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Original Article

Impregnation behaviour of regenerated cellulose fabric Elium® composite: Experiment, simulation and analytical solution

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ABSTRACT

Filling time and volume fill prediction of long and complex parts produced using the method of resin infusion is of prominent importance. Fibre volume fraction, reinforcement type and composite laminate thickness significantly affect the manufacturing behaviour. It is crucial to have an estimate of fabrication parameters such as filling time. The PAM-RTM (resin transfer moulding) commercial software package makes it possible to characterize the production parameters in connection with lab scale experiments. In this work, simulation tools demonstrate an accurate prediction of the resin infusion process of pulp-based fabrics and characterization of the dynamic phenomena are verified using the analytical solution for a simple part. The accurate prediction for fabrication of pulp-based fabric Elium® composite demonstrated here can be beneficial for scaling up the composite part size and production speed. The filling time was accurately predicted until 270 s for the volume fill of 10–100% using the software tool and analytical solution. This proves the rayon fabric processing capabilities as a reinforcement for industry related projects and opens for the possibility of infusion process optimization.

1. Introduction

In the past two decades, the reinforcing potential of lignocellulosic and cellulosic fibres in polymers, in particular thermoplastics, has attracted industry and researchers [1,2]. Besides natural fibres (NFs), particular consideration has been paid to wood and pulp fibres [3], especially in the countries where this sort of natural resources is abundant such as Sweden. These fibres provide CO_2 neutrality, better disposal and recyclability, reduced abrasion to manufacturing machinery and possess a lower density of 1.5 g/cm^3 rather than 2.5 g/cm^3 as compared to that of synthetic glass counterpart [4,5]. Typical NFs lack uniformity. Nonuniformity can reduce their otherwise desirable intrinsic mechanical behaviours. To mitigate this, the individual cellulose fibrils of lignocellulosic fibre (which have the aspect ratios required for efficient reinforcement and are fairly uniform) need to be separated. This has not yet been achieved at a commercial scale and

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poses substantial practical issues. Besides, since the forest industry is focusing on pulp-based products rather than other natural fibres such as flax, ramie, hemp and jute, in this work we focus on these kinds of products. Rayon, which is a viscose type reinforcement, is produced by regeneration of dissolved cellulose and is used in the form of textile yarns. These yarns have been used as tire cord to reinforce car tires. Their low density and high tenacity lead to good impact resistance and high specific strength [6]. The other significant merit of pulp-based (rayon) fibres over NFs is their repeatable mechanical behaviour in composites as a reinforcement. Natural fibres can exhibit dissimilar properties due to the lack of control based on varying conditions during their growth and their posterior chemical modification.

It was shown that short rayon fibre polypropylene (PP) thermoplastic composite demonstrate comparable mechanical properties with those of glass fibre PP counterparts at 30 wt % of fibre [3]. Tensile strength of rayon-PP composite was recorded 78.7 MPa whereas the glass- PP one displayed a slightly lower value of strength (56.3 MPa). However, the modulus was found to be lower for rayon fiber based composite (2.9 GPa) than that of glass fibre composite (4.1 GPa). This proves the applicability of rayon as a reinforcement. The developed rayon PP composite was used for the fabrication of dashboards and door panels in collaboration with Cordenka and Faurecia interior systems [7].

To explore the processability and reinforcing potential of rayon fabrics in an industrial process like resin infusion, Elium® thermoplastic was selected as the choice of resin. The polymer system used in this work is a novel liquid thermoplastic resin, which possesses low viscosity in order to be processed similar to thermosets [8]. Elium®, which is an acrylic based resin, is polymerized at room temperature and provides mechanical performance similar to commercially used epoxy systems. The incorporation of a peroxide curing agent initiates free radical polymerization and subsequently methyl methacrylate (MMA) monomers react to form poly methyl methacrylate (PMMA) [9]. According to material safety data sheet of the methyl ester of methacrylic acid (CAS:80-62-6), the melting and boiling points are −48 and 100 °C, respectively. The chemical structure of the PMMA resin was previously shown by Bhudolia et al. [10], and the processing temperature is in the range of 20–60 °C.

Vacuum infusion (VI) or resin infusion is widely utilized for its ability to manufacture good-quality large parts. Currently, resin infusion design moulding depends to a great extent on user experience [11]. Hence, in order to cut costs and increase the speed and consistency of production, prediction of the vacuum process in the mould design plays a key role. Commercial Finite Element Method (FEM) software tools can be used to simulate the infusion process of fibre reinforced composites, however, some features of these software programs have minor effect on the infusion processes. Hence, these programs are rarely used in practice, especially at medium and small sized enterprises. Furthermore, these software licenses are costly and detailed material data are unavailable [12]. PAM-RTM (resin transfer moulding) software package uses FE analysis to predict the resin flow front. It allows optimization of outlet and inlet locations to ensure the complete impregnation of the laminate part prior to the beginning of the manufacturing campaign [13]. The permeability value depends on the fibre volume fraction or compression level of laminates and type of reinforcement. For reliable manufacturing simulation, accurate permeability values are crucial [14]. PAM-RTM can contribute to both calibrate the permeability values and predict the behaviour of composite processing. In addition, my-RTM uses cellular automata method to physically simulate the process and the approach was first proposed by Neumann et al. [15]. The algorithm used is based on the effect of the individual parameters according to Darcy's law [16] and calculates the state of the distribution of pressure in each cell during resin filling from the pressure difference and also calculates the properties of the cells in the zone at that particular time [17]. Moreover, if the part geometry is simple, an analytical solution can also reduce the cost of experimental production and save lots of time in this regard [18]. The suggested approach is to develop a mathematical solution with minimal characterization of reinforcement. For this purpose, Correia et al. [11] proposed an effective solution by compiling and developing the available formulations from the literature.

In this work, the reinforcing capabilities of bio-based rayon fabrics were investigated in a liquid thermoplastic matrix for resin infusion applications. Production of rayon fibre thermoplastic composites using resin infusion method has not been previously investigated in the scientific literature. Combination of rayon fabric together with Elium® polymer was used for the first time to make a bio-based fibre acrylic based thermoplastic composite. In addition, very few reports were made to measure the permeability of bio-based fibres. Besides, it was probably the first time PAM-RTM was used to calibrate the permeability value in the literature. Two commercial software packages were then used to simulate the resin infusion process and a unified analytical formulation based on substantial assumptions [19–24] was employed. All three methods were revealed to precisely predict the resin behaviour in the preform.

2. Methodology

2.1. Materials

Elium® 150 thermoplastic resin, a two-component system, was supplied by Arkema company, France. Dibenzoyl peroxide was used as curing agent provided by the same company. The viscosity and liquid density of the resin at the room temperature were 0.1 Pa s and 1.01 g/m^3, respectively. The resin is a colourless limpid liquid with a gel time of 30–40 min at 25 °C. 0/90 plain woven rayon fabrics (Cordenka® 700 2440 dtex) were provided by Cordenka company, Obernburg, Germany. The linear density and areal weight were given as 2482 dtex and 442 g/m^2, respectively. A yarn consists of 1350 filaments, and the yarn counts in the fabric were 24 yarns/cm in the weft and 40 yarns/cm in the wrap. It is worth highlighting that dtex stands for decitex and is the unit to display weight (grams) over 10,000 m in textile industry.

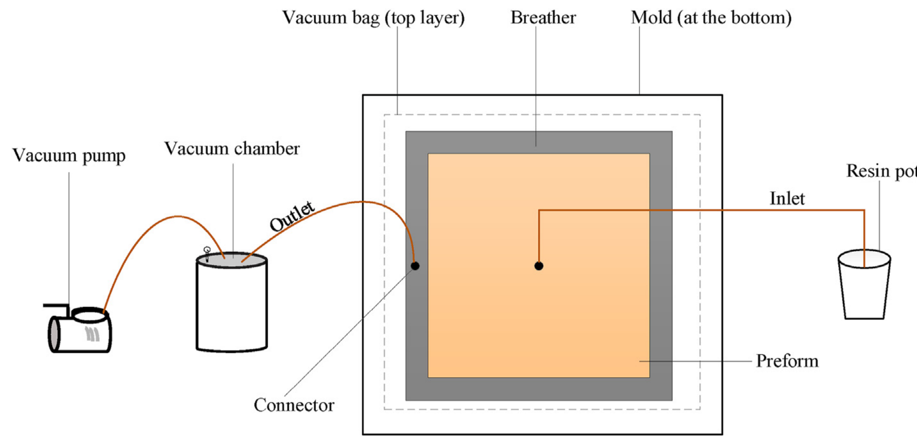


Fig. 1 – Graphical illustration of experimental setup for the permeability measurement.

2.2. *Permeability measurements*

Permeability measurements were carried out on the rayon fabrics according to the previously used method i.e. vacuum infusion [25]. The setup was composed of a flat sheet mould, breather, vacuum bag and preform assembled with a central injection point, as illustrated in Fig. 1. Three-layer square shaped preform measuring 150 mm side length was placed on the mould, which was coated with a spray release agent beforehand. To ensure an even distribution of pressure at the periphery, a 2.5 cm wide strip of breather cloth was positioned around the perimeter of the fabrics. A layer of vacuum bag was sealed on the whole assembly followed by connecting the resin inlet and outlet. The resin inlet port was placed right in the centre of the preform and rulers were located over the vacuum film to perform the measurements of flow front position along with the digital camera fixed with a stand right over the system. The tests were performed under 1 bar vacuum pressure, and the three layers of fabric were compacted cyclically three times by releasing the vacuum in the system and applying it again. The injection time and flow front locations were recorded and pictured at different time intervals to allow for calculations of the permeability values.

It was reported that, in order to obtain exact permeability values, two rigid mould halves are required, therefore a correction factor is required for the current setup [11,25]. Even with an exact mould cavity thickness, due to well-known difficulties involved in the precise permeability measurements [25], the decision was made to use the PAM-RTM software tool to accurately find the permeability value from the initial approximation based on resin infusion setup. In addition, the composite fabrication technique was also resin infusion.

2.3. *Resin infusion*

Since the thermoplastic resin was in the form of liquid, infusion method, which is an open mould technique was selected for composite processing. Closed moulding process such as RTM can be performed as well, if necessary. For the resin infusion test to evaluate the filling time and volume fill percentage, three layers of 0/90 rayon fabric measuring 100×300 mm were cut and then dried in a convection oven for a period of 24 h at 70 °C. They were positioned onto the mould previously surface coated by a layer of release agent (semiprem® monofilm release spray). The testing procedure applied is based on already established protocols [8,26]. A peel ply was placed on the fabrics and two small pieces of infusion mesh were put on the beginning and the end of the fabric to smoothly initiate and finish the infusion process. Subsequently, two spiral tubes, outlet PVC hose and resin feed PVC hose were attached to the system and the whole assembly was sealed with the aid of gum tape and vacuum bag. A double bagging system was used as suggested by the resin manufacturer to prevent shrinkage and provide void and detect free composite laminates [8]. Therefore, two layers of vacuum bag were adhered to two strips of gum tape around the perimeter of the mould. The compaction of rayon fabrics was also performed thrice prior to the resin infusion by applying the vacuum and releasing it. The acrylic based resin and dibenzoyl peroxide at the stochiometric ratio of 100 and 1.5 parts by weight were stirred for 3 min, respectively, then followed by its infusion. The fibre mass and volume fractions were obtained at 55% and 50%, respectively.

2.4. *Simulation and analytical formulation of composite part*

The influence of fibre quantity, composite laminate thickness and permeability can be considered for industrial practice process design to simulate the fabrication method. One of the precise process simulation codes developed by ESI group (Paris, France) to predict such behaviour is the commercial PAM-RTM software [13]. myRTM is another program for the simulation of resin transfer moulding (RTM) and resin infusion method. Both software packages contribute to the design of resin infusion and, hence, make efficient component development possible. The limitation of the myRTM software

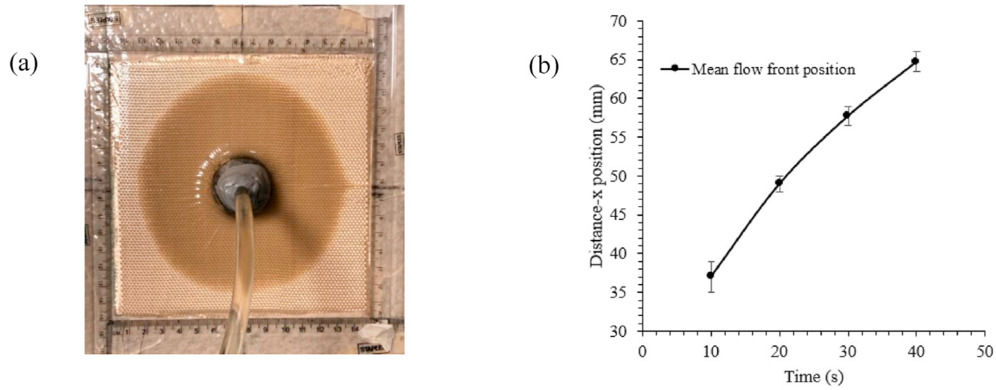


Fig. 2 – (a) The circular flow front position of resin in the preform in the permeability test after 30 s and (b) mean flow front position from the middle of preform with respect to time.

tool is that filling time percentage can be predicted and not the actual times at each step of volume fill unlike the PAM-RTM package.

Analytical formulations of governing equations of vacuum infusion [11] were used in this study to compare the prediction of flow of incompressible resin through compacting porous media with the simulation and experiment. The solution is a very complete development and finalized after compiling several works. The proposed model can quantify the impact of process parameters e.g. fabric lay-up and architecture, outlet and inlet pressures.

3. Results and discussions

3.1. Permeability measurements

In order to calculate the permeability, the plain (0/90) preform was considered orthotropic; however, radial flow front positions along x and y axes corresponding the respective time, which formed a circular shape, Fig. 2. (a), were almost the same after three repetitions of the test. This was expected as the properties and flow front are the same along 0 and 90° fiber orientations, similar to that of an isotropic material, and the laminate is balanced in plane direction. Therefore, the following equation (1) was selected. Chan et al. [27] and Adams et al. [28] provided a solution for the case of an in-plane (pseudo-) steady state flow of an incompressible fluid which in dimensionless form is:

$$F = \rho_f^2 (2 ln \rho_f - 1) = \frac{4 \times K \Delta Pt}{\varnothing \mu R_o^2} \quad (1)$$

where ρ_f is the ratio of the flow front radius (R_f) to the inlet port radius (R_o), and K, ΔP, t, φ and μ represent the permeability, the pressure gradient between the outlet gate and the flow front, the elapsed time, the porosity and the resin viscosity, respectively (Table 1). A linear regression through the origin and the data yields a plot of dimensionless F versus time (s), and the line slope can be used to calculate the permeability value (K). Function F represents the relationship between the flow front radius and the elapsed time.

The flow front positions were recorded after 10 s such that the resin passed the silicon connector radius (16 mm). Fig. 2 (b) shows the mean flow front radius (R_f) at different progressive time and Fig. 3 demonstrates the dimensionless function F curve derived from the R_f-time data. It is worth noting that the testing duration was designed to minimize the effect of alteration in resin viscosity due to the curing.

From the manufactured composite part, the required data was obtained to calculate the permeability, K (Table 1). The

Table 1 – The fitting parameters used to estimate the permeability.

Description	Symbol	Value	Unit
Porosity	Φ	0.5	–
Vacuum pressure	P	100,000	Pa
Resin viscosity	M	0.1	Pa·s
Thickness	T	1.5	mm
Gate diameter	D	5	mm

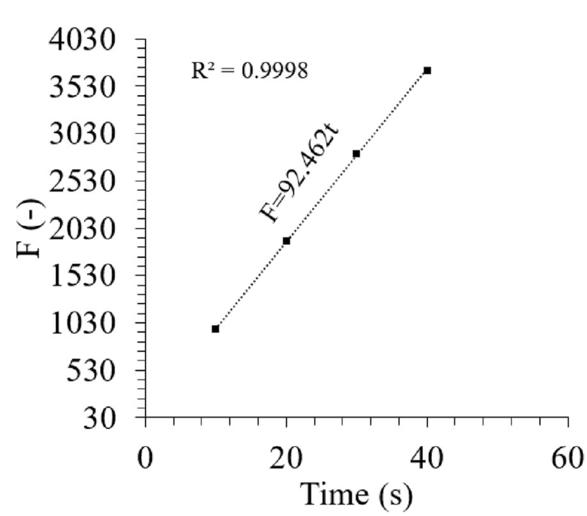
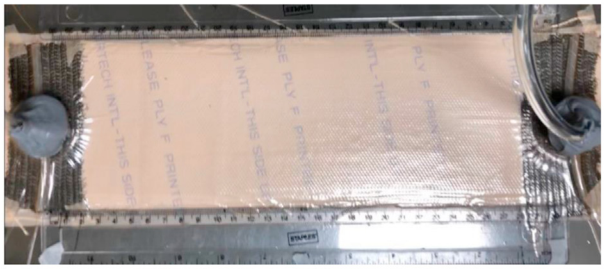


Fig. 3 – Function F vs. time (s) for the pulp-based fabric.



Infused distance (mm)	Mean fill time (s)
0	0
30	8
60	14
90	27
120	45
150	68
180	97
210	127
240	165
270	208
300	270

Fig. 4 – Resin infusion setup and the mean data for the infused composite plate.

permeability for the rayon fabric was measured 0.722 10^{-10} m^2. Lebrun et al. [25] used the same setup to measure the permeability of flax fibre, absorption paper and glass fibre with a resin viscosity of 0.42 Pa s. The K values for flax 200 (200 unidirectional text yarn), flax 1000, absorption paper and glass along the x axis (UD direction) were calculated as 0.0498 10^{-10}, 0.0253 10^{-10}, 0.0065 10^{-10} and 1.0953 10^{-10} m^2, respectively. Therefore, the K is higher for glass than rayon fabric investigated, which is expected as synthetic fibres typically possess a higher degree of K.

This method provides good approximation to calculate the permeability [25] and in the following section the accuracy of the value was validated with the experimental work (composite fabrication). Then, a resin infusion simulation by the PAM-RTM software tool was performed to calibrate the K obtained from the experimental setup.

3.2. *Resin infusion data*

The infusion time and infused distance (flow front progression) were obtained and the experimental setup and values are shown in Fig. 4. The infusion time started as soon as the resin entered the inlet silicon connector (0 s) and the process was considered completed once the resin flow front passed the 300 mm rulers positioned on both sides of the setup.

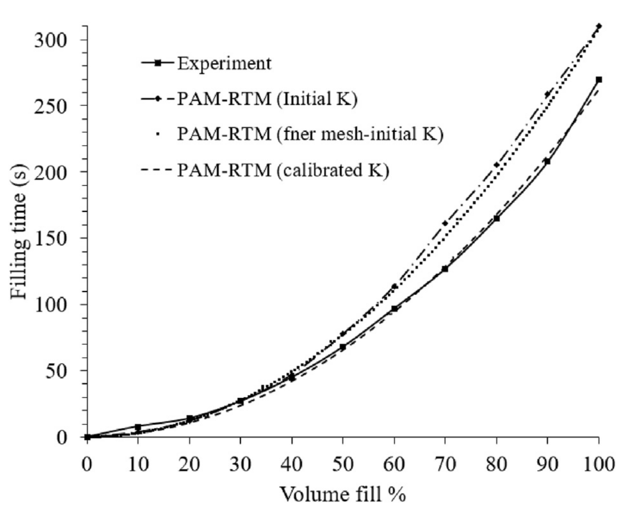


Fig. 5 – Resin infusion experiment vs. PAM-RTM simulation.

Values of fill time directly depend on the fibre volume fraction and permeability [11]. As it can be seen from the data, the fill time prolonged at each step when the resin flowed towards the outlet region, and the time versus infused position plots are second order polynomials, which is expected in the vacuum infusion experimental observations.

3.3. *Optimization of permeability using PAM-RTM and process simulation (PAM-RTM and myRTM)*

Normally, commercial software tools are used to reduce the cost associated with experimental materials and labour, and in this case a resin infusion method was calibrated with PAM-RTM simulation to obtain the exact permeability value without performing RTM fabrication test to measure the permeability. PAM-RTM, a mould filling simulation, works in accordance with two governing equations i.e. Darcy's and continuity equations [29].

Fig. 5 displays the PAM-RTM simulations versus experiment plots and it was found that the initial prediction without calibration of permeability resulted in longer filling times for the volume fill percentage of 50 and above. For the initial K-PAM-RTM results, eleven and thirty-one nodes were selected along the width and length for the meshing purpose whereas for initial permeability value with a finer mesh (K-PAMRTM-finer mesh), twenty-one and sixty-one nodes were chosen, respectively. As seen in the behaviour of finer mesh, filling time was slightly closer to the experiment. Therefore, a finer mesh was utilized for the preform to predict the flow front position with the calibrated permeability (0.85 e^{-10} m^2) extracted from the PAM-RTM simulation. Hence, since the behaviour of reinforcement in terms of permeability for the current thickness and resin viscosity was obtained, and that the filling time can precisely be predicted for large and complex shapes using PAM-RTM software tool. The slight increase in the K value after calibration could be due to the presence of peel ply on the preform in the resin infusion test, which is neglected in the simulation. The simulation results at different volume fill percentages are shown in Fig. 6 and the pink colour illustrates the flow front progression. In the following discussions, the absolute accuracy of calibrated permeability would be validated by the prediction of fill time using another software package and analytical solution.

For myRTM simulation, the preform was first deigned in CATIA V5 and then was imported to the gmsh software tool for meshing before running the myRTM simulation. The

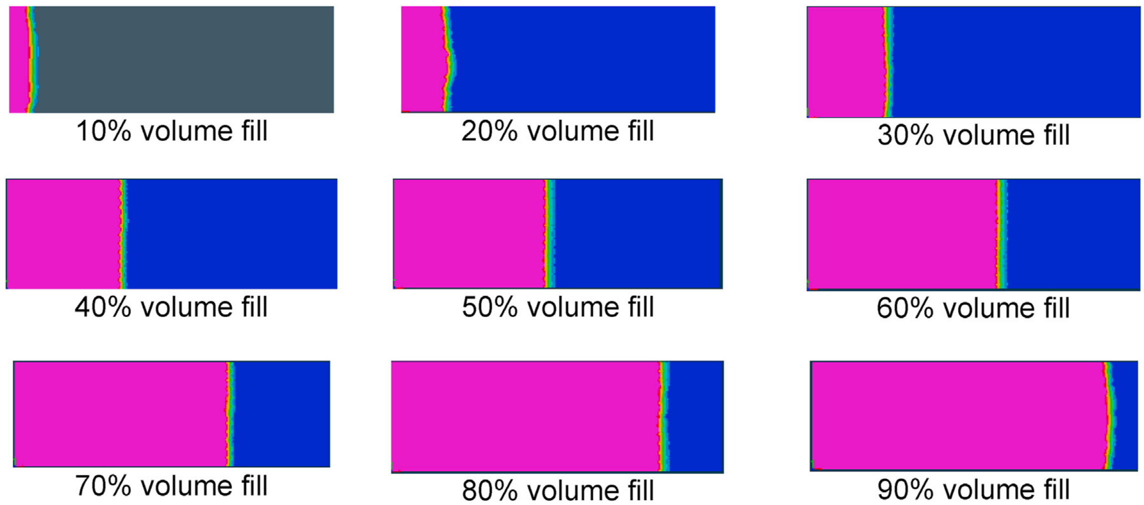


Fig. 6 – PAMRTM simulation results of rayon fabric composite plate (twenty-one nodes along the width and sixty-one nodes along the length).

program is based on the Cellular Automata principle to predict the resin flow position and is known as a preliminary process design package [12]. Fig. 7 shows the filling time percentage and the volume fill percentage of the resin infusion process and myRTM simulation from the beginning state until the end. When the simulation was run with the initial K value, the result did not match well with the experimental work whereas myRTM simulation run with the K value, obtained by PAM-RTM software tool, was found to accurately predict the filling time percentage. Fig. 8 displays the progression of resin from 40% to 90% volume fill in the rayon preform during infusion simulated by myRTM software. The software was successfully used in another work to predict the state of the resin in the preform where a bent composite part was manufactured [30].

3.4. *Analytical solution for filling time*

A unified analytical solution was gathered for resin infusion process [11]. Expressing the pressure gradient at the flow front governs progression of flow front and noting fluid pressure as a function of dimensionless flow coordinate (α), which is the ratio of in plane flow direction (x) over the instantaneous flow front location (L) [11], Darcy's law for resin infusion is obtained as

$$\frac{dL(t)}{dt}=\frac{1}{\mu}\left(\frac{K}{\varnothing}\right)_{\alpha=1}\left[\left(\frac{dP}{d\alpha}\right)_{\alpha=1}\frac{1}{L(t)}\right] \tag{2}$$

where $(dP/d\alpha)_{\alpha=1}$, $(K)_{\alpha=1}$ and $(\varphi)_{\alpha=1}$ represent the pressure gradient, permeability and porosity at the flow front ($\alpha = 1$). The fill time is yielded after integration:

$$t_{infusion}=\frac{\mu}{2}\frac{L^2}{\left(\frac{K}{\varnothing}\frac{dP}{d\alpha}\right)}_{\alpha=1} \tag{3}$$

The material data available in Table 1 was used to obtain the fill times at different volume fill % from Eq. (3). Fig. 9 presents the plots for both experiment and developed analytical solution and it can be found that the prediction is very accurate. The plots of times versus flow front position are second order polynomials [11].

Hence, it was determined that all three means of predicting the flow front position of Elium® resin in the rayon preform (i.e. PAM-RTM, myRTM and analytical formulation) were matching the experimental result. This can ease the initial experimental evaluation of vacuum infusion in particular for long components, and product developers can have an estimation of production process and adjust the parameters e.g. inlet gates and spiral location to control the manufacturing in such a way required.

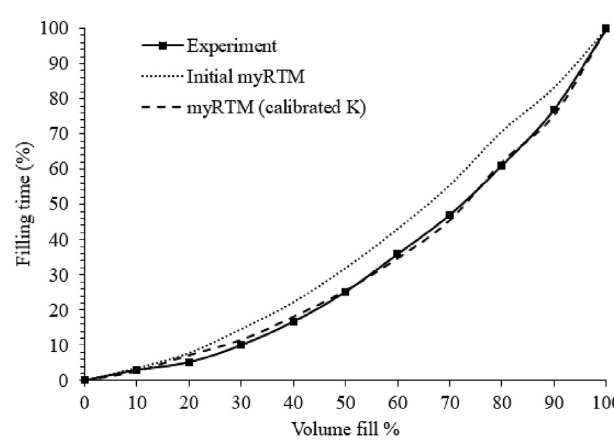


Fig. 7 – Experimental filling time compared to myRTM simulation of resin infusion process.

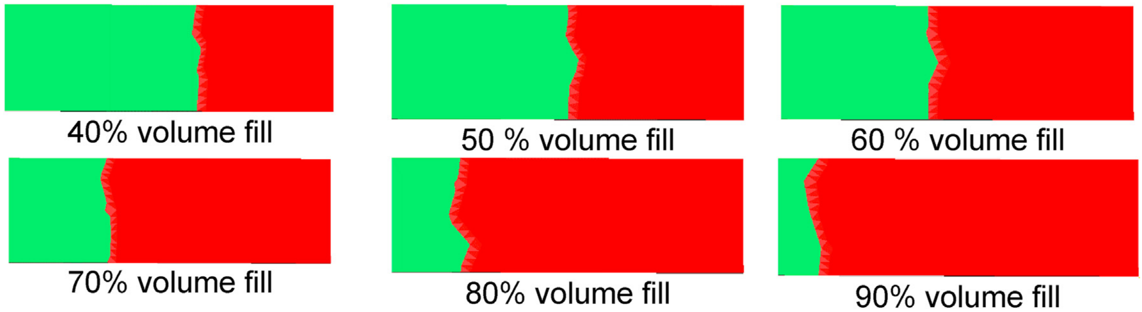


Fig. 8 – myRTM simulation from 40 % to 90% volume fill.

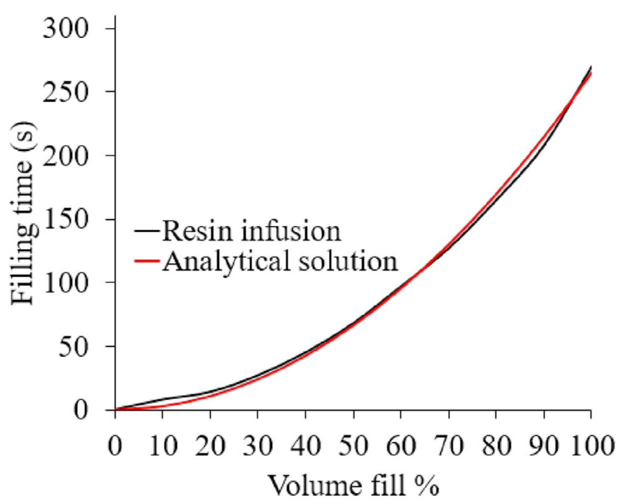


Fig. 9 – Comparison between experimental filling time and the analytical solution, Eq. (3).

4. Conclusions

The permeability and resin infusion tests were successfully carried out for rayon fibre reinforced Elium® composites. The required data was obtained and used for simulations and an analytical solution. It was found that PAM-RTM commercial software package is a very beneficial tool for prediction of rein infusion behaviour and calibration of permeability values. Simulation results were compared against the analytical solution and a very good agreement in terms of the prediction of flow front time at different locations along the preform was demonstrated. This demonstrated the potential of simulation and analytical formulations for industrial usage with regards to process optimization and efficiency. Rayon showed its capability to be processed for resin infusion applications with a liquid thermoplastic resin. The wood-based cellulose fibres are an important resource from the Swedish forest industry which can be used as pulp-based rayon fabrics as bio-based reinforcement material. The combination of permeability and resin infusion experiments together with the PAMRTM simulation for a simple geometry can provide a solid approach to achieve precise permeability values in order to design the processing for complex and large parts. The analytical solution can also be used in such a way to complement and validate the processing design.

In the next step the tensile and flexural performances of this developed composites are compared with other types of natural fibre composites investigated thus far. This demonstrates the potential of replacement of jute and flax fibres with rayon fabrics in countries which have forest industry and can get advantage of their natural resources, in particular in the Nordic region.

Declaration of Competing Interest

The authors declare no conflict of interests.

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