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Multiscale Modeling Approach of Slot Die Coating of Unidirectional Glass Fiber Fabric

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ABSTRACT

Slot die coating with vacuum-assist has been developed as a novel and scalable methodology for coating glass fiber fabrics as a pre-step to composite fabrication. In particular, the slot die coating of cellulose nanocrystal suspension, a material which has been recognized as an emerging renewable material for fiber sizing, is subject to further exploration due to the limited scalability studies. In this paper, a multiscale computational fluid dynamics (CFD) model is developed, representing and combining separate 2D models of macroscale coating flow on top of the fabrics as well as microscale porous flow within to establish a better understanding of the flow dynamics and the effect of processing conditions which control the final coating morphology outcomes. The model simulates the effect of vacuum assist on the coating process and successfully predicts stable operating parameters matching the ones established in prior experimental trials.

1 | Introduction

The utility of nanocellulose nanomaterials as a novel and renewable coating material has led to their incorporation into a wide range of coating technologies, spanning spin coating, blade coating, bar coating, spray coating, and slot die coating to produce self-standing/pure nanocellulose films or functional layers on permeable substrates [1–6]. Nanocellulose materials are cellulose-based nanosized particles extracted from woody plant biomass and can provide mechanical reinforcement to a wide variety of polymers if properly used (e.g., fine-scale dispersion, compatibilizing surface chemistry, etc.) [7]. One type of nanocellulose material is cellulose nanocrystals (CNCs), produced by acid hydrolysis; they have a spindle-like particle morphology (5–20 nm diameter, 50–500 nm length), high crystallinity, low density (1.6 g/cm³), high mechanical properties, and low toxicity.

For the fabrication of polymer composite films, the deposition of a separate nanocellulose layer or multilayers on top of flexible polymer sheets has been shown to improve the material barrier properties as well as their scratch resistance, without the need to disperse the particles within the packaging film itself, avoiding the common challenges with nanocellulose agglomeration [8–10]. A similar principle has since been explored to incorporate CNCs as secondary reinforcement into fiber reinforced polymer composites by coating the CNCs onto the primary reinforcing fibers, in a separate step from resin matrix infusion [11–13]. For the continuous sizing of glass fiber (GF) rovings and fabrics, slot die coating on a roll-to-roll, a traditional pre-metered thin-film deposition method, has been demonstrated to uniformly coat GF fabrics in CNC, using the addition of vacuum assist to control the coating fluid distribution and prevent the formation of barrier coatings which would resist resin wetting during composite fabrication [14–16].

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The novel combination of slot die coating with highly permeable heterogeneous substrates and vacuum assist renders the system difficult to represent using traditional explicit models and simplifying assumptions; as such, computational fluid dynamics (CFD) modeling approaches are best used to gain a better understanding of the parameters and penetration dynamics present in coating fluids on top of complex fibrous substrates [17]. To model the coating mechanism, macroscale models are often used combining governing equations, such as the Navier–Stokes equations, to model the fluid flow above the substrate while relying on Darcy's law, the Kozeny–Karman, Young–Laplace, or Lucas–Washburn equations to represent the fluid imbibition into the porous medium [18–22].

In many macroscale models, whether explicit or numerical approaches are used, the substrate is often represented as an isotropic porous medium with a constant porosity and permeability throughout [17]. In order to better approximate the fluid imbibition into complex fibrous media, the governing equations have been modified to represent the effective pore size, permeability, and capillary forces as functions of fiber radius and spacing [23–25]. To place a larger focus on the coating flow within the substrate, microscale models have also been used to build the porous domains, allowing for more accurate predictions of how substrate microstructure affects fluid distribution and penetration [17, 26].

While microscale models provide more insight into the effect of various microstructural characteristics on porous flow, as static models they can neither capture the web movement of continuous coating processes nor calculate the final film thickness, both of which are better represented by the macroscale coating models [17]. Multiscale computational models have thus emerged as an alternative approach to modeling slot die coating of porous media, coupling the predictions between the macroscale and microscale models to offer predictions of both coating bead flow dynamics and fluid penetration [17, 27].

As a key step in advancing the novel usage of slot die coating as a method for sizing GF fabrics in CNC, CFD models can be used to predict operating parameters, facilitating scale-up operations. In this study, the slot die coating of CNC onto GF fabrics is modeled using both a macroscale only and a multiscale approach, with the fluid imbibition into the GF fabric represented using governing equations and 2D microscale models respectively. The predictions of coating parameters, process stability, and final film thickness from both models are experimentally validated.

2 | Materials and Experimental Methods

2.1 | Coating Materials and System

The study is conducted using the coating solution and E-GF fabric as outlined in [14]. The coating solution, labeled 5EM1CNC, contains 1 wt% CNC functionalized with methyl(triphenyl) phosphonium ($\rho_{\text{CNC}} = 1.6 \text{ g/cm}^3$), supplied from American University (Washington, DC, USA) and 5 wt%

epoxy-emulsion containing 4 wt% thin epoxy (US Composites) and 1 wt% PL123 copolymer (Sigma-Aldrich), and is prepared following procedures as outlined in [12]. The reported density of the epoxy is $\rho_{\text{EP}} = 1.1 \text{ g/cm}^3$, and the density of the PL123 is $\rho_{\text{PL123}} = 1.01 \text{ g/cm}^3$. The slot die coating on a roll-to-roll with vacuum assist system used is likewise described in [14], and the results from the study are used to validate the operating parameter predictions.

2.2 | Contact Angle and Surface Tension Measurement

Contact angle measurements and surface tension measurements were conducted using a Rame-Hart goniometer Model 250 with DROPimage Advanced software. The sessile drop method was used to measure the changing contact angle of a 4 μL droplet of 5EM1CNC dispersion on E-GF fabric. High frame rate recordings were made to observe the change in contact angle and droplet spread in 0.1 s increments. The sessile drop method was also used to measure the static contact angle formed between 5EM1CNC dispersion and the stainless-steel slot die head. Surface tension of the dispersion was measured using the pendant drop method.

2.3 | Characterization of Coating Fluid

The full viscosity curve of the 5EM1CNC formulation was determined using a Rheostress 600 rheometer using a pp20 spindle, that is, parallel plate with a 20 mm diameter, at ambient conditions. Shear sweep measurements were made at shear rates 1 and 1000 s^{-1} . The resulting viscosity curves are compared and fitted against power-law and Carreau fluid models represented in Equations (1) and (2), respectively.

$$\eta_{\text{app}} = k\dot{\gamma}^{n-1} \quad (1)$$

$$\eta_{\text{app}} = \eta_{\infty} + (\eta_0 - \eta_{\infty}) \left[(1 + \lambda\dot{\gamma})^2 \right]^{\frac{n-1}{2}} \quad (2)$$

Both models assume the presence of a shear-thinning region, typical to CNC dispersions, which are represented by the shear thinning index (n) [28]. The apparent viscosity, (η) is a function of shear rate ($\dot{\gamma}$). Zero (η_0) and infinite shear (η_{∞}) viscosities are reported when the coating fluid exhibits Newtonian plateau regions at the lowest and highest shear rates.

2.4 | Characterization of GF Fabric

The porosity of the E-glass fabric was evaluated using a Scanco μCT50 MicroCT scanner with a 50 mm field of view (FOV), that is, millimeter-scale FOV, and $<0.5 \mu\text{m}$ resolution and a Phenom XL G2 SEM with accelerating voltage of 10 kV. For the MicroCT, due to the loose weaving of the E-glass fabrics, a piece was embedded into low viscosity epoxy resin to keep the rovings from fraying. The cured sample was then cut into a $\sim 1.2 \times 1.2 \text{ cm}$ sample which was scanned using 1600 slices under high resolution. Analysis of the scan was done using Dragonfly 3D software to separate the fibers from void space. For SEM imaging, six (6)

transverse cross sections of the embedded GF fabric were cut and polished, and the contrasted SEM images were analyzed using ImageJ to provide an average porosity/void space value and average fiber radius.

2.5 | Measurement of Film Thickness

As measuring wet film thickness (t_{wet}) deposited onto GF fabric is challenging due to its compressibility and uneven surfaces, the conversion between dry film thickness (t_{dry}) and (t_{wet}) is approximated using a mass balance based on the known composition and densities of the coating fluid, (ρ_{wet}) and dried coating (ρ_{dry}). Assuming complete drying of the deposited coating, all of 94 wt% of DI water is evaporated off and only the 0.06 wt% of the coating remains giving the relation in Equation (3):

$$\rho_{\text{wet}} * \text{Area} * t_{\text{wet}} * 0.06 = \rho_{\text{dry}} * \text{Area} * t_{\text{dry}} \quad (3)$$

where ρ_{wet} and ρ_{dry} are calculated using the densities and mass fractions of the individual components, as listed in Section 2.1. Dry film thickness deposited on top of GF fabric is measured using SEM images of 5EM1CNC coated E-GF fabric samples discussed in [14].

3 | Modeling Approach

3.1 | Phase-Field Model Equations

To develop CFD models, the Laminar Two-Phase Flow, Phase Field interface in COMSOL Multiphysics 6.1 is used. Though the Level Set interface has also been used to simulate similar coating processes, the Phase Field model was chosen as it requires less computational power [29]. The Phase Field interface relies on the Cahn–Hilliard model, which describes the free energy as a function of the dimensionless phase field parameter (ϕ) and its gradient ($\nabla \phi$), and temperature (T), although T is neglected for isothermal fluids, as shown by Equation (4):

$$F(\phi, \nabla \phi) = \alpha \int f dV = \alpha \int \frac{1}{2} (|\nabla \phi|^2 - g(\phi)) dV \quad (4)$$

where ϕ is defined for two fluid components such that the volume fraction of each is $(1 + \phi)/2$ and $(1 - \phi)/2$, and α represents the mixing energy [30, 31]. The evolution of ϕ is described by a partial differential Equation (5), where μ represents dynamic viscosity, u is flow velocity (SI units: m/s), and M is the mobility parameter, determined within COMSOL Multiphysics by the interphase thickness.

$$\frac{\partial \phi}{\partial t} + (u \cdot \nabla) \phi = \nabla \cdot (M(\phi) \nabla \mu) \quad (5)$$

In Equation (6), the term $g(\phi)$ is defined as a double potential, where ξ is defined as the capillary width that scales with the thickness of the interface.

$$g(\phi) = \left(\frac{1}{4} \xi^2 \right) \cdot (\phi^2 - 1)^2 \quad (6)$$

The chemical potential of the system (G) is calculated using Equation (7), and the surface tension (σ), of the system relates α and ξ , using the relation in Equation (8).

$$G = \frac{\alpha}{\xi^2} [\phi(\phi^2 - 1) - \xi^2 \nabla^2 \phi] \quad (7)$$

$$\sigma = \left(2\sqrt{2}/3 \right) \cdot (\alpha / \xi) \quad (8)$$

The modified Navier–Stokes equation for incompressible fluid, combined with Equations (6) and (8) yields Equation (9) as shown below, where (ρ) is density and (p) is pressure.

$$\rho \left(\frac{\partial u}{\partial t} + (u \cdot \nabla) u \right) + \nabla p = \nabla \cdot [\mu (\nabla u + (\nabla u)^2)] + G \cdot \nabla \phi + \nabla \cdot u + \text{Eq. 4} + \text{Eq. 6} \quad (9)$$

3.2 | Macroscale Slot Die Coating Model

To model the macroscale slot die coating process, the method used by Ding et al. is adapted for this study [19]. A 2D representation of the slot die head and moving substrate is presented in Figure 1 following the slot die coating system from [14]. The coating gap (H) is set to 0.3 mm [16]. The slot gap (W) is 0.16 mm, and stainless steel upstream and downstream lip lengths (X) are both 1.76 mm. The upstream (θ_u) and downstream (θ_d) slot die lip angles are 35° and 45° respectively. Parameters for inlet

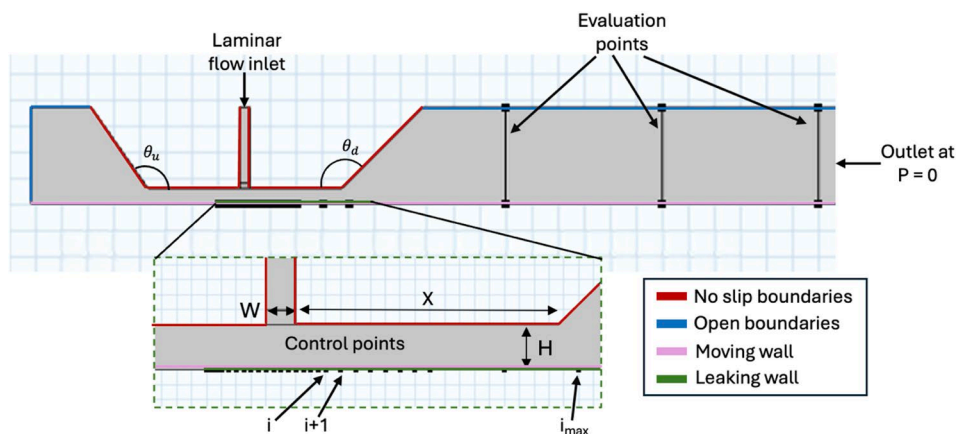


FIGURE 1 | Representative domain for the macroscale slot die coating model showing finer control point spacing for initial penetration area and coarser spacing downstream. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com/doi/10.1002/polb.25000)]

coating flow rate (Q) and web velocity (v) are partially reported in [14].

All walls of the slot die head are defined with no slip boundaries. The reference pressure is set to 0 (zero) at the outlet and open boundaries, with the outlet condition with no backflow enforced at the rightmost boundary, representing the substrate exiting out of frame. To model the progressive fluid penetration velocity into GF fabric, control points indexed by $i = 1$ to $i_{\max}$ are placed onto the bottom boundary with the leaking wall condition applied between the points concurrently with the moving wall condition. A predefined *physics-controlled* free triangular meshing is used.

Two different approaches are used to model the fluid penetration into the GF fabric. For the first approach, the fluid penetration velocity at the leaking walls is determined using Darcy's law, and the final wet thickness on top of the substrate, following GF fabric saturation, is calculated using integration/evaluation lines spaced 3 mm apart downstream from the slot die. Manual mesh refinement is likewise performed on the control point placement. The second approach uses a combined multiscale model, coupling the macroscale coating model with a separate microscale model which simulates progressive fluid penetration through the GF fabric structure. This method resembles the multiscale approach used by Didari et al., but instead of mapping the pressure pulse predicted by the macroscale model onto the microscale one, in this study, the fluid penetration velocity predicted by the microscale model is mapped onto the leaking wall boundary of the macroscale model, discussed in greater detail in Section 3.4 [27].

3.3 | Modeling Fluid Penetration Using Darcy's Law

A simple approximation of fluid infiltration into GF fabric is performed using 1D Darcy's law, shown in Equation (10), similar to models described in [24, 29]. The 1D representation of fluid penetration, which only assumes out-of-plane or transverse flow into the substrate may not be accurate to the unidirectional GF fabric which typically experiences longitudinal wicking within the gaps between the parallel GFs. However, for the purposes of this study, the parallel flow between GFs will be neglected due to the longitudinal movement of the web and substrate, which is assumed to negate the longitudinal capillary wicking. The vertical penetration velocity (v_i), defined at each individual control point, is calculated from local pressure exerted by coating bead (p_i), capillary pressure in the fabric (p_c), and in the cases with vacuum pressure applied (p_v). The penetration depth (h_i), is calculated as a function of time (Δt), iteratively, as shown in Equation (11). The distance between the control points (Δs), is a function of time interval, Δt and the web velocity (v) as defined by Equation (12). Since Darcy's law dictates that the penetration velocity gradually slows as saturation is approached, Δs is kept short in the initial penetration zone and increased downstream from the coating gap, where the velocity gradient is smaller [29].

$$v_i = \frac{K_{\perp}}{\eta \epsilon} \frac{dP}{dz} = \frac{K_{\perp}}{\eta \epsilon} \frac{p_i + p_c + p_v}{h_i} \quad (10)$$

$$h_i = h_{i-1} + \frac{v_{i-1}}{\epsilon} \Delta t \quad (11)$$

$$\Delta s = \Delta t \times v \quad (12)$$

Continuing with the 1D assumptions, transverse permeability ($K_{\perp}$) of the GF fabric is considered, calculated using the Happel model for flow relative to arrays of cylinders, given in Equation (13), as a function of substrate porosity (ϵ), and GF radius (R) [32].

$$K_{\perp} = \frac{-R^2}{8(1-\epsilon)} \left[\ln(1-\epsilon) + \frac{\epsilon(2-\epsilon)}{\epsilon^2 - 2\epsilon + 2} \right] \quad (13)$$

The capillary pressure (p_c) within porous substrates is often expressed as a function of contact angle (θ) between the fluid and solid, fluid surface tension (σ), and cylindrical pore diameter (D), as derived using the Laplace-Young equation [33, 34]. For unidirectional GF fabrics, the model is modified to account for non-circular pores [24, 25]. Accounting for the GF fabric geometry, p_c , as shown in Equations (14) and (15), is therefore calculated using an expression for equivalent diameter (D_e), derived using the GF radius, GF fabric porosity (ϵ), and a geometric factor $F = 2$ for transverse unidirectional flow [24, 25].

$$p_c = \frac{4\sigma \cos(\theta_{\text{GF}})}{D_e} \quad (14)$$

$$D_e = \frac{8R\epsilon}{F(1-\epsilon)} \quad (15)$$

The Darcy's Law model additionally assumes a constant viscosity (η), which is not consistent with the non-Newtonian behavior of the 5EM1CNC formulation. For power-law fluids, the Blake-Kozeny equation has been used to model the fluid penetration, however, this representation would also be inconsistent with the 5EM1CNC behavior which only exhibits power-law properties within the shear rate limits of approximately $20\text{--}200\text{ s}^{-1}$; assuming power-law behavior at higher shear rates could therefore inaccurately predict viscosity values approaching singularity [29, 35]. The shear rate of fluid flowing within fibrous substrates can be estimated using standard packed-bed and cylindrical capillary models as functions of fiber volume fractions, porosity, as well as fiber spacing and diameter [36–39]. Given the inverse relationship between the shear rate versus the pore size and fiber diameter derived by these models, the effective shear rate calculated exceeds the range of the power-law region, falling within the Newtonian plateau with an infinite shear viscosity (η_{∞}). Thus, η_{∞} is used in place of η within the Darcy's Law equation.

3.4 | Modeling Fluid Penetration Using Microscale Models

To simulate the fluid penetration within the GF fabric, an SEM image of a transverse cross section or slice of the E-GF fabric was used to directly trace a 2D domain geometry using MATLAB and re-plotted in COMSOL using an Image-to-Curve add-in to produce a representative domain volume (RVE) with a porosity matching that of the GF fabric as a whole. The domain maintains the full GF fabric thickness of 0.34 mm and a representative width of $66\text{ }\mu\text{m}$. The reference image and traced domain are shown in Figure 2.

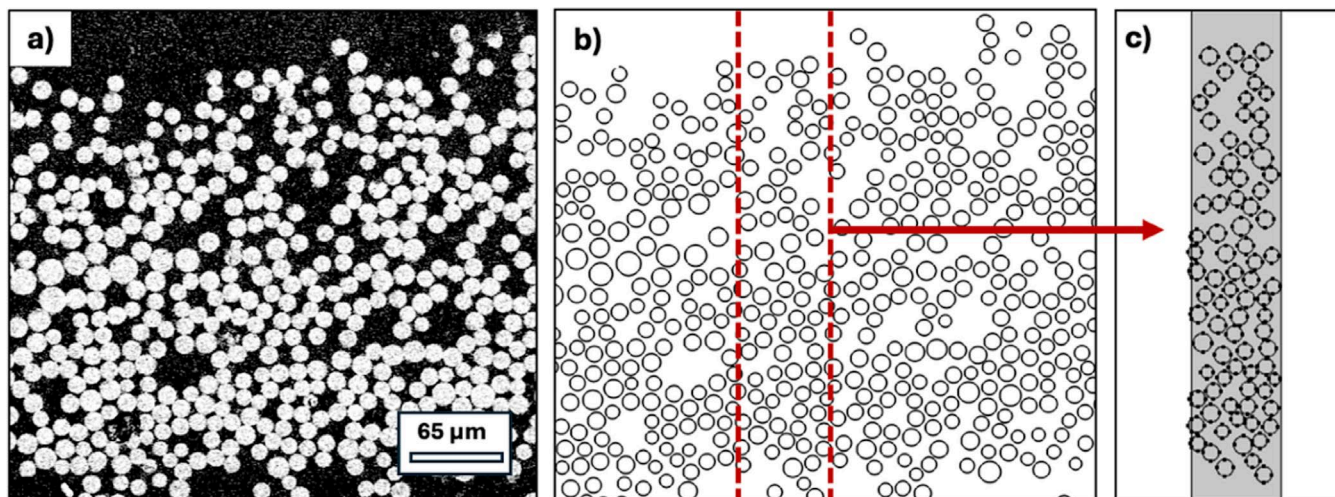


FIGURE 2 | GF fabric (a) SEM image of polished cross section, (b) traced using MATLAB, and (c) replotted as an approximated domain into COMSOL. [Color figure can be viewed at wileyonlinelibrary.com]

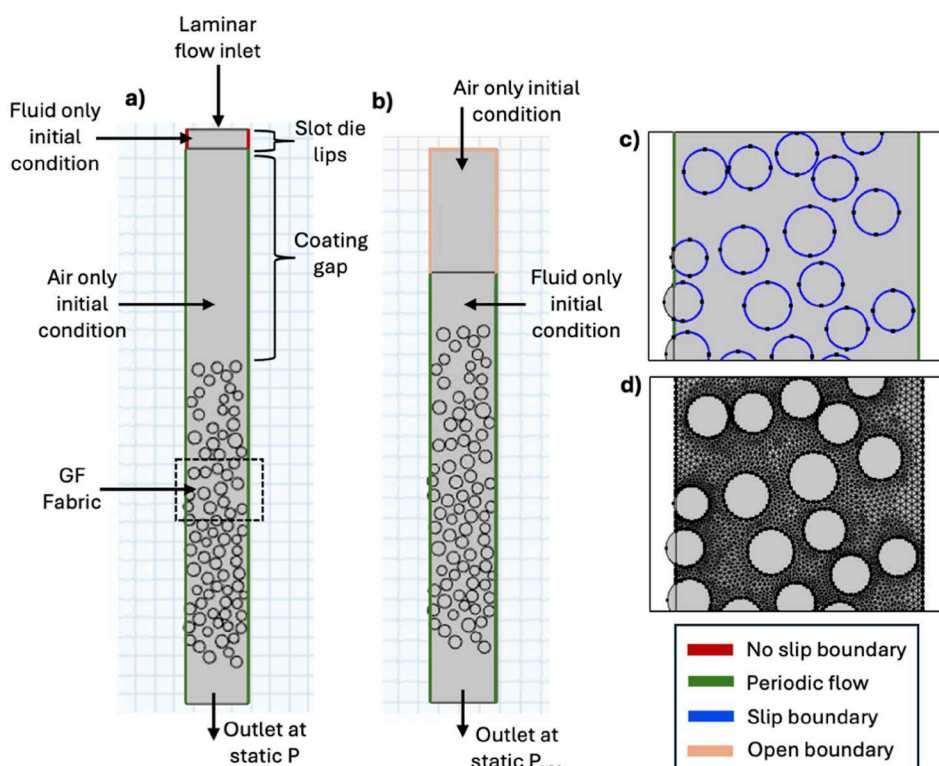


FIGURE 3 | Domain geometry and boundary conditions of the 2D GF fabric microscale models simulating (a) initial fluid penetration and saturation of the GF fabric, (b) vacuum-driven fluid flow following saturation, (c) close ups of showing boundaries of GFs, and (d) free triangular meshing within the domain. [Color figure can be viewed at wileyonlinelibrary.com]

To model the fluid penetration up until saturation, a 2D microscale model is built, as shown in Figure 3a, comprising not only the plotted GF array but also the coating gap (H) above it to represent the coating fluid exiting the slot die head and entering into the GF fabric. This domain is comprised of two layers; the top layer represents the stainless-steel slot die lips with a no slip boundary, and the bottom includes the coating gap and GF fabric, contained within periodic flow boundaries on the sides.

The fluid penetration velocity profile yielded by the model in Figure 3a is mapped onto the leaking wall boundary of the

macroscale model with a moving web, which predicts wet film thickness following GF fabric saturation. However, for slot die coating with vacuum assist, the vacuum pressure continues to drive fluid flow even following saturation, thus the no-vacuum wet film thickness is used as a starting condition for the microscale model with vacuum, shown in Figure 3b, which models the continued fluid removal which is mapped downstream from the coating bead within the macroscale model.

To model the effect of vacuum pressure on fluid flow following GF fabric saturation downstream from the coating bead, a

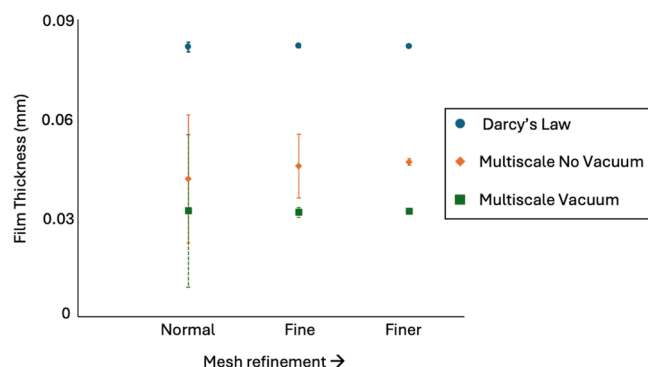


FIGURE 4 | Progressive error reduction with mesh refinement for macroscale model using Darcy's Law, and Multiscale Models with and without vacuum applied. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

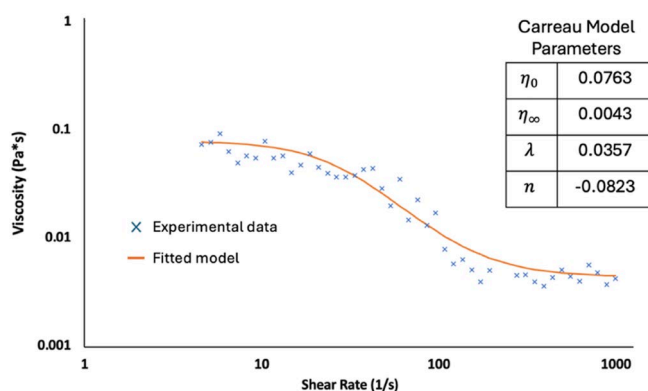


FIGURE 5 | Viscosity determined experimentally and Carreau model-fitting parameters for 5EM1CNC coating fluid. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

similar domain without the coating fluid inlet and gap is built, as shown in Figure 3b, with the vacuum pressure applied at the outlet driving the fluid flow out of the domain. As shown in Figure 3c, slip boundary conditions are introduced onto GF fiber surfaces due to the presence of interfacial slip within fibrous flow which impacts the mass flow rate [40, 41]. A detailed view of free triangular meshing within the microscale model is additionally shown in Figure 3d.

The inlet velocity is defined using the flow rate (Q) of slot die coating process divided by the area of the slot die opening, the reference pressure is set to 0, and the outlet pressure equals to reference pressure or p_v , if applied. The fluid penetration velocity is calculated by tracking the moving two-phase fluid-air interface within the GF fabric domain, and the resulting penetration depth (h) versus time (t) plot is used to calculate the leaking wall/penetration (v_l) velocities for the slot die coating model described by Equations (11) and (12). Gravity is neglected within the simulation due to its negligible influence compared to the coating, capillary, and vacuum pressures.

3.5 | Convergence Study

A predefined *physics-controlled* free triangular meshing is used for both macroscale and microscale modeling. For the macroscale model, to compare wet film thickness predictions across

three mesh size limits; these are named “Normal” size range of $0.175 \leq \Delta d \leq 61.3 \mu\text{m}$, “Fine” size range of $0.07 \leq \Delta d \leq 49 \mu\text{m}$, and “Finer” size range of $0.0263 \leq \Delta d \leq 22.8 \mu\text{m}$. The calculated wet film thickness determined at the three separate integration lines downstream in the macroscale coating model are plotted and found to converge to a single solution. The average wet film thickness across different mesh sizes for all macroscale models, that is, Darcy's Law, Multiscale without vacuum, and Multiscale with vacuum are summarized in Figure 4. Error bars representing one standard deviation are added to show the variation in wet film thickness calculated at each integration line. For the Multiscale model, as the coating thickness decreases, finer meshes are required to accurately predict the fluid interfaces correctly. For the microscale model, the “Normal” mesh size range between $0.162 \leq \Delta d \leq 36.2 \mu\text{m}$, and subsequent mesh refinement is performed using “Fine” setting of $0.162 \leq \Delta d \leq 28.6 \mu\text{m}$, and “Finer” $0.0675 \leq \Delta d \leq 2 \mu\text{m}$. For all meshes, the predicted time for the fluid to fully saturate the GF fabric domain varies by less than 1% for both no-vacuum and with vacuum conditions.

4 | Results

4.1 | Model Inputs

The surface tension of the coating dispersion was found to be 32.8 mN/m and the static contact angle between 5EM1CNC and the stainless-steel slot die, θ_{steel} , was measured as 56° . While previous contact angle measurements between 5EM1CNC and single E-GFs evaluated using the liquid bridge method were reported to be approximately 35° , the rapid absorption of the fluid into a GF fabric results in a changing contact angle, approaching 0° within 0.3 s from the first point of contact [42]. For the purpose of the macroscale model, the contact angle formed between the fluid and GF fabric is thus approximated as a perfect wetting condition where $\theta_{\text{GF fabric}}$ is set to 0° . However, to model the microscale flow within the fabric itself, the contact angle between the fluid and single fibers, $\theta_{\text{GF}} = 35^\circ$, is used [42].

For CFD modeling, the full viscosity curve of the 5EM1CNC coating dispersion, between shear rates 1 and 1000 s^{-1} , is fitted against the Carreau model as it exhibits regions of Newtonian plateau at very low and high shear rates, resembling values of zero-shear and infinite-shear rate viscosities [43]. The viscosity curve and fitted model parameters are shown in Figure 5.

Using the MicroCT, the void space unoccupied by the GFs was determined, giving a porosity (ϵ) of 35.2%. Using six separate SEM images, the porosity was found to be $38.1\% \pm 2.9\%$. The measurements given by the two separate methods were close, with the MicroCT measurement falling within one standard deviation of the porosity determined using SEM. Since MicroCT provides a more comprehensive measurement of a larger GF fabric volume, for the Darcy's law model a value of $\epsilon = 0.35$ is used. Using measurements from SEM images, an average GF radius, $R = 8.1 \pm 0.87 \mu\text{m}$, is reported.

4.2 | Microscale Model of Flow Through GF Fabric

To model coating flow through the 2D E-GF fabric geometry, both outlet conditions of no vacuum and with vacuum (p_v)

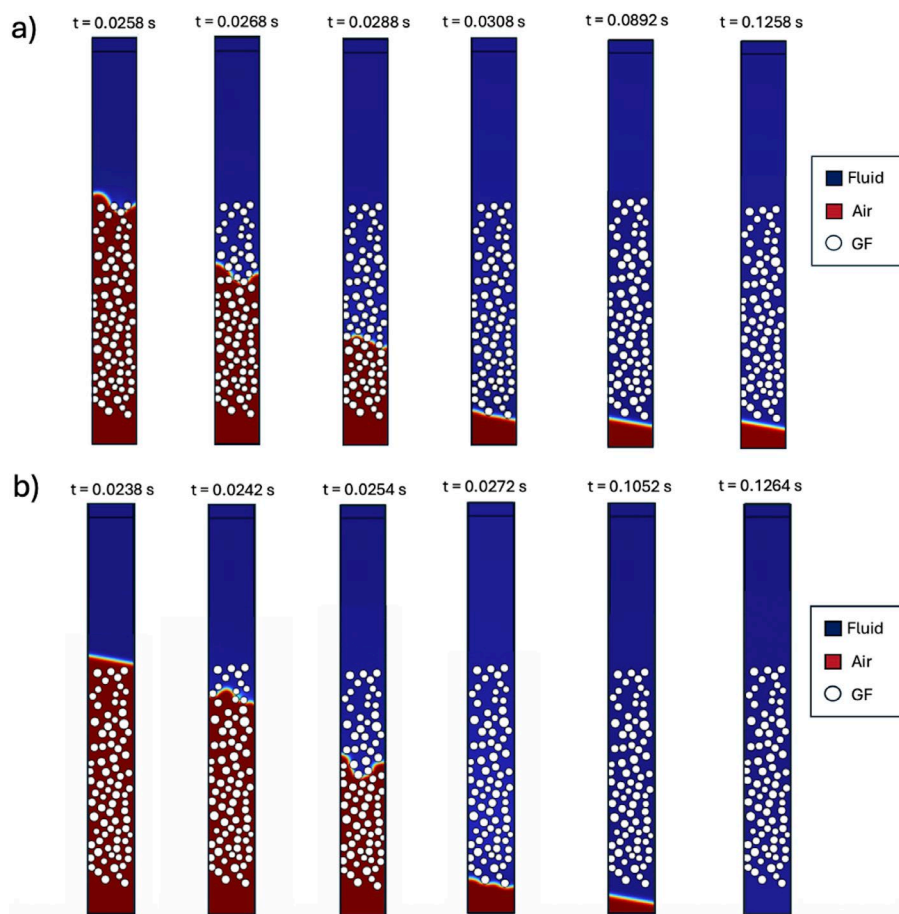


FIGURE 6 | Fluid penetration through GF fabric with (a) no vacuum applied, (b) vacuum applied. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

applied are tested. Using the measured total vacuum pressure at the vacuum plate inlet of -8.8 kPa, the value is divided by the total surface area of the vacuum plate holes underneath the GF fabric, yielding an average value of -21 Pa applied through each hole. While the value of $p_v = 21$ Pa is used to model the overall outlet conditions, the pressure mapping of the vacuum plate could provide a more accurate insight on the vacuum exerted on different areas of the GF fabric under real conditions. To calculate the velocity from initial penetration to saturation, the fluid penetration depth (h) is tracked as a function of time using the two-phase interface, as shown in Figure 6a,b, for no vacuum and vacuum conditions, respectively. In both cases, the fluid saturates the GF fabric rapidly until it reaches $h_f = 0.34$ mm and then slows down. However, the vacuum pressure applied at the outlet aids the fluid outflow from the domain with higher fluid velocity following saturation, as seen in Figure 6b and the $h(t)$ plot in Figure 7a.

The model convergence is checked using mesh refinement to verify that the predicted time to reach saturation converges to the same solution. For all meshes, the predicted time it takes for the fluid to fully penetrate the GF fabric domain varies by less than 0.7% for the no-vacuum conditions and less than 0.8% for the model with p_v . The penetration velocity (v_i) is calculated as a function of the penetration depth and time using the expression $(h_{i+1} - h_i) / \Delta t$, and the resultant velocities are applied to the macroscale leaking wall model to define fluid penetration using

Equation (10). The steady state pressures of the no-vacuum and vacuum model are plotted in Figure 7b,c, respectively, showing that the non-zero pressure of p_v at the outlet continues to drive coating flow through the GF fabric even following saturation; this continued removal of fluid through the domain plays an important role in reducing wet film thickness which is reflected in experimental trials using vacuum-assist [14].

To model the fluid flow through the GF fabric, as it transitions from being driven by capillary and coating bead pressures to being driven solely by the applied vacuum pressure (p_v) at the outlet, a second model simulating flow out of a saturated domain where inlet $Q = 0$ mL/min is used. The same method of tracing the two-phase interphase is used to calculate velocity, v_i , which represents the rate decrease in wet thickness, using the gradual drop in fluid level above the GF fabric over time, as shown in Figure 8a,b. The application of vacuum reduces wet film thickness by $12\mu\text{m}$ within 8 s, though the rate diminishes with time. The calculated v_i is used in the multiscale approach by mapping onto the leaking wall of the macroscale model using Equation (11) downstream from the coating bead to model the continued fluid removal facilitated by p_v . The drop in fluid level along with v_i is plotted in Figure 8c, showing the continued yet slow impact of vacuum on reducing wet film thickness on top of the fabric. The steady state pressure is plotted in Figure 8d, showing the applied p_v at the outlet and reference pressure of 0 Pa at the top of the domain.

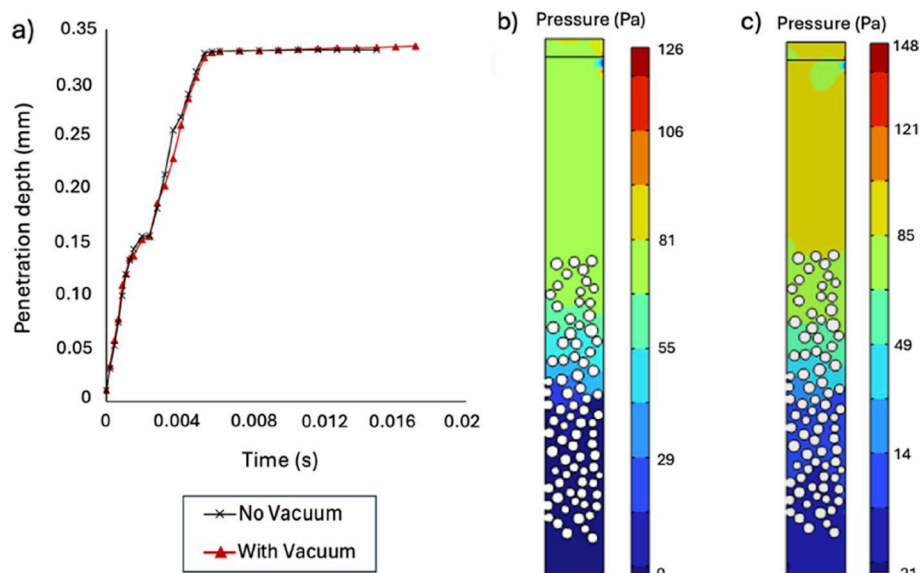


FIGURE 7 | (a) Penetration depth $h(t)$ plotted for both no-vacuum and vacuum model, and steady state pressure throughout the domain for (b) no-vacuum and (c) vacuum model. [Color figure can be viewed at wileyonlinelibrary.com]

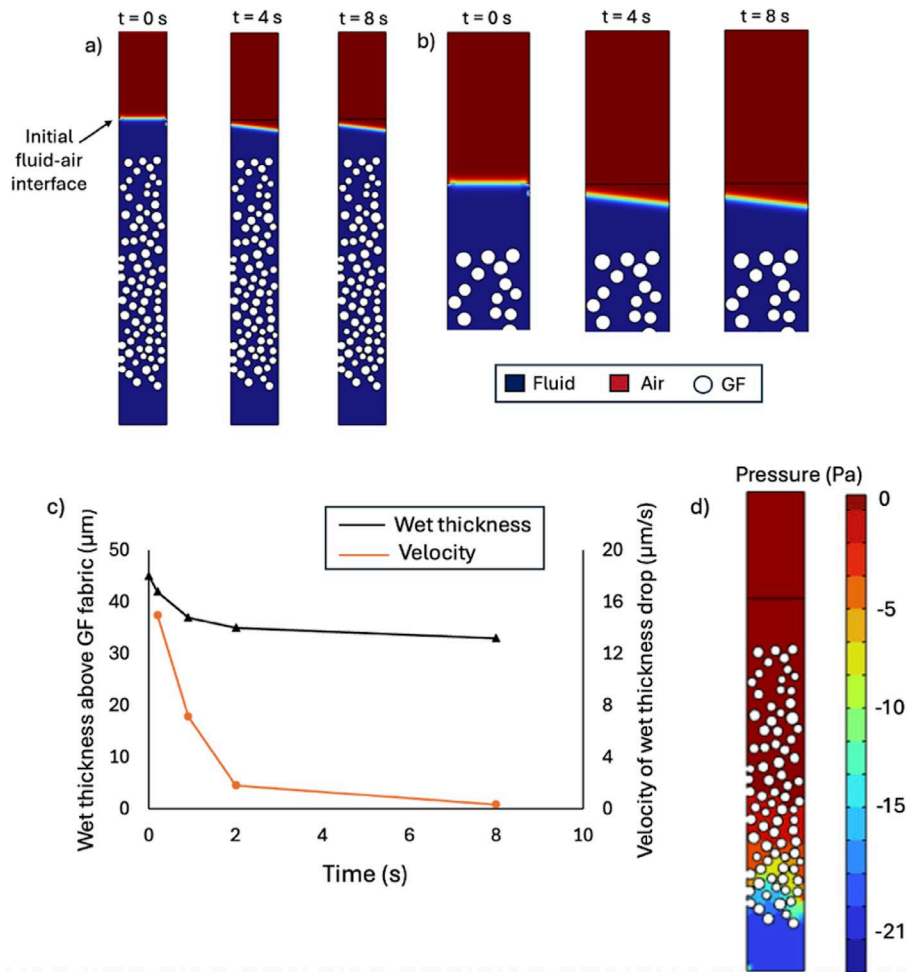


FIGURE 8 | Fluid removal from GF fabric domain driven by vacuum pressure (p_v) applied at the outlet shown for (a) the full domain, and (b) close ups above the GF fabric. (c) Change in measured wet film thickness and velocity of wet film thickness decreases above GF fabric over time, and (d) steady state pressure throughout the domain with p_v applied at the outlet. [Color figure can be viewed at wileyonlinelibrary.com]

4.3 | Bead Stability Prediction

Both the macroscale only and multiscale model are used to predict operating parameters Q and v , and the results are experimentally validated; no vacuum is applied. Two test cases are simulated, Combination A: $Q=3\text{ mL/min}$, $v=5\text{ mm/s}$, and Combination B: $Q=5\text{ mL/min}$ and $v=5\text{ mm/s}$, and the coating stability results are summarized in Table 1. For Combination A, the macroscale model predicts a stable coating bead with concave upstream and downstream menisci pinned at the corners of the slot die, producing a continuous wet film with no defects or breakage, as shown in Figure 9a,b. Using Combination B, the macroscale model shows the downstream and upstream menisci rising above the edges of the die lips, as shown in Figure 9c. This

TABLE 1 | Coating outcomes for macroscale model, multiscale model, and experimental evaluation.

Method	Coating flow rate (Q) (mL/min)	Web velocity (v) (mm/s)	Coating outcome
Macroscale + Darcy's Law	3	5	Stable
Macroscale + Darcy's Law	5	5	Dripping defect
Multiscale	3	5	Unstable
Multiscale	5	5	Stable
Experimental	3	5	Unstable + Break line defect
Experimental	5	5	Stable

observation is associated with a Q becoming too high for a given v , at which the defect of dripping begins to appear, describing the phenomenon where the fluid accumulates behind the upstream die-lip and begins to drip down onto the substrate [44].

The multiscale model returned different predictions. Using Combination A, the model predicts an unstable coating bead which breaks, as shown in Figure 10a since the chosen Q is too low for both the established web velocity and the rapid fluid penetration. In contrast, raising the coating flow rate to $Q=5\text{ mL/min}$ to match Combination B, a stable coating bead was produced, reaching steady state without breakage, as shown in Figure 10b. The predicted coating bead shape is also notable, showing the upstream meniscus moving right into the slot gap, a phenomenon that typically occurs when the minimum wet thickness boundary is approached, signifying that Q could not be further reduced for a given v without the appearance of ribbing or break-line defects [29, 44].

Experiments carried out using Combination A and Combination B parameters reflected the findings of the multiscale model. As shown in Figure 11b, Combination A parameters yielded an unstable coating bead with breakage, producing an incomplete coating with dry spots, that is, break-line defect, with bright reflective uncoated GF fabric visible; a reference image of uncoated GF fabric is shown in Figure 11a. The results imply that the chosen Q and v combination falls outside of the operating limits, specifically below the lower bounds of a coating window. Experimentally increasing the flow rate to $Q=5\text{ mL/min}$ was found to be necessary to produce a consistent coating and coverage of the E-GF fabric, as shown in Figure 11c, with the opaque white coating providing complete coverage over the GF fabric. These findings also reflect the stable coating parameters tested in [14] for coating E-GF fabric.

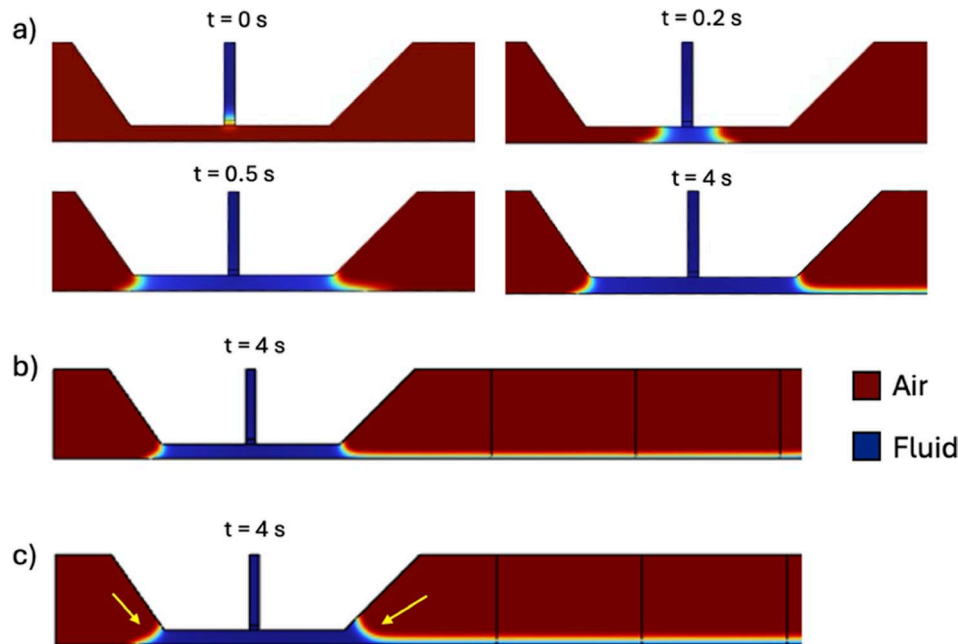


FIGURE 9 | Macroscale model predictions showing (a) a timelapse of coating bead formation and shape from $t=0$ to 4 s, (b) an extended view of the slot die coating domain at steady state for parameters of Combination A, and (c) of Combination B. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

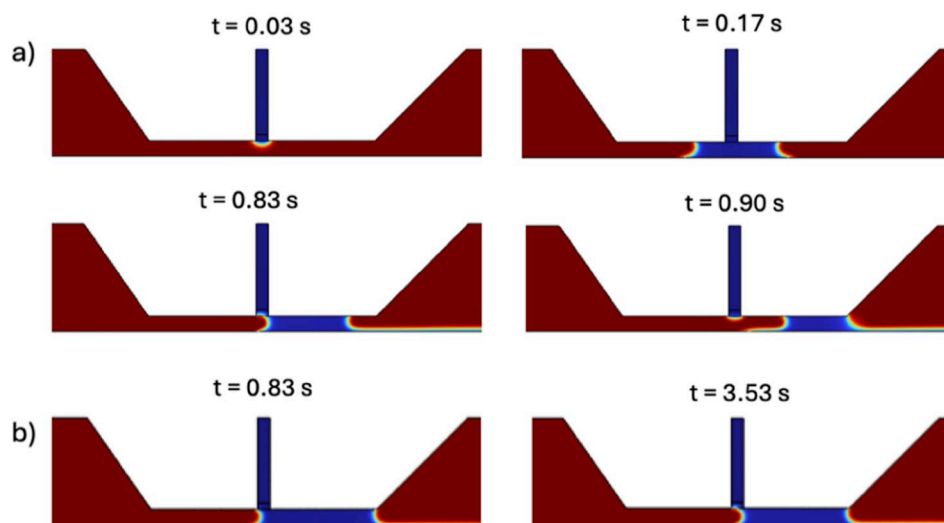


FIGURE 10 | (a) Coating bead at Combination A, showing instability and breakage at a time stamp of $t = 0.90$ s, and (b) coating bead at Combination B showing stable coating bead shape at steady state. [Color figure can be viewed at wileyonlinelibrary.com]

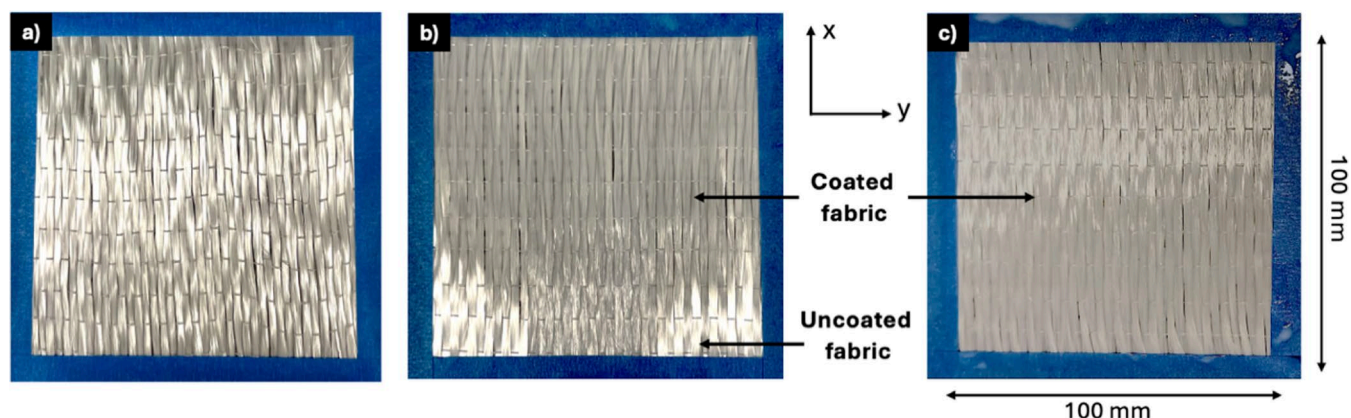


FIGURE 11 | GF fabric (a) uncoated and coated using (b) Combination A: $Q = 3$ mL/min and $v = 5$ mm/s, and (c) Combination B: $Q = 5$ mL/min and $v = 5$ mm/s. [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 2 | Comparison of film thickness model predictions and experimental values.

Method	Vacuum	t_{wet} (μm)	t_{dry} (μm)	Error (μm)
Experimental	No	N/A	1.49 ± 1.19	N/A
Experimental	Yes	N/A	0.84 ± 0.61	N/A
Macroscale	No	82	4.20	2.71
Multiscale	No	47	2.40	0.91
Multiscale	Yes	32	1.64	0.80

4.4 | Film Thickness Prediction

Since Combination B was found to produce steady operating conditions experimentally, the film thickness produced at these parameters are compared to macroscale and multiscale predictions. The multiscale model was additionally evaluated for the ability to predict film thickness for cases with applied vacuum pressure.

Since the models can only predict wet film thickness (t_{wet}), the conversion to dry film thickness (t_{dry}) is carried out using the mass fractions and densities of the constituents as described in Section 2.5. Refinement of control point spacing (Δs) did not significantly impact the wet film thickness predictions, differing by less than 4.5% suggesting solution convergence. The film thickness of both models, along with experimental measurements taken from [14], is summarized in Table 2. The error of each model prediction compared to experimental data is also included.

Echoing the findings from prior inaccurate coating bead stability predictions, the macroscale model falls short in predicting film thickness by a large error of 180% when compared to average experimental measurements. Using the multiscale model to simulate the coating process and fluid penetration yields values closer to the experimental measurements although the model continues to significantly overpredict the film thickness in both no-vacuum and vacuum cases, exceeding the measured mean film thickness by similar absolute errors of 0.91 and 0.80 μm . Given the high standard deviation of the measured coating film thickness due to the uneven surface heights and the difficulty of measuring in situ wet film thickness on the coated GF fabric,

establishing a reliable conversion between wet and dry thickness remains a gap in achieving better model validation.

However, the multiscale approach does clearly outperform the macroscale-only model. Examining the pressure plots in Figure 12, the plot for the macroscale and multiscale models differ significantly, although both show the initial penetration region with high pressure generated primarily by the fluid being pulled into the GF fabric by the dominant p_c . Where the macroscale model uses the Young–Laplace equation to calculate a constant p_c until saturation as shown in Figure 12a, the multiscale model shows variation in pressure caused by irregular GF packing causing local fluctuations and resultant fluid velocity, as shown in Figure 12b. The influence of vacuum can be observed from the plot as well, showing that the model with p_v sustained higher outlet pressure applied at the leaking wall boundary, driving the continued removal of coating fluid following saturation, resulting in changes in the wet film thickness.

The inadequacy of using 1D Darcy's law to describe fluid penetration into GF fabric can be explained by the numerous simplifying assumptions. The original implementation of this approach for slot die coating carbon paper, as described in [19] stated the assumption that the wetting length of the coating bead is much longer than the penetration depth. While this assumption may hold true for porous media with low permeability exerting low capillary pressure, the rapid wetting of GF fabric, stemming from large p_c may render this condition untrue. Within the GF fabric model, the penetration depth is found to be within one order of magnitude of the wetting length of 0.1 mm, which diverges from previous applications where the penetration was only on the order of single microns [19]. These findings support the conclusions from Goda et al.'s study which modeled a similar process of slot die coating of hydrophilic porous media on a curved surface and has noted that the 1D depth-dependent penetration approach was not representative of systems for which $p_c \gg p_i$ [22]. However, this model still assumed an isotropic porosity and constant pore diameter, which are criteria not met by complex porous media or fibrous materials like a GF fabric.

Further inaccuracy may stem from the simplified representation of a GF fabric as an array of parallel cylinders with regular spacing in between. The Happel model, which was used to approximate GF fabric permeability is a popular approach

for modeling fluid flow across aligned cylinders; however, it relies on the independent unit cell model concept in which the GFs are individually spaced without touching, and the GF fabric has a constant porosity value [32, 45, 46]. As was shown in SEM images in Figure 2, the fabric is made of irregular arrays of GFs with occasional tight packing and large pockets. While the Happel model, may apply to selected regions with high porosity, it offers a poor approximation for areas with higher volume fractions of fibers (V_f), that is, $V_f = 0.6$ or higher, failing to account for changing tortuosity and geometric randomness [47]. Within lower porosity regions, lubrication theory is often more suitable for modeling fluid flow [46]. To more accurately represent fluid flow through GF fiber fabrics with irregular GF distribution, improvements could be achieved by using unified models that combine both the cell model and lubrication theory with governing equations, similar to ones described in [46, 48]. The calculated capillary pressure (p_c) which drives the fluid penetration into the GF fabric experiences similar pitfalls to the permeability calculations, as it also relies on a constant effective pore diameter D_e , calculated from a constant radius (R) and constant porosity (ϵ). This approach results in a uniform p_c , as plotted in Figure 12a, a prediction that is incongruent with the real GF fabric geometry.

The microscale model using an RVE of the GF fabric allows for greater accuracy in representing GF fabric geometry including the uneven porosity and GF distribution throughout the entire GF thickness. However, the size of the RVE is also a major factor, with smaller structures inadequately capturing the alternating weave of GF fabrics. In this study, the small RVE could be a source of error as it was produced based on a GF roving within the fabric without capturing the presence of polyester yarn (i.e., weft) that horizontally weaves the otherwise unidirectional fabric. Even so, the multiscale approach allows for improved representation of fluid penetration dynamics, accurately predicting stable coating parameters and film thickness closer to experimental measurements over a purely macroscale model.

5 | Conclusions and Future Works

In this study, a multiscale CFD modeling approach was taken to simulate the slot die coating of CNC dispersions onto GF fabric

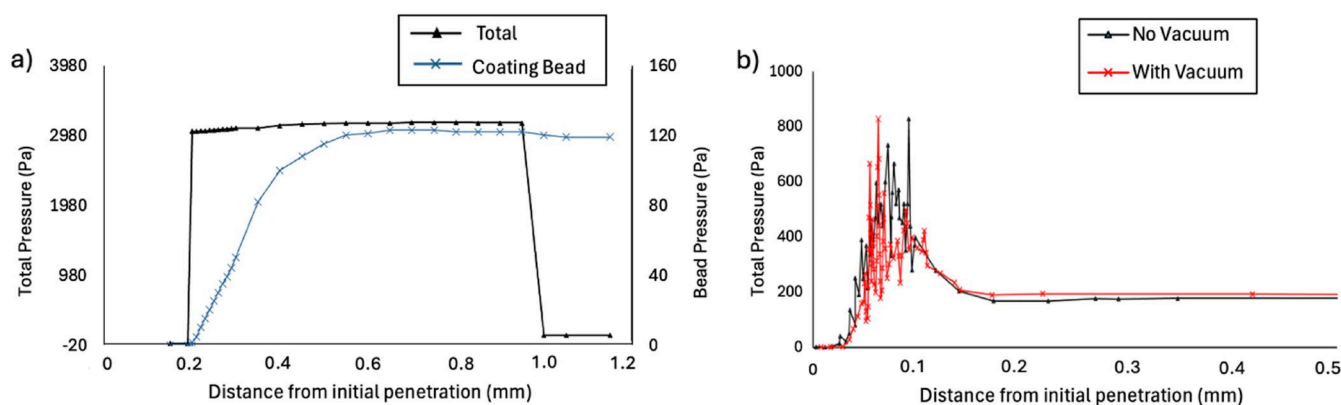


FIGURE 12 | Coating pressure plots for Combination B for (a) macroscale model, (b) multiscale model. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

for systems with and without vacuum assist. The multiscale modeling approach yielded coating bead stability predictions congruent with experimental coating trials and also outperformed typical macroscale-only models, being able to directly capture the primarily capillary pressure-driven fluid penetration within the anisotropic substrate geometry relying on simplified equations for porous flow.

While the current models can be used to predict coating bead stability, producing accurate film thickness predictions remains a major challenge. Expanded models of both the microscale model, capturing a larger cross section of the GF fabric, and a larger macroscale model with an expanded downstream region could both be implemented to increase the accuracy of the overall fluid penetration and removal. Better predictions and conversions between wet film thickness and dry film thickness could also be targeted by exploring more accurate in situ wet film thickness measurement using finer wet film thickness gauges.

To provide improvement to the current domain, 3D dynamic CFD models which simultaneously capture the substrate movement along with the fluid penetration into a GF fabric are under development, providing a more detailed representation of GF fabric structure. This approach would simplify the model inputs by removing the need for the geometric mapping between two separate models, thus overcoming the difficulty of decoupling and re-coupling of coating regions. Furthermore, a 3D model would offer a clearer prediction of fluid volume distribution across the GF fabric surface and also allow for the establishment of coat weight to film thickness conversions.

Overall, this work provides a pathway forward for the use of multiscale CFD modeling for predicting operating parameters and coating outcomes for vacuum-assisted slot die coating, a novel scalable method for sizing GF fabrics in CNCs.

Author Contributions

Robert J. Moon: writing – review and editing, supervision, resources, methodology. **Kyriaki Kalaitzidou:** funding acquisition, writing – review and editing, methodology, supervision, resources. **Apoorva R. Mundhe:** investigation. **Kim Anh Pham:** investigation, conceptualization, writing – original draft, writing – review and editing, software, validation, formal analysis, data curation, methodology. **Tequila A. L. Harris:** conceptualization, writing – review and editing, methodology, supervision, resources, funding acquisition.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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