flows exclusively through the gap. Furthermore, resin distribution is not homogeneous and the penetration depth varies depending on the distance from the injection point, being deeper near the inlet point. Simulation results based on the quantitative analysis of the amount of resin penetrating into the preform demonstrates that the most influent parameters are the initial fiber content, the gap percentage and the overflow percentage. In fact, the ratio between the permeability of the gap and the preform (*r*) governs the tendency of the resin to flow through the gap or to penetrate into the preform. Increasing *r* reduces the amount of resin which penetrates into the preform, but there is an upper asymptotic limit from which no differences are noted. Finally, simulation results have also demonstrated that the preform compression can be neglected during the injection stage, as the pressure into the preform is low. POLYM. COMPOS., 00:000–000, 2017. © 2017 Society of Plastics Engineers

INTRODUCTION

Nowadays energy efficiency is a big challenge for road transportation, and lightweight materials are on the strategic agenda of all car-makers [1]. Carbon fiber-reinforced polymers (CFRP) are interesting candidates as they can reduce 70% of the vehicle weight and can dissipate five times higher impact energies than metals [2]. However, the high operational manufacturing costs involved, in combination with the technologies currently employed, have restricted a wider use of CFRP [3]. Consequently, considerable effort has been made toward finding and developing alternative cost-effective routes for manufacturing CFRP components.

Resin transfer molding (RTM) of thermoset polymers is a well-established processing method [4]. However, there are still some problems when high fiber content parts (typically >40% for structural applications) must be manufactured. As the fiber content increases, the permeability of the preform decreases, which produces a longer mold filling time, incomplete impregnation of the preform, and high void content in the component. To overcome these problems, several strategies have been presented to modify the conventional RTM process. An alternative approach to reduce simultaneously the mold filling time and void content is to add a compression stage to the RTM process. This process is called compression resin transfer molding (CRTM) and can be a suitable technique to manufacture structural composites having most of the advantages of RTM [4, 5]. In CRTM, unlike in conventional RTM, the mold is only partially closed when resin injection begins. This increases the cross-sectional area available for the resin flow, and decreases the flow resistance by providing a high porosity in the reinforcement. In some cases, a small gap is left between the surface of the preform and the mold wall which facilitates further the resin flow. Once the required amount of resin is injected into the gap and the gate is closed, the mold plate moves down to close the mold and squeeze the resin into the preform in thickness direction, which also undergoes compaction to achieve the desired volume fraction. The flow pattern during CRTM is more complex than in conventional RTM, as during the injection stage, resin flows preferentially through the gap, but it also penetrates the preform [6]. The resin distribution at the end of the injection stage determines the flow during the compression stage, and consequently the quality of the final part, the force required compressing the material and the total cycle time [7].

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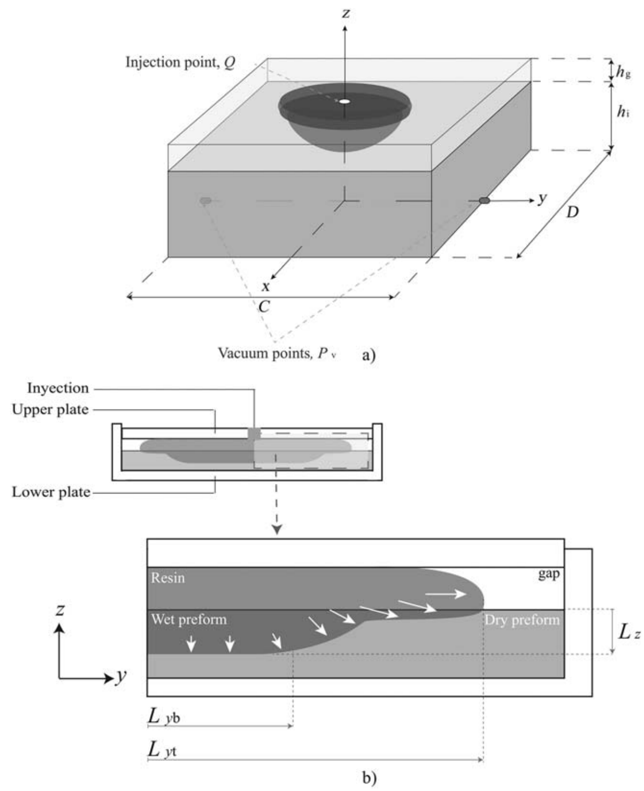

FIG. 1. Injection variables scheme. (a) Scheme of the geometric variables. (b) Cross-sectional view of the sample.

There are numerous materials and processing parameters that must be considered in CRTM, which makes the understanding of the role of each of them more difficult. To understand the complex flow path during the injection stage of CRTM, experimental methodologies should be complemented with simulation approaches. In the published research works, two different modeling approaches can be found; those that consider that the resin flow front flows homogeneously through the thickness direction [8–11], and those that assume that the resin flows nonhomogeneously into the dry preform [12–15], which is more realistic. Simacek et al. [6, 12] developed a three-dimensional model that takes into account the through thickness flow, and showed that the model describes correctly the influence of parameters such a as the gap thickness and the preform permeability. However, qualitative or quantitative analysis regarding the flow patterns were not reported, and they did not validate their model by comparing their results with experimental ones. Shojai [14] considered the injection stage to be dependent on more parameters (gap thickness, permeability of the preform, and the amount of injected resin), but without quantifying the influence of each parameter, neither correlating the results with experiments.

It can be concluded that the assumption that the resin flows exclusively through the gap is not valid. It is also evident that there is a lack of knowledge about the role and influence of each material and processing parameter on the flow pattern. Furthermore, nobody has correlated the simulation results with experimental ones. Thus, the scope of this work is to study how the material and processing parameters affect the flow pattern during the injection stage of the CRTM process, both qualitative and quantitatively, based on simulation models and experimental trials.

INJECTION STAGE PARAMETERS DEFINITION

Nomenclature

A	Area of the part
C	Length of part on x axis
D	Length of part on y axis
h_i	Initial preform thickness
h_f	Final part thickness
h_g	Gap thickness
$\mathbf{K}$	Permeability tensor
k_0	Carman–Kozeny equation constant
K_{ii}	Permeability on function of fiber content
L_{xb}	Impregnated length on x axis bottom view
L_{yb}	Impregnated length on y axis bottom view
L_{xt}	Impregnated length on x axis top view
L_{yt}	Impregnated length on y axis top view
L_z	Impregnated length on z axis
P_v	Vacuum pressure
Q	Injection flow rate
r	Ratio between permeabilities
sv	Standardized effect value
t_f	Injection filling time
V_i	Impregnate volume
V_f	Final part volume
V_r	Resin volume
v_{fi}	Initial fiber content
v_{ff}	Final fiber content
$\bar{\mathbf{u}}$	Superficial velocity vector
μ	Viscosity
$\emptyset$	Porosity
p_o	Overflow percentage
p_g	Gap percentage
p_{gf}	Gap filled percentage

Parametric Study

The injection analysis is carried out for a rectangular sample with a central injection point, which generates a circular flow path, and vacuum is applied in the midpoint of the short sides of the rectangle. Figure 1a shows the studied geometry for the injection phase. The dimensions of the rectangle are 150 × 260 mm. The coordinate system of the model is defined as z is the through thickness direction. Owing to the symmetry with respect the x and y axis, only a quarter of the part has been modeled to reduce the computational cost. For analyzing the flow pattern, three measurements are obtained (Fig. 1b): impregnation length in the z direction (L_z), impregnation lengths in the plane (top, L_{xt}, L_{yt}, and bottom view, L_{xb}, L_{yb}), and impregnated volume (V_i).

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TABLE 1. Filling parameters considered in this study. The input parameters divided into two groups influenced by design and process, and output parameters measured after the test.

Design		Process	
Length of part on x axis, C	260 mm	Gap thickness, p_g	$h_g = h_c(1-v_{ff})\,(p_o)\,(p_g)$
Length of part on y axis, L	150 mm		$K_g = \frac{{h_g}^2}{12}$
Final part thickness, h_f	3.5 mm	Injection flow rate, Q	
Final fiber content, v_{ff}	60%	Initial fiber content, v_{fi}	$h_i = h_f \frac{v_f}{v_{fi}}$
Viscosity, μ		Overflow percentage, p_o	$V_r = (1-v_{ff})\, h_f A\,(p_o)$

The nine independent materials and processing parameters to be considered in the present study are reported in Table 1. They are divided into two groups: (1) parameters related to the design, influenced by the final part geometry and (2) processing parameters. The final properties at the composite are fixed, whereas these parameters related to the resin and injection conditions are changed.

Owing to the big number of input and output variables that can affect the process, 2^k Factorial Design experiments have been used to separate the most influencing and noninfluencing variables. This design of experiment has been applied to simulation results. Table 2 shows the range of values analyzed for each parameter. The range of μ, v_{fi}, and Q has been defined based on Ref 16, which is a similar CRTM injection-stage study. The overflow percentage (p_o) refers to the exceed of volume injected, taking as reference the resin volume at the final composite. $p_o = 1$ involves that the volume of the resin injected is the minimum volume required to fill the cavity without being voids and a p_o value of 1.2 represents that the volume of the resin injected is 20% greater than the minimum. The last variable parameter analyzed is the gap percentage (p_g), which is the volume of the empty gap between the fiber surface and the upper mold wall. When the value of p_g is 1, it means that the volume of the gap is equivalent to the resin volume in the final composite, and 1.2 values means that the volume of the gap is 20% greater than this resin volume in the final part.

SIMULATION MODEL

The conventional RTM module of PAM–RTM has been used to simulate the injection stage of CRTM. The calculations of the flow are based on Darcy's law and the continuity equation, which have been widely used by researchers to describe the flow mechanisms during the RTM process [5].

In RTM process, as the velocity of the flow through the thickness can be considered to be uniform, it is enough to use a two-dimensional mesh to capture the flow front. However, in CRTM process, as there is a preferential flow into the gap, the flow through the thickness is not uniform and to capture the flow front three-dimensional elements must be used. Consequently, a three-dimensional porous medium with fixed dimensions has been employed to model the flow. As the permeability of the gap is homogeneous and the initial dry-preforms permeability is different in the three main directions, the model is divided into two parts; the gap (h_g) and the dry-preform (h_i). The convergence analysis carried out with different mesh sizes has shown that 1.5 mm is the optimum size for the linear tetrahedral elements. Consequently, the gap mesh has 27,742 elements, and that of the dry-preform has 55,486 elements. The simulation is completed with the following assumptions:

- The preform thickness and consequently its permeability is considered constant, when the pressure during the injection stage is lower than 20 kPa [13].
- The mold is undeformable.
- There is no racetraking.
- The resin is uncompressible.
- The resin viscosity is constant, as during the earliest stage of the CRTM, conversion degree is very low and viscosity changes are negligible.

Material Characterization

The preform is a biaxial carbon fiber 50 k noncrimp fabric (reference HPT 610 C090) with 610 g/m^2. The binder is an epoxy resin in powder state (Araldite LT 3366 BD) and the amount of binder applied per layer is 15 g/m^2. The test fluid used is a commercial silicon oil with a constant viscosity of 0.1 Pa s in room temperature (Siameter 200 PMX), which is Newtonian behavior.

The permeability of the gap K_g (m^2) can be estimated using the equation for creeping flow in narrow channel of gap thickness, h_g (m). This equivalence is acceptable when the thickness of the gap is much smaller than the in-plane dimensions [6].

TABLE 2. Range studied for each variable parameters.

	Level		
Factor	1	2	
μ	0.05	0.5	Pa s
v_{fi}	40	45	%
Q	200	500	cm^3/min
p_o	1	1.2	
p_g	1 (h_g = 1.44 mm)	1.2 (h_g = 1.72 mm)	

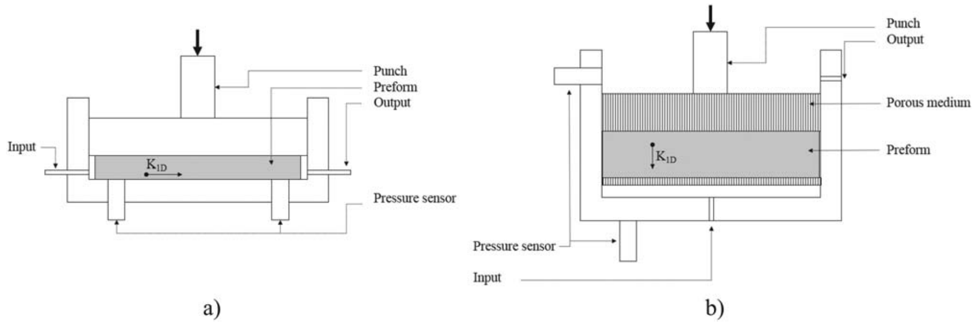

FIG. 2. Permeability devices: (a) in-plane [17] **and** (b) through thickness [18].

$$K_g = \frac{h_g^2}{12} \quad (1)$$

The plane permeability measurement has been performed based on the testing methodology proposed by Vila et al. [17] (Fig. 2a). Through thickness permeability has been measured following the methodology proposed by Ouagne et al. (Fig. 2b) [18]. The basis of the test is the same in both cases and permeability measurements have been carried out under saturated conditions.

The evolution of the in-plane and through thickness fabric permeability with the fiber volume fraction, v_f, is plotted in Fig. 3. It can be seen that through thickness permeability is one order of magnitude smaller than in plane and four orders of magnitude smaller than gap permeability.

The experimental results followed the Carman–Kozeny equation [19], where k_0 is an empirical constant which depends on the specific preform was obtained by the least squares fitting the experimental results in Fig. 3, listed in Table 3.

$$K_{ii} = k_0 \frac{(1 - v_f)^3}{v_f^2} \quad (2)$$

RESULTS

Simulation Results

Parametric Study. The design of the experiment study has permitted to determine the influence of the different

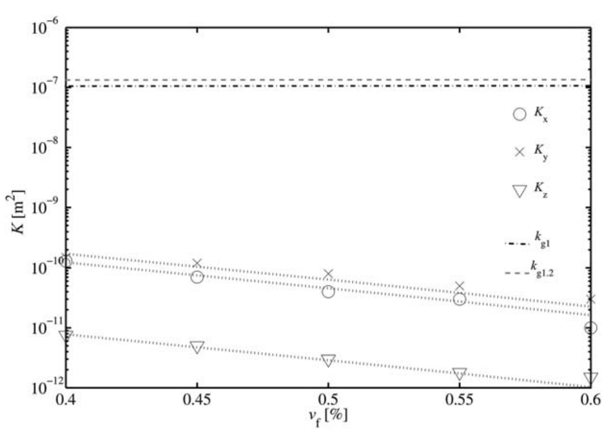

FIG. 3. Permeability in-plane and through thickness direction in function of fiber content. Dotted lines represent the models.

parameters involved in the injection phase. Those variables on the Pareto chart of the factor effect <5% were discarded for further studies. This 5% threshold value corresponds to a standardized effect value (*sv*) given by *Minitab* of 2.2. Any effect that extends beyond this reference line is potentially important. As shown in Fig. 4, only three variables (h_g, v_{fi}, and p_o) exceed this threshold, so the study will focus on them; the most influential are gap height, followed by the fiber content of the initial preform and finally the volume of injected resin. It should be mentioned that even if the influence of the viscosity is very low, increasing viscosities may increase the pressure and this could also increase the preform compaction and thus reduce the permeability. These higher viscosity systems are out of the scope of this study.

Analyzing these three parameters, the volume of the resin injected is the only one that does not modify the distribution of the resin flow front. It can be seen that the greater the volume of injected resin, the greater the possibility of filling the gap is, but it should be taken into account that the material cost and injection time also increase. However, the gap height and the fiber content of the preform determine the permeability of each zone within the mold and can modify the filling pattern. Consequently, the study will focus on these two parameters.

Up to now, the parameter study is based on a 2^k Factorial Design experiments. If the tendency of the influence of each parameter is to be analyzed, then two values for each are not enough. Therefore, another three intermediate values have been chosen. The new range of values analyzed is the following one: fiber content (v_{fi}) of 40%, 41%, 42%, 43%, 44%, and 45%; and gap height (h_g) 1.44, 1.51, 1.58, 1.65, and 1.72. Figure 5 shows the relationship between the gap height, fiber content, and filling percentage of the gap, taking into account that in Figs. 5 and 6, it is assumed that the resin injection dose was

TABLE 3. Carman–Kozeny equation constant.

	Constant
$\boldsymbol{k_{0xx}}$	9.13×10^{-11}
$\boldsymbol{k_{0yy}}$	1.27×10^{-10}
$\boldsymbol{k_{0zz}}$	5.76×10^{-12}

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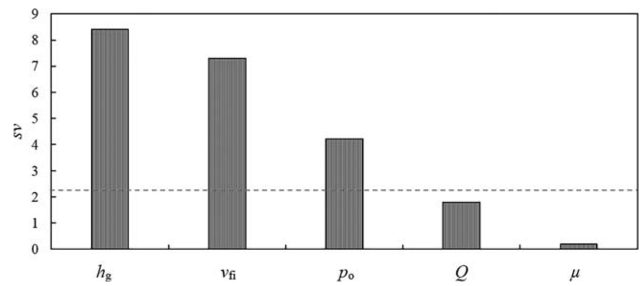

FIG. 4. Standardized effect (% of impregnated; α = 0.05).

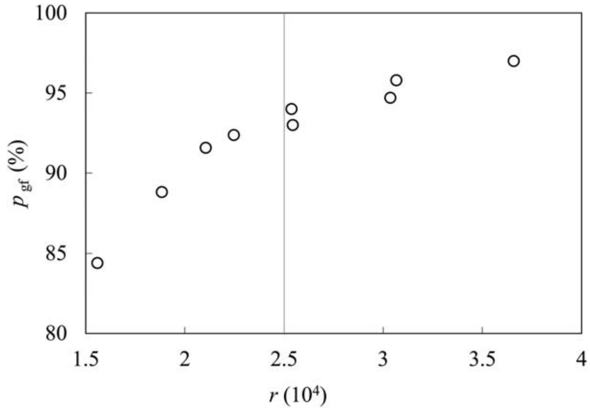

FIG. 6. Relationship between gap filling percentage (p_{gf}) and r ratio.

exact. Figure 5a shows that increasing the gap height and fiber content, the filling percentage increase. Additionally, analyzing the maximum value of each parameter (Fig. 5b), it can be seen that for the higher value of gap height (h_g: 1.72) the gap filling is 90%, whereas for the higher value of fiber content (v_{fi} = 45), it is 95%. Consequently, it can be deduced that the initial fiber content influence is higher than the one of the gap height.

From the phenomenological point of view, there is a competition between the case for flowing into the gap and the case for penetrating into the preform. In fact, this confrontation between trends can be represented by the ratio between both permeability's, *r,* where the boundary resistance of the two media is analyzed:

$$r = K_g / K_{zz} \tag{3}$$

Figure 6 shows the influence of r in the gap filling percentage. It can be seen that up to 2.5×10^4, the gap filling increases and for r values higher than this threshold, the filling establishes in an approximately value of 95%. From this optimal threshold, r value is stable, but the process conditions vary. To increase the r value, one option is to increase the gap height, which leads to a higher mold closing time; knowing that the resin dosage injected is exact, if the height of the gap increases, the resin volume injected will be less than the gap volume, so the gap will never get filled. Another strategy to increase r is to increase the fiber content, which makes penetration difficult. To compact the dry preform to such content in fiber and obtain the desired thickness, greater press tonnage is needed for compaction. Also, increasing the fiber content, the permeability in thickness direction decrease, needing higher press tonnage to subsequent impregnation phase in thickness direction.

Impregnation Patterns. The understanding of the injection stage comprises a quantitative analyzation (Parametric Study), but it must be completed with a qualitative study.

Based on the three parameters, two different groups of impregnation patterns are identified: (1) those in which the bottom of the piece is not impregnated (pattern I and pattern II), considering that the percentage of volume of resin penetrated into the preform is <20%, so the subsequent impregnation will be in thickness direction (Fig. 7a and b); and (2) those in which the impregnation arrives to the bottom face of the preform, which means that the flow will follow a more complex pattern including both,

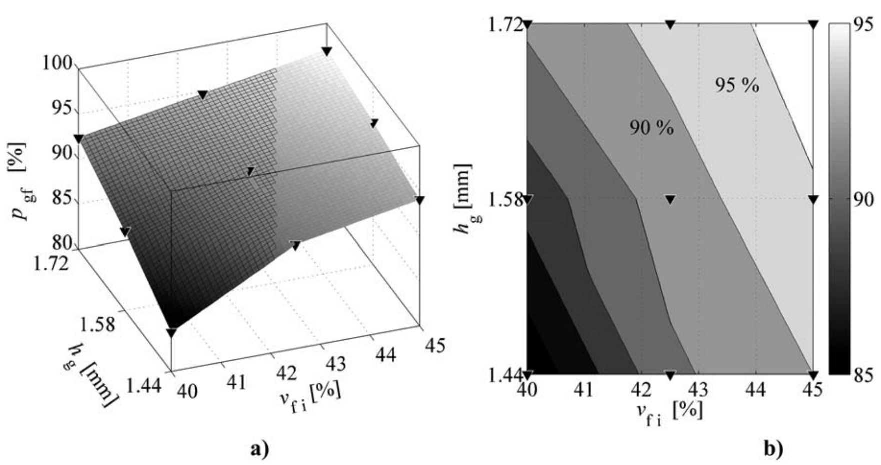

FIG. 5. (a) Relationship between gap height, fiber content, and gap filling percentage. (b) Vary of gap filling percentage between gap height and fiber content.

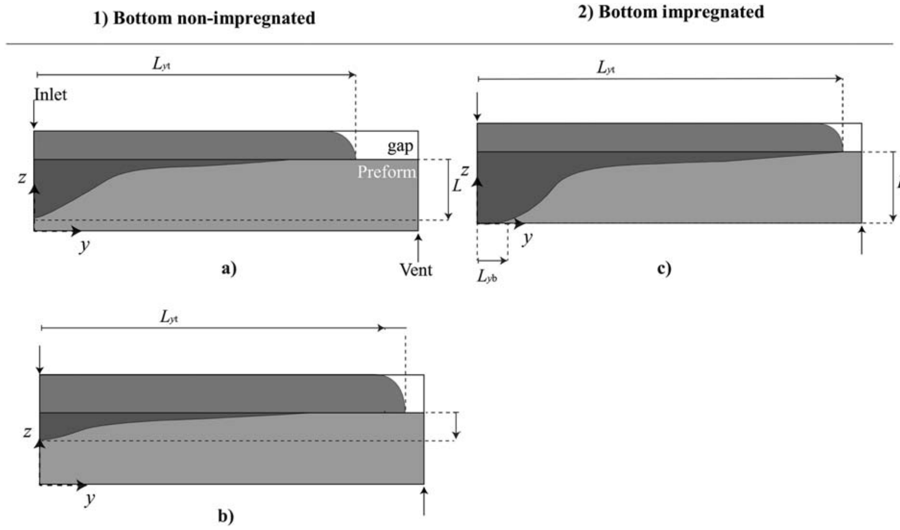

FIG. 7. Different impregnation patterns: (a) pattern I (v_{fi} = 40, h_g = 1.44, p_o = 1); (b) pattern II (v_{fi} = 45, h_g = 1.72, p_o = 1); (c) pattern III (v_{fi} = 45, h_g = 1.44, p_o = 1.2).

in-plane and through thickness directions. Depending on the impregnation direction (in plane or through thickness), the pressure distribution of the mold varies and especially does the quality of the part impregnation (Fig. 10). So, it will be interesting to analyze the parameters that change the impregnation pattern. Starting with the first group, among all the simulations performed, two patterns are selected; pattern I (v_{fi} = 40, h_g = 1.44, p_o = 1), where the resin has penetrated into the preform 16% but does not impregnate the bottom view, and the pattern II (v_{fi} = 45, h_g = 1.72, p_o = 1), only 4% of the volume of resin penetrated into the preform. Regarding the second group, pattern III was detected (v_{fi} = 45, h_g = 1.44, p_o = 1.2), where the resin impregnates the bottom of the preform, penetrating a 27% of the volume of resin. Comparing the two groups, it is shown that the volume of the resin injected and the r value of each pattern influence the impregnation pattern. It can be concluded that the pattern II comes closest to the ideal impregnation of CRTM, due to the homogenous impregnation and subsequence impregnation through thickness direction.

Validation

Experimental Setup. To validate and verify the simulation-based results shown in the previous section, the three selected impregnation patterns have been replicated experimentally. A transparent mold has been designed to analyze the flow pattern. The mold consists of an upper and a lower transparent PMMA plate; an intermediate metallic framework plate is introduced between them, which allows defining the thickness of the gap and the preform and it works as a preform stripper. The injection point is located in the center of the top plate, and the vent points are at the mid-points of the shortest sides at the bottom plate. As shown in Fig. 8, the mold is placed in a high base to see the bottom view. A mirror is placed on the bottom of the table to record the top and bottom view flow patterns at the same time. In all cases, the final thickness is the same, 6 layers of NCF are used. Otherwise, as initial preform thickness is a key parameter, binder powder is added to keep the compacted thickness during the injection trials. The forces needed to compact the preform to an initial v_f of 40% and 45% have been obtained from compression tests on dry fiber and are 0.2 kN and 0.3 kN, respectively.

Correlation. Figure 9 shows the results of the final impregnation obtained by simulations and experimentally. The correlation criteria have been (i) the shape of the impregnated zone and (ii) the difference between the longer flow-path (L_{yt}). In the case of the pattern I (Fig. 9a, experimental and Fig. 9b, simulation cases), the difference in L_{yt} is 4%. In some simulation and experimental tests, the resin has not impregnated the bottom of the preform, and the view of the resin distribution in the upper mold is similar in both cases. In the pattern II (Fig. 9c and d), the impregnation length error is 9%, and it shows

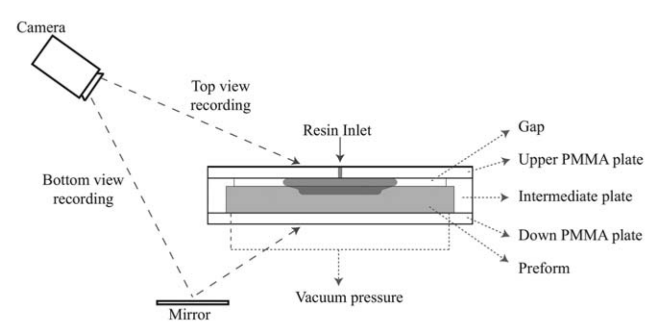

FIG. 8. Experimental test setup.

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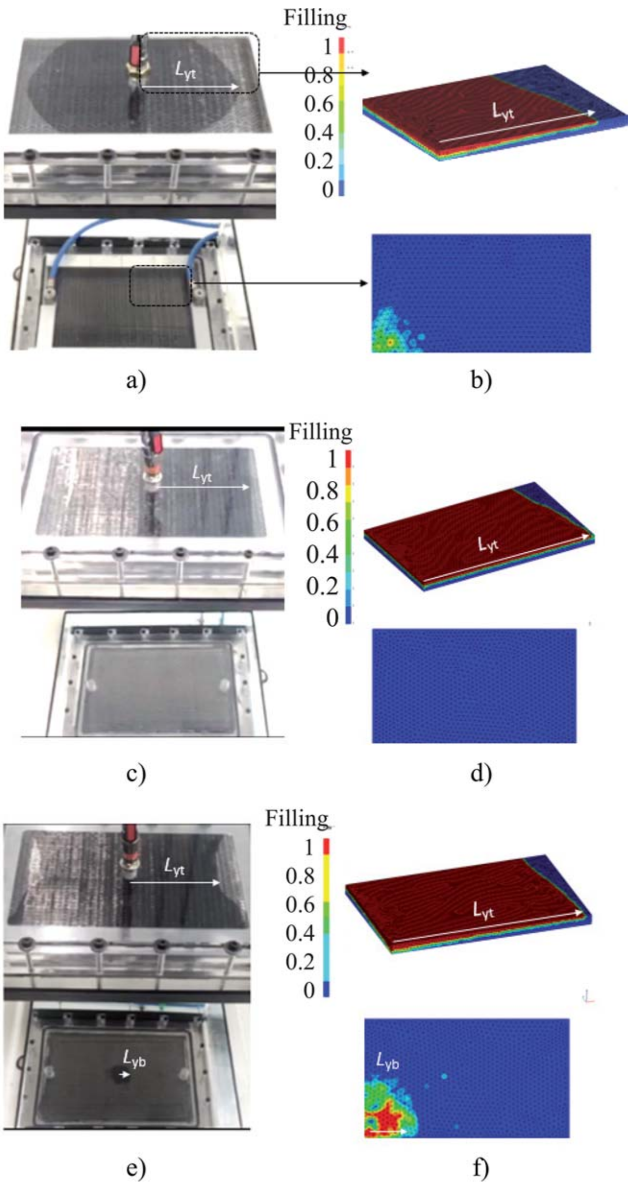


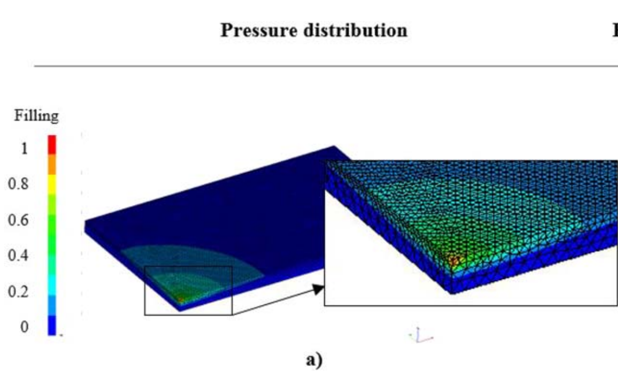
FIG. 9. Final flow pattern (3D transversal impregnation and top view): (a) Pattern I, (b) Pattern II, and (c) Pattern III. [Color figure can be viewed at wileyonlinelibrary.com]

the most homogeneous gap filling. Resin does not impregnate the bottom face in experimental and simulation trials, and the upper resin distributions are similar. Finally, the differences in the pattern III (Fig. 9e and f) are the biggest ones, as in the x axis is 16% and in the y axis is 25%. Analyzing simulation and experimental result, it can be seen that resin impregnated the bottom of the preform. Additionally, the gap is filled in a higher degree that in the other two cases, which is due to the fact that the gap volume is smaller and that the amount of resin injected is also higher. This phenomenon has not been detected in the simulations. The higher difference between simulation and experimental results are variability of the material property and the racetraking. Although it should be mentioned that CRTM is the process, where racetraking has less influence than conventional RTM, once the resin touches the corner of the piece, it tends to go around the corner. Another reason to have those errors is that the total areal weight tolerance of the material used is ±7%. Given the consistency of the mass of the samples, the differences in the filling pattern must be due to stochastic variability of fiber density. The variation in filling pattern is attributable to the fact that the spatial variability of the reinforcement is sufficiently significant to affect the accuracy of predictions made, assuming an homogeneous reinforcement [20].

In the simulation of the injection phase, it has been assumed that the injection pressure is small enough to neglect the preform deformation in the injection phase. To validate this hypothesis, Fig. 10 shows the maximum pressure obtained in each of the patterns and it has been seen that the pattern III generated the maximum pressure. Long Li et al. have shown [21] that NCF deformation with the aforementioned preform compaction pressures could be negligible.

CONCLUSION

In this work, a parametric study of the CRTM injection phase has been carried out. The main conclusions are that the flow-front pattern depends on the manufacturing parameters considered as variables in this study (Table 2)



Pattern	Max Pressure (kPa)
I	17
II	9
III	21

b)

FIG. 10. Injection pressure, distribution pressure, and maximum pressure. [Color figure can be viewed at wileyonlinelibrary.com]

and that resin always impregnates the preform. Consequently, the assumption that resin flows exclusively in the gap and that the resin distribution is homogeneous is not realistic. The simulation results show that the most important parameters are the initial fiber content, the gap thickness, and the overflow percentage. In fact, the ratio between the permeability of the gap and the preform (*r*) controls the amount of resin flowing in the gap. As *r* increases the resin volume also increases, approaching to the ideal fully filled gap, but it tends to an asymptotic value near 95%. It is shown that for *r* values beyond 2.5 $\times$ 10^4, the increase of the gap filling percentage is minimal and would require increasing both the mold closing time and the required compression force for the compaction of the preform. This confirms the existence of the optimal threshold that defines the beginning of the inefficiency of the process.

The correlation of the experimental results obtained in a transparent mold and those of simulation is good, which indicates that modeling the gap as a solid zone with a permeability calculated with the equation for creeping flow in narrow channel of gap thickness is a valid approach.

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