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PROBABILISTIC ANALYSIS OF PROPERTY UNCERTAINTIES USING RESIN INFUSION FLOW MODELING AND SIMULATIONS – RESIN VISCOSITY AND PREFORM **PERMEABILITY**

by

Henok Shiferaw

A thesis submitted to the graduate faculty in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE

Department: Computational Science and Engineering Major: Computational Science and Engineering Major Professor: Dr. Ram Mohan

> North Carolina A&T State University Greensboro, North Carolina 2011

This is to certify that the Master's Thesis of

Henok Shiferaw

has met the thesis requirements of North Carolina Agricultural and Technical State University

> Greensboro, North Carolina 2011

> > Approved by:

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DEDICATION

I dedicate this work to my parents and my two younger brothers. Without their support, this work would not have been possible.

BIOGRAPHICAL SKETCH

Henok Shiferaw was born on September 13, 1985 in Addis Ababa, Ethiopia. He joined North Carolina Agricultural and Technical State University, Greensboro, NC in 2005 to pursue a Bachelor of Science degree in Manufacturing Technology and received his degree in May 2009.

In 2009, he joined College of Engineering to pursue Master of Science in Computational Science and Engineering at North Carolina Agricultural and Technical State University, Greensboro, NC.

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I would also like to acknowledge with sincere gratitude the members of my thesis committee, Dr. Ajit Kelkar and Dr. Vinaya Kelkar for their valuable suggestions and comments on my work, in particular Dr. Vinaya Kelkar for all her contributions and guidance with the probabilistic analysis and SPSS. My gratitude is also expressed to Dr. Ron Bolick for teaching me the manufacturing and testing processes for composites. Special thanks go to Ms. Lydia Leak of CSE Department for her invaluable help and support.

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ABSTRACT

Shiferaw, Henok. PROBABILISTIC ANALYSIS OF PROPERTY UNCERTAINTIES USING RESIN INFUSION FLOW MODELING AND SIMULATIONS - RESIN VISCOSITY AND PREFORM PERMEABILITY. **(Advisor: Dr. Ram Mohan),** North Carolina Agricultural and Technical State University, Greensboro

Physics based flow modeling provides an effective way to simulate the resin infusion process in liquid composite molding processes for polymer composite structures. These are effective to provide optimal injection time and locations for given process parameters of resin viscosity and preform permeability prior to resin gelation. However, there could be significant variations in these two parameters during actual manufacturing due to differences in the resin batches, mixes, temperature, ambient conditions for viscosity; in the preform rolls, compaction, etc., for permeability. The influence of uncertainties in these parameters on the resin infusion time is investigated via a probabilistic modeling methodology using resin flow modeling and statistical analysis. The probabilistic methodology built upon computational analysis and tools for mesh generation, resin flow modeling, statistical analysis and visualization is presented. The application of this methodology for individual and simultaneous variations of these two parameters is presented, along with experimental comparisons validating the flow modeling. The probabilistic modeling methodology resulted in confidence envelopes to determine the probability for successful resin infusion prior to gelation, and estimate infusion time for any combination of viscosity and permeability for a composite part and injection condition. The effectiveness of these confidence envelopes to determine the probability for resin infusion success and estimate the infusion time without a need for additional simulations is presented.

CHAPTER 1 INTRODUCTION

1.1 Composites

Composites are combinations of two or more materials embedded in another material called matrix. This combination offers properties, which are superior to individual component properties. Composites are known for their high weight specific mechanical properties and are therefore used in numerous lightweight engineering applications. Their high strength to weight ratio, high creep resistance, high tensile strength and high toughness are the major reasons behind the use of composites in different applications. These materials are used not only in aircraft industry, but also in civil, mechanical and other application areas.

In general, there are three types of composites:

- 1. Particle-reinforced composites: Figure 1.1 illustrates a particle reinforced composite. In this, iron carbide particles are embedded in an iron matrix, and carbon particles embedded in a rubber matrix [1].
- 2. Fiber-reinforced composites: Figure 1.2 illustrates continuous aligned fibers, discontinuous aligned fibers, and discontinuous random oriented fibers in a matrix [1].
- 3. Structural woven fiber composite: Figure 1.3 illustrates structural woven fiber matrix configuration [1].

Figure 1.1. Illustrative example of particle reinforced composites

Figure 1.2. Illustrative example of Fiber orientation in fiber reinforced composites

Figure 1.3. Illustrative example of woven fabric structural composites

1.2 Liquid Composite Molding (LCM) Process Design

For a wide variety of industries, Liquid Composite Molding (LCM) manufacturing processes have evolved as an appealing method of producing composite components [7]. LCM involves a family of molding processes where the reinforcement fibers are placed into a mold defining the component geometry, and then the liquid resin matrix is introduced. The composite is then cured in the mold, developing into a near net shape composite component [6].

As shown in Figure 1.4 LCM process can be broken into four basic steps. First, reinforcement is placed into a mold defining the component geometry. The mold is closed, capturing the reinforcement into the mold cavity. Many times, to achieve acceptable component volume fraction (ratio of fibers to matrix) as specified by the design, the reinforcement must be compacted from its natural resting state. Therefore, closing of the mold is often termed the compaction phase. Next, the injection phase involves the forcing of liquid polymer resin into the mold cavity, filling the mold and saturating the reinforcement. Injection can take place through the use of fluid pumps producing positive pressure or through much simpler means of being drawn in by an induced vacuum. The fourth phase is the cure cycle and the complexity is dependent upon the resin chemical reaction requirements and may include the need for heating or cooling of the mold system. As a result, resin cure cycle and exothermic behavior directly influence the mold design. Last, the de-molding phase involves separation of the final cured component from the mold [6].

The variants of the liquid composite molding process include: Resin Transfer Molding (RTM) with two sided mold configurations, VARTM: vacuum assisted RTM with a flexible one-sided tool to corn the net-shape woven fiber preform, H-VARTM, etc [6].

Figure 1.4 Liquid composite molding process [2]

Even though the LCM process is relatively new compared to traditional manufacturing processes, the general LCM product development process is similar to other products involving several key steps — product definition, geometry design, prototype production and process optimization.

The activities involved in the LCM product development typically include, cost, static, dynamic and thermal analysis as well as geometry design. From the composite processing/manufacturing point of view resin infusion is a critical step in LCM process. The success of this manufacturing process depends upon complete resin infusion prior to gelation. To aid in the understanding and analysis of resin flow infusion, numerous LCM physics based flow simulation analysis packages have been established, for example Mohan *et. al*. FERTM, LIMS, RTM-Worx, PAM-RTM [3-5].

Currently, most resin infusion optimization work is based on the development of the optimal operation setup in terms of the shortest filling time and minimizing injection pressure, injection location, etc. In many cases, these optimized parameters cannot be accurately controlled in the manufacturing process and in a production environment due to inherent materials and process variations. These result in the variations during the resin infusion resulting in inconsistent part quality for the same composite part and injection conditions. In the present work the statistical variations characteristics of the two key factors involved in the LCM flow infusion process parameters, particularly preform permeability and resin viscosity are investigated.

1.3 Problem Statement

For LCM processes, substantial flow infusion process modeling and optimization research has been conducted over the years. Several researches have been based on onedimensional mold filling configurations, thin-shell molds, and three-dimensional models that simulate the mold filling, heat transfer and curing stages [8]. For process flow optimization, Spoerre *et al.* [9] utilized the genetic algorithms (GA) in conjunction with the cascade correlation neural network architecture (CCA-NN) to build a model to predict and optimize performance and quality of LCM/RTM parts. From resin infusion simulation models, we can understand the resin infusion during the LCM process, and the factors that significantly affect the part quality determined by both quantitatively and qualitatively by successful resin infusion. However, in practice, variations in process and property parameters that influence the infusion process are inevitable that severely impacts the repeatability of optimal tooling design during the actual manufacturing process. Ranganathan *et al*. [10] described that nonuniform raw material quality, improper preform preparation, loading, and mold assembling result in variations in preform microstructures and handling conditions, which often make the permeability and viscosity largely different under the same theoretical circumstances. Other error sources in RTM processes may exist in the skill level of the operator, mold temperature or fiber material quality. Pan *et al*. [11] developed an experimental method to measure the fiber permeability and found that the probability distribution characteristics of perform permeability is actually normal distribution.

Key parameters that influence the resin infusion are resin viscosity and preform permeability. Variations in resin viscosity and permeability will affect the completion of resin infusion prior to resin gelation. In this thesis, a probabilistic based modeling

methodology for understanding of the preform permeability and resin viscosity variations for a given composite part configuration and infusion condition is presented. The probabilistic methodology takes into account the combination of variations in fiber preform permeability and resin viscosity and analyzes their effect on the resin infusion time statistically. SPSS statistical analysis software is used to generate statistically distributed variations and uncertainties for permeability and viscosity. The effect of these variations on the resin infusion fill time is analyzed using the resin flow infusion modeling analysis software FERTM developed by Mohan *et. al*. These predicted resin infusion times are analyzed for the success of resin infusion prior to resin gelation. These analysis are used for the generation of a confidence envelop that can be used estimate the success of resin infusion prior to gelation under any given conditions of these two process parameters, and the associated estimated resin infusion time, without a need for another flow modeling and simulation with these specific process parameters.

1.4 Research Objectives

The goals of this thesis work are:

- To present and demonstrate a probabilistic modeling methodology for analysis of process uncertainties during resin infusion in Liquid Composite Molding employing resin infusion process flow modeling and simulation.
- Investigate the stochastic property variations of two of the key input parameters preform permeability and resin viscosity on the resin infusion time, for a given composite part and injection conditions.
- Obtain confidence level curves for the success of resin infusion prior to resin gelation

that are influenced by the process parameter uncertainties in permeability and viscosity.

• Demonstrate and present the methodology in a simple and complex composite part configuration.

CHAPTER 2 LITERATURE REVIEW

2.1 LCM Mold Filling Simulation

Liquid Composite Molding (LCM) processes stretch from the traditional Resin Transfer Molding (RTM) to vacuum-assisted RTM (VARTM), resin film infusion (RFI), Seeman's Composite Resin Infusion Molding Process (SCRIMP) and other RTM variations [12]. No matter how complex these liquid composites molding (LCM) techniques are, they involve similar basic processes: mold filling and resin curing. Considerable research attention has been given to analyze, predict and simulate the behavior of resin flow inside the mold, with mold filling considered as the process of flow through porous media, which has a governing effect on the final microstructure and overall quality of the composite parts. The success of the manufacturing process depends on the successful infusion of resin without any dry spots that are resin unwetted regions prior to gelation.

Gonzalez *et al.* and Chan *et al.* studied 1-D isotropic RTM resin infusion model in a disk-shaped mold and a rectangular mold, individually [13, 14]. By neglecting the chemical reaction and heat transfer during the filling stage, both analytical and numerical methods were utilized to simulate flow process.

Porous media flow approach based on Darcian flow through a porous media, was used by many researchers [15 18]; to model more complicated thin shell 2.5D and 3-D flow in complex mold configuration. Some of the past work considered not only heat transfer but also curing and rheological changes for both isotropic and anisotropic preforms. However multi-physics models are still limited in scope.

Several methods including Finite Difference Method (FDM), Finite Element Method (FEM) and Boundary Element Method (BEM) were utilized by researchers for the computational modeling of the resin infusion process based upon the mathematical models of resin flow. The mathematical models are a set of partial differential equations for the process variables and keep track of the moving flow front during resin infusion. To simulate a twodimensional RTM flow process, FDM was one of the first attempts used. By comparing with experimental results, it was proven that due to edge effects, the computing errors were over a reasonable range, which limited further application. Um *et al.* [18] applied the boundary element method. Their case was two-dimensional flat molds in which the permeability and the resin viscosity were constant. They reported that it took less time to generate mesh at each time step than required by FDM or FEM. Yoo *et al.* [19] and Osswald *et al.* [20] determined that under the limitations of simple geometry parts and isothermal Newtonian flow conditions, the BEM method gave very accurate simulation results. Finite element and control volume (CV), i.e. FE/CV, to solve for the associated process flow variables and for the tracking of flow front inside the mold cavity employing Eulerian computational mesh, is a common method that has been applied by several researchers [21-24]. Lagrangian deforming mesh approaches where the flow computational domain evolves with the resin front advancement requires the computational domain to be redefined and the computational mesh generated, resulting in a very time-consuming procedure. A major advantage of FE/CV or other Eulerian mesh approaches is that the simulation of the flow front can be conducted without re-meshing the filled regions, although flow front changes continuously during resin infusion in the preform mold configuration. Joshi *et al.* [24] concluded that three major steps are needed in the FE/CV flow simulations: (1) use the FE solution to obtain the pressure

distribution in the resin-filled region; (2) calculate the resin flow rates; and (3) trace the resin flow front, employing a CV methodology.

Youssef *et al.* developed an interactive simulation technique in which during the resin flow simulation process, the user can: (1) change the locations of the inlet and vents; (2) remove, open and close inlet and vents; and (3) change the inlet pressure or flow rate at the inlets.

FE/CV approaches, though effective are computationally restrictive in the time step increment that can be utilized. The transient flow problem is treated as a quasi-static problem and the flow is advanced by time step increments at each quasi-steady state. This resulted in limiting the time step increments to ensure stability of the computational solution though such time step resolutions are not needed and significantly increased the computational cost for large composite simulation. An effective simulation methodology with efficient computational and physical attributes is the pure finite element method originally developed by Mohan *et al*. [25, 26]. The pure finite element methodology is based on the transient mass conservation equations for the analysis of flow through porous media in which both the flow field variables (pressure, P) and the state variable (fill factor defining the infused state) are solved in an iterative manner [25, 26].

Finite element method is used to solve many different kinds of engineering problems, but the focus of this study is the simulation of the resin flow infusion in liquid composite modeling process. The simulation of LCM flow process involves the isothermal process flow solution of the conservation of resin mass as the governing equation in finite element computational developments. In the pure finite element method the governing equation is discretized and the fill factors and pressure values are solved in an iterative manner [25,26].

The fill factors define the state variable and pressure defines the field variable. This method is followed and is the basis of the 2.5 D thin shell flow modeling code (FERTM) employed in this work. The pure finite element method is described briefly next.

2.1.1 Resin Mass Conservation

Following the discussions in reference [25,26], the resin flow through the fiber preform contained within the mold cavity is represented by the transient mass conservation equation. The physical mass conservation equation (formed by coupling the mass conservation equation with the momentum equation via Darcian velocity field) is given by

$$
\frac{d}{dt} \int_{\Omega} \Psi d\Omega = \int_{\Omega} \nabla \left(\frac{\overline{R}}{\eta} \nabla P \right) d\Omega \tag{2.1}
$$

where, \overline{K} is the permeability tensor, η is the resin viscosity, P is the pressure field, and Ψ is a state variable representing the infused state of the resin [25, 26]. Further details are available in reference [25] and [26]. Finite element discretizations are employed for both pressure and fill factor and the resulting linear system of equation are solved in an iterative manner.

2.2 Optimal LCM/RTM Process Design

The most important procedure in a typical RTM process cycle is mold filling. To wet out the reinforcement preform the resin is injected and driven by the pressure. During this segment, many process factors are involved, such as location and size of gates and vent, injection pressure and mold temperature. Designing optimal RTM processes in terms of minimizing cycle time avoiding dry spots, and increasing the yield of successful parts has been done in this field via process flow modeling and simulations.

Lin *et al.* [27] discussed the strengths and weaknesses of the genetic algorithm and

the gradient based methods. Two different types of RTM process optimization have been documented. In first case, the Quasi-Newtonian method was coupled in the code Global Local Optimizer (GLO), and gate locations were optimized to minimize the filling time. In the second case, a graphical search was explored for adding the varied high permeability layers to minimize resin waste in addition to minimizing the filling time. They reported that these two methods have their specialties, and if the design variables are discrete, for example number of gates and vents, the combination of two methods should be used. In addition, they also pointed out the limitation of finite element method used in analysis, i.e. the noticeable error was incurred if a single node was used to model the gate.

A design and control methodology was established by Lawrence *et al.* [28]. In this work, by using sensor and actuators, the flow disturbance was identified and the resin flow was redirected to complete the mold filling without any void. The researchers developed software for defining the position of the sensors in the mold to identify disturbances and suggest flow control actions for adding actuators at auxiliary locations to change the direction of flow. To validate the effectiveness of the methodology, they tested complex mold features including rib structure, thick regions, and tapered regions. They documented that the feedback from sensors did have the ability to automate and actively control the flow of the resin, which led to consistently impregnating all the reinforcements even though disturbances were present in the process.

Jiang *et al.* [29] initiated a new mesh distance-based method in genetic algorithm. The basic idea for this technique was to find the optimum arrangement of gates and vents to achieve the objective of minimizing the maximum distance between gates and vents to avoid dry spot formation. By comparing with the examples available in the literature, it was found that this method was very efficient and effective in optimizing the locations of gates and vents and saving computational time. However, limited work exists to understand the uncertainties and variations that can exist in the key process material parameters even when optimal injection configurations are employed. Brief discussions of the two key parameters that influence resin infusion, namely, preform permeability and resin viscosity are presented next.

2.3 Permeability Measurements and Characterization

Resin flow and permeation through fiber preform in liquid composite molding is governed by many process parameters, such as resin chemistry and rheology, injection pressure, mold temperature, fiber reinforcement microscopic and macroscopic structure, and mold complexity. All of these parameters influence the resin propagation and successful infusion prior to gelation in a LCM process and impact the predicted flow progression and infusion time in flow modeling and simulation. Any deviations from these parameters employed in the flow simulation during actual processing will impact the resin progression and infusion time on any given day in the production process. It is thus essential to understand the effect of the variations and uncertainties of these influencing process parameters. Among these parameters, preform permeability, the physical property of the fibrous material, indicates the resistance to the pressure driven flow affecting the resin progression pattern inside any given composite part configuration and infusion time. Permeability is measured by a mathematical model of Darcy's law, plays a crucial role in the success of resin infusion and is a key parameter in LCM flow simulation analysis. The filling time and flow progression pattern depends heavily on the preform permeability in the

composite part geometry and its variations. Complex composite parts have fiber preforms oriented in various directions, presence of bends etc, that results in variations in the permeability's within the composite part geometry.

2.3.1 Theoretical Background

A porous medium is contained within a vessel, or some control volume consisting of pores between particulate phases. The fluid flow rate through this vessel or control volume is $Q(m^3/ s)$ and the cross sectional area is *A* (m^2) . Thus the superficial velocity U_0 is the total flow rate divided by the cross sectional area.

The particles existing within the vessel reduce the area available for fluid flow resulting in preserving fluid continuity with the entering superficial flow. Therefore, the fluid has to squeeze through a smaller area. This phenomenon makes velocity within the pores in the vessel greater than the superficial velocity. The volume fraction of the pores has the most important effect compared to the mass fraction. The volume fraction of solids is usually referred to simply as the volume concentration or solids fraction, and the remaining fraction is that of the voids. The void fraction is also called the porosity. The dimensionless quantity porosity (ϕ) of a porous material is defined as the fraction of the vessel volume occupied by voids as shown in equation 2.2 and 2.3.

$$
\phi = \frac{V_p}{V_c} = \frac{Volume\ of\ Pores}{Vessel\ Volume} \tag{2.2}
$$

and

$$
\phi + \varepsilon = 1 \tag{2.3}
$$

Where ϕ is void fraction and ε is solid fraction.

The porosity is usually an isotropic property that means it is the same in all directions; therefore, the interstitial velocity is simply related to the superficial velocity by the expression 2.4, which comes from a consideration of fluid continuity. Figure 2.1 shows the relationship between superficial velocity and interstitial velocity.

$$
U = \frac{U_0}{\varepsilon} \tag{2.4}
$$

where *U* is interstitial velocity and U_0 is superficial velocity.

Porous Medium Figure 2.1 Illustration of flow through porous medium

Darcy's law is the basic equation used to describe the flow behavior in porous media (1856) [30], which states that the flow rate in a porous media is proportional to the pressure gradient in the medium. The constant of proportionality is defined as the permeability, and the magnitude is a function of the pore structure (porosity or fiber volume content). The Darcy's law is valid as long as the following conditions are satisfied:

- The flow should be a laminar flow that has a low Reynolds number, and
- The fluid should have a Newtonian behavior

The fluid velocity in the porous media is thus given by

$$
\vec{V} = \frac{[K]}{\eta} \nabla P \tag{2.5}
$$

where: \vec{V} = Velocity of the flow front

 $[K]$ = Permeability tensor

 η = Viscosity of the fluid

 ∇P = Pressure gradient (∇ : gradient operator)

From expression 2.5, permeability [K] characterizes how ease that a fluid goes through the porous material driven by an applied pressure gradient. The unit of permeability is a dimension of length scale squared and the most widely employed unit is the Darcy (D): one Darcy permeability corresponds to, a pressure gradient of 1 atmosphere that produces a flow rate of 1 cubic centimeter per second of a fluid with 1 centipoise viscosity through a 1cm² cross sectional area. Other units, such as m² and in² are also widely used as well.

$$
1darcy = \frac{1\left(\frac{cm^3}{sec}\right) * 1(cp)}{1(cm^2) * 1(\frac{atm}{cm})} = 1\mu m^2
$$
\n(2.6)

2.3.2 Permeability Measurement Methods

Permeability is one of the most important factors governing resin flow through a composite preform, which makes it a critical input parameter for liquid composite molding manufacturing flow simulations for understanding the flow progression and in optimal tooling design. For most parts fabricated by RTM processes, the in-plane dimensions are noticeably greater than the thickness direction. Therefore, most research has focused on the in-plane, i.e. one-dimensional and two-dimensional flow permeability characterizations

experiments [30-38]. As the parts become more and more complex, large, thick components must be manufactured by RTM processes. Some attention has been drawn to threedimensional flow permeability experiments [39]. Three commonly used methods for one, two and three-dimensional flow permeability characterization experiments are as follows.

2.3.2.1 Unidirectional Flow Method

For one-dimensional flow, a rectangular cavity mold with an edge or line injection gate is a typical setup. Two techniques are widely applied, including saturated flow method and advancing flow front method. For the saturated permeability measurement, onedimensional Darcy's law is given as:

$$
\frac{Q}{A} = \frac{K}{\eta} \frac{dP}{dx} \tag{2.7}
$$

where Q is flow rate;

 A is cross sectional area of the mold cavity $\frac{dP}{dx}$ is the pressure gradient along the length of the fabric η is the viscosity of the fluid, and K is an experimentally derived permeability constant.

 ϕ is the porosity

Based on the flow front location (x) at any time (t) , the permeability can be calculated by the following equation:

$$
\frac{dx}{dt} = \frac{K}{\phi \eta} \frac{(P_0)}{x} \Rightarrow \int_0^x x dx = \int_0^t \frac{K}{\phi \eta} P_0 dt \Rightarrow \frac{x^2}{2} = \frac{K}{\phi \eta} P_0 t
$$

$$
K = \frac{\phi \eta}{2P_0 t} x^2 \tag{2.10}
$$

Where P_0 is the constant injection pressure. t is real filling time starting from the moment that test fluid begins to saturate the preform, *x* is the flow front location from the end injection line corresponding to t.

Figure 2.2 Diagram of flow front one – dimensional flow

2.3.2.2 Bi-directional Flow Method

In one directional flow method race-tracking is present that alters the uniform flow and does not capture the bi-directional permeability flow effect. To account for these, researchers developed another experimental method based on a circular mold configuration for permeability characterization. This is based on the analytical solution based on Darcy low with a test fluid injection node in the center of the mold. For the isotropic case, the flow front advanced as a circular shape, and the following solution was proposed:

$$
\left(\frac{R_f}{R_0}\right)^2 \left[2\ln\left(\frac{R_f}{R_0}\right) - 1\right] + 1 = \frac{4k\Delta pt}{\phi \eta R_0^2}
$$
 (2.11)

where: R_f = Flow front radius at time t;

 R_0 = Inlet radius (The radius of the circular hole where the reinforcement stacks is cut through at the center injection point;

 Δp = Pressure gradient;

- $t =$ Elapse Time
- η = Test fluid viscosity;
- ϕ = Porosity;
- K = Permeability ($K = K_x = K_y$).

For the anisotropic case, $K_x \neq K_y$ results in the flow front having an elliptical shape.

Several simplification methods have been presented in the literature for this case.

Figure 2.3 Illustration of flow front for bi-dimensional flow

2.3.2.3 Out-of-Plane Flow Method

The most common method to measure permeability in the out-of-plane or through thickness direction is one directional channel flow apparatus. Also as it does in one and twodirectional flow, Darcy law plays a prevailing role in computing the permeability value in the our-of-plane flow method. Figure 2.4 illustrates the through thickness, out-of-plane flow configuration for obtaining through thickness preform permeability.

Figure 2.4 Illustration of flow front for through thickness flow

2.4 Resin Viscosity Measurement and Characterization

A typical polymeric composite material is composed of fiber reinforcements and a polymer resin matrix. The reinforcement provides strength to the composite structure, while the function of the matrix is to bind the reinforcements together and transmit load between the individual reinforcements. The matrix can be either a thermoplastic or a thermoset polymer. Thermoset resins and thermoplastic resins differ in molecular structure. Thermosets are crosslinked and thermoplastics are not crosslinked. Thermoset resins are converted from a liquid to a solid using an initiator or heat and the process is irreversible. Thermoplastic resins are melted and formed and can be re-melted and re-formed and the process is reversible. The most commonly used thermosetting polymers are unsaturated polyesters, epoxies, vinyl esters, polyurethanes, and phenolics. Among the various resins, unsaturated polyester (UP) is most widely used, representing about 80% of the total resin used in the thermoset composite market [40].

2.4.1 Theoretical Background

LCM processes utilize liquid resins of a thermosetting type such as epoxies, polyesters, polyamides, and vinyl esters. The type of resin used for a given application is dependent upon many factors such as performance, strength, cost, and viscosity. When designing a LCM process, viscosity and cure kinetics heavily influence the injection time and total cycle time. Cure kinetics determines the amount of time before gelation and the total time required for complete cure. Resin viscosity is an important factor determining injection time. More viscous resins require higher pressures to maintain the same injection time as lower viscosity resins. In Table 2.1 the viscosities for common polymer resins used in liquid composite molding (LCM) processes are given.

Table 2.1 Viscosities for most common polymer resin

Resin	l'vpe	Viscosity Range (cps ω 25°C)
Dow DERAKANE 411-350	Epoxy vinyl ester	350
Dow DERAKANE 510N	Epoxy vinyl ester	250
Nuplex PP8476	Polvester	200

2.4.2 Resin Viscosity Measurement Methods

The resin viscosity is generally a function of the degree of cure α and the temperature T

$$
\eta=\eta(\alpha,T)
$$

The viscosity decreases with increasing temperature, until a significant degree of cure due to chemical curing reactions of the resin is achieved. The viscosity increases with increasing degree of cure, which again is temperature dependent. Both temperature and degree of cure are functions of time.

Models frequently used for description of the thermo-reactive resin viscosity are

$$
\eta(\alpha, T) = A \exp\left(\frac{B}{T}\right) \exp(C\alpha) \tag{2.11}
$$

and Castro and Macosko Model:

$$
\eta(\alpha, T) = A \exp\left(\frac{B}{T}\right) \left(\frac{\alpha_g}{\alpha_g - \alpha}\right)^{C + D\alpha} \tag{2.12}
$$

With constants *A*, *B*, *C*, *D* and α_g is the degree of cure of the resin at the gel point, at which the state of the resin changes due to chemical cross-linking from viscous liquid to gel-like semisolid. It is essential for the success of LCM, resin infusion has to be completed prior to initiation of cure reactions. During manufacturing, appropriate resin inhibitors are added to delay the resin kinetics. This provides a time duration in which the resin remains in liquid flow state providing a "pot-life" for a resin system. For the calculation of viscosity after the initiation of curing, T and α need to be given as functions of position and time.

It is clear from the above discussion that the two key influencing parameters for the success of resin flow infusion are preform permeability and resin viscosity. The flow infusion step in LCM is to be completed prior to gelation and the resin viscosity is generally constant during this stage. The flow simulations are thus based on an isothermal Newtonian resin viscosity in the present study. The flow simulations thus do not taken account any temperature variations during the resin infusion. Most temperature changes occur after the completion of infusion.

2.5 Statistical Analysis

In the present work, normal distribution is used to generate the viscosity and the permeability data variations that could occur. Normal distribution is a mathematical model with the function:
$$
f(x) = \frac{1}{\sqrt{2\pi\sigma^2}} e^{-\frac{(x-y)^2}{2\sigma^2}}
$$

that fits many real life data. Fundamentally a normal distribution is when a set of values for any variable, when displayed in a histogram or a line graph is unimodal has one peak (mode) and looks like a bell shape. An example of a normal distribution is shown in Figure 2.5. In this illustration, the mean height of a population is 5 feet 8 inches with the other height values in the population distributed around this mean.

Figure 2.5 Example of normal distribution of data using height. [41]

The basic characteristics of a normal distribution function are: smooth, bell-shaped curve, symmetric about the mean, asymptotic tails, the median, mean, mode are the same value, and the area under the curve is equal to one or 100%. The data are symmetric about the mean as shown is Figure 2.6, and the standard deviation determines the data spread. A small standard deviation makes a tall, thin curve; and a large standard deviation make a flat, low curve. In a normal distribution curve, approximately 68% of the data falls within ± 1 standard deviation

of the mean, 95% of the data falls between ± 2 standard deviations of the mean, and approximately 99% of the data falls in between ± 3 standard deviation of the mean.

Figure 2.6 Example of standard normal distribution as related to its standard deviation [41]

A normal distribution is defined by two parameters ' μ ' and ' σ ', which represent the mean and standard deviation of the distribution respectively. In a standard normal distribution, a Z_{α} -score represents the 100(1- α)th percentile. Different types of normal data are often standardized to enable comparisons. A normal variable can be standardized using the following formula creating a standard normal variable $Z \sim N(0,1)$:

$$
Z = \frac{x - \mu}{\sigma} \tag{2.14}
$$

where: $x = original Normal (μ, σ) variable$

 μ = the mean value of the original variable,

 σ = the standard deviation of the original variable.

CHAPTER 3

PROBABILISTIC ANALYSIS METHODOLOGY

In this work the effect of the uncertainties and variations in two key parameters (resin viscosity and preform permeability) on the infusion time and flow front progression for a vacuum based resin infusion process is studied using resin flow modeling simulation. For a given composite part configuration and injection condition, the resin infusion time is dependent upon the variation in the resin viscosity during the actual infusion and the permeability variations in the preform used.

The success of resin infusion depends upon the complete infusion of dry fiber preform prior to gelation. Any resin system has a certain gelation time or pot life of the resin, and it is important to complete flow infusion prior to this gelation time. The resin flow infusion time depends upon the resin viscosity and preform permeability. Resin infusion process modeling simulation allows the determination of optimal injection conditions that can guarantee successful infusion before resin gelation. However, these are based on specific resin viscosity and preform permeability conditions employed in the simulation. Any variations from these can lead to infusion times different than the predicted time in the actual manufacturing process. The present work analyzes such variations based on a probabilistic modeling methodology to develop confidence envelope employing process flow modeling simulations. A flow chart of the probabilistic analysis methodology is illustrated in the Figure 3.1. The various computational modeling analysis tools employed are also identified in this figure. These are (1) mesh generation (ANSYS), (2) FERTM, (3) SPSS, and (4) TecPlot

Figure 3.1 Flow chart for probabilistic analysis methodology

3.1 System development

Typical activities that are involved in the production of LCM process includes: geometric design of the given part, static, dynamic and thermal analysis during process and servicing, process optimization and simulation. The variations and uncertainties in the process parameters can be analyzed and understood following the probabilistic methodology shown in figure 3.1. The methodology applicable for any composite part and processing configuration involves the following steps.

- Obtain optimal injection conditions for a composite part geometry, preform and permeability conditions. The computational finite element mesh geometry is generated using ANSYS. Resin flow infusion modeling simulations were performed using an in-house resin flow modeling analysis code FERTM.
- Generate the parameter space data for the variations in two key parameters, namely permeability and resin viscosity. Variable values for permeability and viscosity were generated with normally distributed errors around a fixed mean using statistical analysis software SPSS.
- Perform resin flow modeling simulations using FERTM for various distributed values of resin viscosity and preform permeability, and obtain the corresponding distribution of predicted resin infusion time. These sets of simulations were conducted using LCM resin flow modeling analysis code FERTM.
- Obtain the probability for successful resin infusion prior to resin gelation. This requires the simulated resin infusion time under the different permeability, resin viscosity variations to be less than the resin gelation time. Statistical analysis software SPSS was employed for this analysis.

The probabilistic methodology discussed above is applied and demonstrated for two composite process geometry configurations based on:

- 1. Simple composite flat plate model geometry
- 2. Complex helicopter complex model geometry.

3.1.1 Probabilistic Modeling Methodology Applications

We studied two different composite geometry configurations; a simple 2D composite 2d model and a complex 3D helicopter composite part. Initially to demonstrate the methodology presented in Figure 3.1 and verification of the probabilistic modeling approach employing flow simulations, we use the simple composite flat plate model. Subsequently the same approach and methodology as presented the flow chart in Figure 3.1 is extended and demonstrated for the complex 3D model. The discussions of the probabilistic methodology application to a simple composite flat plate geometry are presented next.

3.2 Application 1: Composite Flat Plate 2D Model

As shown in Figure 3.2, the simple composite flat plate is 20" x 10" with 0.2" thickness. The computational domain consisted of a quadrilateral finite element mesh with 325 nodes and 288 thin shell elements. The injection was line injection from the left end based on a constant pressure injection corresponding to the atmospheric vacuum pressure differential in vacuum resin infusion. The fiber preform permeability is taken to be 5.0×10^{-6} in² and the viscosity is taken to be 3.63×10^{-5} lbf-s/in². The flow front progression contour under these conditions is shown in Figure 3.3. The computed infusion time in this case is 135 second.

Figure 3.2 Mesh view of a 20 x 10 simple 2D composite plate model

Figure 3.3 Flow front progression in a 20 x 10 simple 2D composite plate model

3.2.1 Statistical Modeling for Permeability and Viscosity Variations

Statistical analysis software SPSS has been used to generate the viscosity and permeability value from the mean values employed in the flow simulations. We generated viscosity values that varied by $\pm 20\%$ from the mean value given by the manufacturer, and permeability values that varied by $\pm 50\%$ the mean value. Such variations could be expected in a production phase from different resin batches and fiber preform rolls. Table 3.1 shows

Viscosity (lbf-s/in²) Permeability (in²) Standard Deviation (σ) 0.34×10^{-5} 0.89 x 10^{-6} Mean (µ) 3.63 x 10^{-5} 5.0 x 10^{-6}

statistics (mean and standard deviation) for the generated viscosity and permeability values. Table 3.1 Standard deviation and mean for the generated Viscosity and Permeability values.

3.2.2 One Parameter Model - Resin Viscosity (η) Variations

To analyze the effect of the resin viscosity variations, we generated 100 normally distributed values of viscosity using SPSS. The viscosity values generated ranged $(±)$ 20% of the mean value of 3.63 x 10^{-5} (lbf-s/in²). Table 3.2.1 shows all the statistical parameters for the generated viscosity values, and as shown in figure 3.4, the histogram validated that the generated viscosity values follows a normal distribution. The permeability values can vary between \pm 50% of the mean permeability. Such variations can be expected due to variations in the preform roll, placement etc. Prior literature has shown that the permeability's can very as much as $\pm 50\%$ [6].

Table 3.2.1 statistical parameters for viscosity

Viscosity			
\overline{N}	Valid	100	
	Missing		
Mean		3.6335	
	Std. Error of Mean	.03340	
Median		3.6520	
Mode		2.85^{a}	
Std. Deviation		.33404	
Variance		.112	
Skewness		$-.118$	
Std. Error of Skewness		.241	
Kurtosis		$-.294$	
	Std. Error of Kurtosis	.478	
Range		1.48	
Minimum		2.85	
Maximum		4.33	

Statistics

Figure 3.4 Histogram for the viscosity values.

For this simple composite flat plate 2-D model, the gelation time or the resin pot life, is taken to be equal to 100 seconds ($GT = 100$ seconds) or approximately 2 minutes. In this illustration example application, even with the variations in the viscosity and permeability values, the resin infusion has to be completed before the gelation time. Resin infusion time depends on the viscosity and permeability values. As shown in the flow chart in Figure 3.1, FERTM flow modeling simulation is used to obtain the fill time for each of the generated viscosity values. The generated 100 viscosity values were employed to obtain corresponding 100 values for resin infusion time using the flow model simulations. Figure 3.5 presents the variation of resin infusion fill time for various resin viscosity values. As shown in Figure 3.5, as the viscosity values increase the fill time also increases. Subsequently, the fill time data were analyzed and a 95% confidence envelop developed for the completion of resin infusion prior to gelation time. In this case, it corresponds to a resin infusion fill time of less than or equal to 100 seconds (FT \leq GT).

Figure 3.5. Scatter diagram relationship between viscosity and fill time

Histogram

Figure 3.6 Histogram of simulated resin infusion time

Figure 3.6 presents the histogram of the computed fill time values obtained from 100 viscosity values. Figure 3.6 suggests, that the obtained fill time values correspond to an approximately normal distribution of data. By using the obtained fill time data, an envelope for the confidence level or probability for complete resin infusion for various viscosity values within the gelation time is obtained. This is achieved by using the cumulative density function (CDF) of fill time calculated within SPSS. The cumulative density function (CDF) describes the probability that a real-valued random variable, viscosity with a given probability distribution will be found to give resin infusion time values that are less than or equal to the gelation time 100 seconds (approximately 2 minutes). Figure 3.7 and Figure 3.8

present, the cumulative density function (CDF) for the resin viscosity and the corresponding fill time. From the figure, as an illustration, we can conclude that if the viscosity value is less than or equal to 3.65 \times 10⁻⁵ ($\eta \leq 3.65 \times 10^{-5}$) there is a 50% confidence interval that the fill time for this given geometry and injection conditions will be less than or equal to the gelation time of 100 second (FT \le GT). If the viscosity values are less than or equal to 3.10 \times 10⁻⁵ (η $\leq 3.10 \times 10^{-5}$) there is a 95% probability that the fill time will be less than or equal to gelation time of 100s. Clearly, reduced viscosity lead to higher probability for successful infusion prior to gelation.

Figure 3.7 Probability plot for the 50% and 95% confidence interval

Figure 3.8 Probability Vs resin infusion time plot for 95% confidence interval

3.2.3 One Parameter Model - Permeability (K) Variations.

To assess the impact of permeability (K) variations, statistical analysis software SPSS was utilized to generate 100 normally distributed values of permeability around the mean permeability used in the flow modeling. The permeability values generated ranged $\pm 50\%$ from the mean value of 5.0 x 10^{-6} (in²). Table 3.2.2 shows all the statistics for the generated permeability values, and as shown in Figure 3.9 the histogram validated that the generated permeability values follow a normal distribution.

Table 3.2.2 statistical parameters for permeability

Permability		
$\overline{\rm N}$	Valid	100
	Missing	
Mean		5.1586
Std. Error of Mean		.08341
Median		5.2731
Mode		2.78^{a}
Std. Deviation		.83406
Variance		.696
Skewness		$-.294$
Std. Error of Skewness		.241
Kurtosis		.217
Std. Error of Kurtosis		.478
Range		4.57
Minimum		2.78
Maximum		7.34
Sum		515.86

Statistics

Figure 3.9 Histogram of permeability (K)

As was used for the viscosity variations, the gelation time or the resin pot life is taken to be equal to 100 seconds (GT = 100 seconds) or approximately 2 minutes (GT \leq 2 minutes). Keeping viscosity constant, 100 FERTM simulations were completed to obtain the fill time for each of the generated permeability values. The generated 100 values of permeability were used to obtain 100 values of fill time employing the flow modeling simulations. As shown in figure 3.10 , as the permeability (K) values decrease (less permeable), the fill time increases. In the following section, the fill time data for the permeability variations were analyzed, and a 50% (as an illustration) and 95% confidence interval envelope was developed that corresponds to an infusion time of less than or equal to gelation time (GT) of 100 seconds (FT \leq GT) for the successful completion of infusion prior to gelation.

Figure 3.10 Scatter diagram relationship between permeability and fill time

To obtain the confidence level envelope, statistical analysis software SPSS is used to calculate the cumulative density function (CDF). The cumulative density function (CDF) describes the probability that a real-valued random variable (permeability (K)) with a given probability distribution will be found to give a resin infusion time less than or equal to the gelation time of 100 seconds (approximately 2 minutes). Figure 3.11 and 3.12 presents the cumulative density function (CDF) for permeability and the associated fill time respectively. It can be concluded from these figures that if the permeability value is greater than or equal to 6.56×10^{-6} in² (K $\ge 6.56 \times 10^{-6}$ in²) it presents s a 95% confidence interval that the fill time for this given geometry and injection condition will be less than or equal to gelation time of 100 seconds (FT \leq 100 GT). The associated infusion time for each probability level can be determined from Figure 3.12.

Figure 3.11 Probability plot for the 95% confidence levels for permeability (K)

Figure 3.12 Probability Vs resin infusion time plot for 95% confidence interval for permeability (K)

3.2.4 Two Parameter Model - Resin Viscosity and Permeability Variations

For this given model, the simultaneous variations in both resin viscosity and permeability effects are studied. Since the resin viscosity showed a linear variation in resin infusion time, only five values around the mean in viscosity are considered for reducing the analysis sample space for the flow modeling simulations. Using SPSS 50 values for permeability were generated within its earlier range. Using FERTM simulations, the fifty values of permeability were applied to each of the five viscosity values and two hundred fifty values of the corresponding fill time are obtained. Table 3.2.3 shows the descriptive statistical output. It is clear from this table, that the mean and median are different, especially considering the significant standard deviation. Figure 3.13 and Figure 3.14 clearly shows a skewed distribution of fill time data, even though the permeability variations showed a normal distribution. This was further confirmed using normal probability plots (QQ plots) a graphical method for assessing normality of a distribution.

Table 3.2.3 statistical value for simulated resin infusion time

Statistics

Figure 3.13 Resin infusion time Histogram

Normal Q-Q Plot of FillTime

Figure 3.14 Resin infusion time normality test

Since the normality tests demonstrate that the data are not a normal distribution, the raw values of the infusion time were converted to natural logarithmic values. As Table 3.2.4 shows, this transformation presented the descriptive statistic output where the mean and median are very close. The logarithmic transformed fill time data was further analyzed to check the normality of the data through graphical methods. Figures 3.15 and Figure 3.16 clearly shows that the natural log transformation resulted in a normal data distribution, with a closer mean and median.

LnFT		
\overline{N}	Valid	250
	Missing	
Mean		4.4219
Median		4.4067
Mode		4.19
Std. Deviation		.28274
Variance		.080
Range		1.37
Sum		1105.48

Statistics

Figure 3.15 Transformed resin infusion time

Histogram

Figure 3.16 Histogram of transformed resin infusion Time

From the normalized fill time data, the cumulative density function (CDF) and the probability to obtain resin infusion fill times that are less than or equal to the gelation time $(FT \leq GT)$ for different viscosity, permeability combinations were investigated. This was achieved through the statistical analysis software SPSS. As it is shown in Figure 3.17, a 95% confidence interval is developed for the model that is affected by both permeability (K) and viscosity (η) variations at the same time. Furthermore for this composite part and injection conditions, viscosity contribute to a 16% of the variation in resin infusion fill time (r^2 = 0.16), while the permeability contribute to 76.6% variations in the resin infusion time ($r^2 = 0.0.766$) and the supplementary 7% could be from other factors that affect the LCM simulation such as pressure, injection location, etc.

Figure 3.17 95% confidence interval for permeability (K) and viscosity (η)

The data ranges for permeability (K) and viscosity (η) that will give a fill time of less than or equal to gelation time of 100 seconds (FT \le GT) is given in Table 3.2.5 and Figure 3.18. For a resin viscosity of 2.05 x 10^{-5} , any preform permeability values of greater than or equal to 2.8 x 10^{-6} , presents a probability of successful resin infusion to be 95% for completion prior to gelation. It can also be inferred from Figure 3.18 that any combination of viscosity and permeability value below and to the right of the permeability – viscosity line corresponds to a 95% probability for successful infusion prior to gelation.

Figure 3.18 95% confidence interval range for permeability (K) and viscosity (η)

3.3 Application 2: Complex 3D Composite Helicopter Part

The probabilistic modeling methodology is now extended to a complex 3D composite helicopter part. This application also serves as the verification and validation for the resin infusion flow modeling. Flow modeling simulations were employed to find a practical and optimal injection configuration for this complex composite part, and were used in the actual infusion of a prototype composite helicopter part. Comparisons between front progressions based on flow visualization to the simulations are presented. The deviations are analyzed and modifications to the injection conditions based on the deviations experienced during actual processing are discussed. Subsequently, the application of the probabilistic modeling methodology for the analysis of two key property parameter variations for this complex composite part is presented. These discussions are organized as follows:

- Complex part geometry and mesh configuration
- Potential injection configurations and selection of practical, effective injection scheme
- Comparison of simulation and experimental flow progression and analysis
- Application of probabilistic modeling methodology for analysis of process parameter uncertainties

Figure 3.19 and Figure 3.20 shows the complex 3D helicopter model geometry and the finite element mesh configuration. The complex composite part is approximately 40" x 25" with 0.07" thickness for the preform configuration and part thickness. The computational finite element model has 2648 nodes and 5083 shell elements. A brief discussion of injection strategies in VARTM is presented next followed by the discussions on the different injection schemes studied employing the resin flow modeling simulations.

Figure 3.19 Complex 3D composite helicopter model configuration

Figure 3.20 Complex 3D composite helicopter model computational FE mesh **3.3.1 Optimal Injection Strategies**

Polyester, polyurethane, epoxy and phenolic resins are the main resin systems that are used for polymer-based composites. The reinforcements are made out of glass, carbon, and Kevlar fibers. These fibers are usually available in mat rolls (randomly distributed, long fibers) or fabrics (non crimped, woven, etc.).

In the standard liquid composite molding process, resin is injected at low pressure or at near atmospheric vacuum. This results in minimum tooling costs. It requires only a simple pump and a much less expensive mold than for similar injection processes (SMC, thermoplastics, etc.). The injection can originate from one or several injection ports (internal or external), exterior injection lines or tree-like injection channels for large parts. Figure 3.21 illustrates the most common used injection strategies that have been used for liquid composite molding.

For this composite helicopter part, to select the injection scheme that will give the minimal fill time with a simplest and practical infusion setup for injection, seven different injection strategies were considered and analyzed exclusively using flow modeling simulations as shown Figure 3.22 – Figure 3.28. Preform permeability value of 3.5×10^{-6} in² and a resin viscosity value of 5.07×10^{-5} lbf-s/in² was employed in the simulations. The vacuum driven infusion is emulated by an equivalent pressure differential of one atmosphere.

To account for the pressure loss in the feed line, a pressure gradient that linearly varies from the maximum value at the inlet is employed in all cases.

Injection configuration A has a line injection with a resin feed line in the middle of the part. This resulted in a simulated infusion time of 2072 seconds (37 minutes). The flow progression contour is shown in Figure 3.22. Injection configuration B has a line injection with a resin feed line along the left short side of the model. This resulted in a simulated infusion time of 6502 seconds (115 minutes). The corresponding flow progression contour is shown in Figure 3.23. Injection configuration C has a line injection on the right short side of the model and the infusion time is 6755 seconds (120 minutes). The corresponding resin progression contour is presented in Figure 3.24. Injection configuration D has a line injection on the upper long side of the part and a simulated infusion time of 1585 seconds (28 minutes). Figure 3.25 presents the flow progression contour in this case. Injection configuration E has a line injection on the bottom long side of the part and the simulated infusion time is 1308 seconds (23 minutes). The associated resin flow progression contour is shown in Figure 3.26. Injection configuration F has a line injection on the middle along the long side of the part and the infusion time is 432 seconds (8 minutes). The associated flow progression contour is presented in Figure 3.27. Injection configuration G has a line injection located in the two short side of the part and the infusion time is 1772 seconds (31 minutes). The associated flow front progression contour is shown in Figure 3.28. The simulated fill time for the seven different injection configuration locations as discussed above for the given model is summarized in Table 3.3.1

Figure 3.22 Injection configuration A

Figure 3.23 Injection configuration B

Figure 3.24 Injection configuration C

Figure 3.25 Injection configuration D

Figure 3.26 Injection configuration E

Figure 3.27 Injection configuration F

Figure 3.28 Injection configuration G

Subsequently injection configuration B and C gives the highest fill time and injection configuration F gives very low fill time. Due to its minimal time and simplest set up preparation, infusion configuration A is selected to use as the injection gate location.

3.3.2 Experimental and Simulation Comparisons

This complex composite part infusion configuration A was setup in our composite processing laboratory. Experimental observation of the resin infusion indicated an infusion time of 45 minutes and the following was observed. Figure 3.29 (b) presents the experimental flow front progression, during the experimental resin infusion process. It was observed that during the infusion undesired air bubbles were created. This required the pressure injection to be closed and restarted after sealing during the process. Air bubble or vacuum leaks significantly influence the resin propagation and infusion progression. This corrective action resulted in an altered and a new filling pattern that deviated from the simulated front progression shown in figure 3.29 (a). To understand the effect of this change in the infusion and study the effectiveness of the process flow modeling to emulate these effects, the injection boundary condition in the selected injection configuration A is modified to match the experiential change of modified infusion. This was emulated through a pressure drop varying only half way through the feed line from the injection end. The modified injection condition employed in the flow modeling simulation resulted in a total fill time of 47 minutes, which is only a two minute difference when it is compared to the experimental result. Furthermore, the simulated resin front progression under these modified injection conditions showed an excellent agreement to the experimental flow progression as shown in Figure 3.29 (b) and 3.29 (c). This is a clear indication of the capability of flow modeling and simulations to capture the flow process variants during resin infusion in liquid composite molding.

(a) Unadjusted pressure injection flow (simulation)

(b) Adjusted pressure injection flow font (experimental)

(c) Adjusted pressure injection flow font (simulation)

(d) Adjusted pressure injection flow font (Full simulation)

Figure 3.29 Experimental Vs. simulation comparison

3.3.3 Probabilistic Modeling of Process Parameters Variations

As for the simple model, statistical analysis software SPSS has been used to generate the viscosity and permeability data to understand the effect of their variations and uncertainties on the resin infusion fill time. As before, a $\pm 20\%$ from the mean value of the viscosity and ±50% the mean value of the permeability was considered in this analysis. Table 3.3.2 shows the statistical values (mean and standard deviation) for viscosity and permeability data distributions.

3.3.4 One parameter Model - Resin Viscosity (η) Variations

As discussed earlier and shown in the flow chart in Figure 3.1 by using SPSS we generated 100 normally distributed viscosity values. The viscosity values generated had a range of plus or minus (\pm) 20% the mean value of 5.07 x 10⁻⁵ (lbf-s/in²). Table 3.3.3 shows all the statistical parameters for the generated viscosity values, and as shown in Figure 3.30 the histogram validated that the generated viscosity values follow a normal distribution.
Table 3.3.3 Statistical parameters for viscosity

Statistics

a. Multiple modes exist. The smallest value is shown

Histogram

Figure 3.30 Histogram for the viscosity value

For this given complex 3D model and the resin system employed, the gelation time or the resin pot life of the resin is 55 minutes ($GT = 55$ minutes) requiring the infusion to complete prior to gelation. Even with the variations in the viscosity, permeability values, the resin infusion has to be completed prior to gelation, which requires the fill time (FT) to be less than or equal to the gelation time (FT \leq GT). The generated viscosity values were employed to obtain corresponding 100 values for the resin infusion time using the flow modeling simulation, FERTM. As shown in Figure 3.31, the viscosity increase resulted in a linear increase in the resin infusion time. Subsequently, the fill time data were analyzed and a 95% confidence envelope developed for the completion of resin infusion prior to gelation time. In this case, this corresponds to a resin infusion fill time to be less than or equal to 55 minutes ($FT \leq GT$).

Figure 3.31 Scatter diagram relationship between viscosity and fill time

Figure 3.32 presents the histogram of the computed fill time variations obtained for the 100 viscosity values. This clearly conveys the fact that the obtained fill time values corresponds to an approximate normal distribution of data.

Histogram

Figure 3.32 Histogram of resin infusion time

By using the obtained fill time data, an envelope for the confidence level or probability for complete resin infusion for various viscosity values required to make the given complex 3D model composite part within a given fill time ($FT \leq GT$) prior gelation was developed. This is obtained using the cumulative density function (CDF) within SPSS. The CDF describes the probability that a real-valued random variable, viscosity with a given probability distribution will be found to given resin infusion time values less than or equal to

the gelation time 55 minutes. Figure 3.33 and Figure 3.34 presents the cumulative density function (CDF) for the resin viscosity and fill time for this composite part. From these figures, we can conclude that if the viscosity value is less than or equal to $(\eta \le 5.1 \times 10^{-5} \text{ lbf}$ $s/in²$) presents a 50% confidence interval that the fill time for this composite part geometry and injection condition will be less than or equal to gelation time of 55 minute ($FT \leq GT$). If the viscosity value is less than or equal to $(\eta \leq 4.64 \times 10^{-5} \text{ lbf-s/in}^2)$ it presents a 95% confidence interval that the fill time will be less than or equal to gelation time. Clearly, reduced viscosities lead to higher probability for successful infusion prior to gelation.

Figure 3.33 Probability plot for the 50% and 95% confidence interval

Figure 3.34 Probability VS simulated resin infusion time plot for 95% confidence interval

3.3.5 One parameter Model - Permeability (K) Variations

To conduct the permeability (K) variations, statistical analysis software SPSS was utilized to generate normally distributed 100 values of permeability around the mean permeability previously used in the flow modeling simulations. The permeability values generated ranged from \pm 50% the mean permeability value of 3.5 x 10⁻⁶ (in²). Table 3.3.4 shows all the statistical parameters for the generated permeability values, and as shown in Figure 3.35 the histogram validated that the generated permeability values follow an approximate normal distribution.

Table 3.3.4 statistical parameters for permeability

a. Multiple modes exist. The smallest value is shown

Histogram

Figure 3.35 Histogram of Permeability (K)

As it was used for the viscosity variations, the gelation time or the resin pot life is equal to 55 minutes (GT = 55 minutes). By keeping the viscosity constant at 5.07×10^{-5} (lbf $s/in²$), 100 FERTM simulations were used to obtain the fill time for each of the generated permeability values. The generated 100 values of permeability were used to obtain 100 corresponding values of fill time employing the flow modeling simulations. As shown in Figure 3.36 as the permeability (K) values decreases, the fill time increases. In the following section, the fill time data for the permeability variations were analyzed, and a 50% (for illustration) and 95% confidence interval envelope was developed that corresponds to an infusion time of less than or equal to gelation time of 55 minutes ($FT \leq GT$).

Figure 3.36 Scatter diagram relationship between permeability and fill time

The cumulative density function describes the probability that a real-valued variable, permeability (K) with a given probability distribution will be found to give a resin infusion time value less than or equal to the gelation time 55 minutes. Figure 3.37 and Figure 3.38 presents the cumulative density function (CDF) for permeability and the associated fill time respectively. It can be determined from these figures that if the permeability value is greater than 4.40×10^{-6} , it represents a 95% confidence level that the fill time for this composite part and injection condition will be less than or equal to gelation time of 55 minutes ($FT \leq GT$). A lower permeability value of $\leq 2.73 \times 10^{-6}$ in² indicates a probability for successful infusion prior to gelation to be less than 15%

Figure 3.37 Probability plot for 95% confidence interval for permeability (K)

Figure 3.38 Probability Vs simulated resin infusion time plot for 95% confidence level for permeability (K)

3.3.6 Two parameter Model - Resin Viscosity and Permeability Variations

As a final step for this complex 3D helicopter composite part, variations in both the resin viscosity and permeability are considered. As the viscosity showed a linear variation with resin infusion fill time (FT), five different viscosity values are selected and by using SPSS fifty different values of permeability's were generated around the mean value of $3.5 \times$ 10^{-6} ± 50%. Using a FERTM flow modeling simulations, the selected fifty values of permeability were applied to each of the five viscosity values and two hundred fifty values of the corresponding fill time are obtained. Table 3.3.5 shows the descriptive statistical output

for the fill time. As seen from this table, the mean and median are different, especially considering the significant standard deviation. Figure 3.39 and Figure 3.40 clearly shows a heavily skewed distribution of the resin infusion time data, even though the permeability variations showed a normal distribution. This was further confirmed with a descriptive statistical analysis with normality test performed using SPSS.

Table 3.3.5 statistical values for simulated resin infusion time

a. Multiple modes exist. The smallest value is shown

Figure 3.40 Resin infusion time normality test

Since the normality tests expressed that the fill time data are not normally distributed, the raw values of the infusion time were converted to natural logarithmic values. We looked at different normalization functions such as square root, inverse, and square transformation however the logarithmic transformation gives the best normal fit for the given data. As Table 3.3.6 shows, this transformation led to the mean and median being very close. The logarithmic transformed fill time data was further analyzed to check the normality of the data via a graphical statistical normality test. Figure 3.41 and Figure 3.42 clearly shows that natural log transformation resulted in a normal data distribution with a closer mean, median, and mode. .

Statistics

a. Multiple modes exist. The smallest value is shown

Figure 3.41 Histogram for transformed resin infusion time

Figure 3.42 Transformed resin infusion time

From the normalized fill time data, the cumulative density function and the probability to obtain resin infusion times that are less than or equal to the gelation time were studied for different viscosity, permeability combinations. As it is shown in Figure 3.43 a 95% confidence interval is developed for the complex 3D model that is affected by both permeability (K) and viscosity (η) at the same time. Furthermore for this composite part and injection conditions, viscosity contributes a 25% of the variation in resin infusion fill time (r^2) $= 0.25$), while the permeability contributes 68% variations in the resin infusion time ($r^2 =$ 0.68) and the remaining 7% variation could be from other factors that affect the LCM simulation such as pressure, injection location, etc.

Figure 3.43 95% confidence interval for permeability (K) and Viscosity (η)

The data ranges for permeability (K) and viscosity (η) that will give a fill time less than or equal to gelation time of 55 minutes (FT \le GT) is given in Tables 3.3.7 –3.3.9 and Figures 3.44 - 3.46 for 95%, 90%, and 80% confidence levels. From Table 3.3.9, for a resin viscosity of 4.56 x 10⁻⁵, any preform viscosity values of greater than or equal to 2.70 x 10⁻⁶, presents a probability of successful resin infusion to be 80% for completion prior to gelation. Table 3.3.7 95% confidence interval range for viscosity and permeability variations.

Figure 3.44 95% confidence interval range for permeability (K) and viscosity (η)

Viscosity (lbf-s/in ²)	Permeability (in ²)
4.56×10^{-5}	$K > 2.80 \times 10^{-6}$
4.73×10^{-5}	$K > 2.94 \times 10^{-6}$
5.07×10^{-5}	$K \ge 3.18 \times 10^{-6}$
5.36×10^{-5}	$K \ge 3.45 \times 10^{-6}$
5.92×10^{-5}	$K > 3.80 \times 10^{-6}$

Table 3.3.8 90% confidence interval range for viscosity and permeability variations.

Figure 3.45 90% confidence interval range for permeability (K) and viscosity (η)

Viscosity (lbf-s/in ²)	Permeability (in ²)
4.56×10^{-5}	$K > 2.70 \times 10^{-6}$
4.73×10^{-5}	$K \ge 2.80 \times 10^{-6}$
5.07×10^{-5}	$K \ge 3.00 \times 10^{-6}$
5.36×10^{-5}	$K > 3.32 \times 10^{-6}$
5.92×10^{-5}	$K > 3.49 \times 10^{-6}$

Table 3.3.9 80% confidence interval range for viscosity and permeability variations.

Figure 3.46 80% confidence interval range for permeability (K) and viscosity (η)

CHAPTER 4

Analysis of Results and Discussion

Process simulations are based on given values of parameters employed in the simulations. As discussed earlier, significant variations in these parameters can occur during actual manufacture. The resin viscosity can very between different resin batches, mixes and the viscosity is also influenced by temperature and then ambient conditions. The preform permeability can also vary significantly as discussed before.

Due to their high strength, durability, reduction in weight, and chemical stability, composites materials offer many performance advantages for a wide variety of structural applications. Due to the complex nature of reinforced polymer composites, many different techniques for manufacturing of components have been and will continue to be developed. The objective of any composite manufacturing process is to combine the polymer resin and fiber reinforcement in an acceptable manner during processing to obtain the component design. Manufacturing processes are developed depending upon the critical aspects of the finished component, product performance, quality, cost, and application.

The development of a manufacturing process to utilize LCM can be complex due to the determination of required support equipment. Molds, reinforcement materials, resins, filling equipment, and curing cycles must be investigated to assure the finished component meets the design requirements. With cost an important factor in any manufacturing process, the elimination of trial and error iterations in the development of a successful process is critical.

In the LCM process, the principal factors that determine the resin flow process and final part quality can be grouped into two types: deterministic factors and stochastic factors. Injection pressure, flow rate, mold temperature, etc., are generally the deterministic factors, which means they can be measured or controlled as desired. The primary sources of uncertainty are the preform permeability dominated by its microstructure; differences in preform material, effect of lay-up, compaction, etc., and the variability of rheological and kinetic properties of the resin viscosity. The effect of uncertainties in two key process parameters that influence the success of resin infusion are investigated in the present work.

The technique and methodology that has been studied in this thesis research can be used during the actual manufacturing process of LCM. By using the developed confidence interval envelopes, obtained through probabilistic modeling methodology presented in this work, manufacturing engineers have an analytical tool available to determine the probability of successful infusion prior to gelation and estimated infusion time for any combination of permeability and resin viscosity. This can be obtained without a need for additional flow modeling simulations, and are effective to not only understand the effect of these parameter variations but also estimate the process success. This is more desirable in large complex parts where the simulations can take significant computational time. In the next section, three scenarios are presented to show how the developed confidence envelopes for the viscosity and permeability variations for the complex composite helicopter part can be utilized in the actual manufacturing.

Scenario: 1

For a give composite part configuration manufacturing (composite helicopter part in this example), during in the actual process the manufacturing engineer collects the resin

viscosity data and conducts the preform permeability characterization for the permeability of the used resin and preform roll. On a given day, if these values for resin viscosity and preform permeability are $\eta = 4.85 \times 10^{-5}$ lbf-s/in² and K = 3.25 $\times 10^{-6}$ in². By taking this data combination and using the developed confidence interval envelope, the manufacturing engineer can determine the probability of success of the resin infusion prior to gelation. As shown in Figure 4.1 for day one collected data, $(\eta = 4.85 \times 10^{-5} \text{ lbf-s/in}^2 \text{ and K} = 3.25 \times 10^{-6} \text{ m}^2 \text{ m}^2 \text{ m}^2$ \sin^2), the confidence interval level for the resin infusion time lies in a 95% confidence interval range. This indicates that there is a 95% confidence for the infusion time of this composite part under these permeability and viscosity conditions prior to gelation. The 95% confidence envelope as determined from the probabilistic modeling is employed (Figure 3.44) for the helicopter part is used. The corresponding infusion time for this composite part can be obtained form Figure 3.43 to be 48 minutes.

Figure 4.1 Scenario: 1 – 95% confidence interval

Scenario: 2

For day two, the manufacturing engineer collects the resin viscosity and preform permeability data for the same part configuration. If the resin viscosity and permeability on this day are $\eta = 4.60 \times 10^{-5}$ lbf-s/in² and K = 2.85 \times 10⁻⁶ in². By using the developed confidence interval envelop, the resin infusion time prior to gelation is in a 90% confidence interval range as shown in Figure 4.2, based on the prior developed confidence envelope shown in Figure 3.45. The estimated infusion time for this case can be obtained from figure 3.43 to be 52 minutes.

Figure 4.2 Scenario: 2 – 90% confidence interval

Scenario: 3

For day three, if the manufacturing engineer collected different resin viscosity and preform permeability data ($\eta = 4.56 \times 10^{-5}$ and K = 2.75 $\times 10^{-6}$ in²). The probability of successful infusion can be obtained from the prior developed confidence envelopes for this composite part and injection condition. This permeability viscosity combination indicates that there is 80% confidence interval level range, that the resin infusion time is to be completed prior to gelation (FT \leq GT). This permeability and viscosity, combination is covered by the 80% confidence envelope shown in Figure 3.46. The estimated infusion time in this case would be 53 minutes.

Figure 4.3 Scenario: 3 – 80% confidence interval

All these above scenarios clearly indicate that the probability for successful resin infusion prior to gelation and the associated infusion time can be estimated without a need for additional flow modeling and simulations. This demonstrates the effectiveness of the presented probabilistic methodology for the determination of resin infusion success and confidence in a production environment and to provide an estimation of the resin infusion time without a need for additional simulation analysis.

CHAPTER 5

Concluding Remarks

Liquid composite molding processes such as resin transfer molding and its variants have become desirable processes for the processing and manufacture of woven fiber polymer composite structures. The success of resin infusion depends upon the complete infusion of dry fiber preform prior to gelation. Two key parameters that influence the resin infusion progression and infusion time are resin viscosity and preform permeability. For a given composite part configuration and injection condition, the resin infusion time and progression are dependent upon variations in the resin viscosities during actual infusion and permeability variations in the preform used at the time of processing. The present work investigated the effect of uncertainties and variations in these two key parameters for a vacuum based resin infusion liquid composite molding process utilizing physics based flow modeling simulations and statistical analysis. In particular, the present work

- Presented and demonstrated a probabilistic modeling methodology for analysis of uncertainties in the resin infusion process parameters of resin viscosity and permeability employing process flow modeling simulations and statistical analysis.
- Investigated the stochastic variations of the viscosity and permeability on the resin infusion time to determine the probability for successful resin infusion prior to gelation.
- Employed statistical analysis to obtain confidence levels and a computational modeling enabled analytical tool to determine the probability for successful infusion

and estimate the infusion time for any combination of permeability and resin viscosity for a composite part and injection conditions.

- Demonstrated the applicability of the present methodology in two composite structural configurations based on a simple composite plate and a complex composite helicopter part.
- Validated the effectiveness of the process flow modeling capability based on experimental flow progression and infusion time comparisons for the composite helicopter part.

The computational modeling framework and probabilistic methodology presented in this work provide manufacturing engineers with a computational modeling based analytical tool to determine the probability for successful infusion prior to resin gelation, and an estimated resin infusion time for any combination of permeability and resin viscosity for a given composite part and injection conditions. This can be obtained without a need for additional flow modeling simulations, and are effective to not only understand the effect of these parameter variations but also estimate the probability for successful infusion under varying process parameter conditions on any given day during actual manufacture. Furthermore, the probabilistic modeling methodology presented in this work is applicable and extendable to any other composite part structure following the analysis framework presented in the flow chart and associated discussions in chapter 3.

Although this thesis work focused on two key parameters that influence the resin infusion, future study can be extended to more parameters that affect the liquid composite molding process, and would require multi variable statistical analysis techniques. The

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probabilistic methodology for process parameter uncertainties can be further coupled with other optimization approaches based on continuous sensitivity analysis, genetic algorithms, etc., built further upon appropriate physics based process modeling simulations for other parameters to include the variations in the injection pressure, flow rates, mold temperatures, etc., and provide directions for future study.

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