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SYNERGISTIC STRATEGIES IN MANUFACTURING OF WOVEN GLASS FIBRE AND POLYPROPYLENE COMPOSITES: STAGED-COMPACTION AND MULTI-MODAL IMAGING FOR POROSITY QUANTIFICATION

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Abstract

This experimental study evaluates multi-modal imaging techniques for characterizing microstructures in partially impregnated thermoplastic matrix composites made from woven glass fibre (WGF) and polypropylene (PP). The research focuses on the impregnation quality of composite plates manufactured through a displacement-controlled thermo-compression method called Simplified Compression Resin Transfer Moulding (CRTM). This method, which bypasses the injection stage, allows matrix flow through the plate thickness, simulating CRTM's impregnation phase. For comparison, a reference plate was produced using a film stacking method. The study employs extended field 2D imaging techniques like Polarized Light Microscopy (PLM), Fluorescence Microscopy (FM), and Scanning Electron Microscopy (SEM) to visualize microstructures at the fibre scale throughout the plate. These techniques are integrated via correlative imaging to generate detailed synthetic images, providing quantitative assessments of glass fibre and porosity area fractions. Results indicate the method's sensitivity to fibre breakage, with porosity levels around 8.2% and 8.7% in layers 1 and 5 of the reference plate, respectively. Notably, the Simplified CRTM plate shows a transverse polymer flow, highlighting the potential to characterize meso-scale microstructure in a 0/90 composite stacking configuration. These findings underscore the need for further development to quantify uncertainties and enhance understanding of fluid-structure interactions in CRTM processes.

1. Introduction

Compression Resin Transfer Molding (CRTM) is a manufacturing process categorized under liquid composite moulding. In CRTM, a polymeric resin in the liquid state is forced to flow through a fabric of reinforcing fibres, filling the spaces between individual fibres (micro-scale) and between fibre bundles (meso-scale). The impregnation vector during CRTM can be controlled by applying compressive mechanical pressure or a controlled displacement at the scale of the preform's thickness (macro-scale). During compression, the applied pressure reduces the porosity and thus, alters the permeability of the preform, promoting resin flow by creating streamlined pathways between and within the fiber bundles, which consequently fills the empty spaces. This ensures close adherence of the polymeric matrix at the liquid state to the solid fiber components, facilitating effective saturation of the initially dry preform and reducing porosity. For more detailed insights into how analytical models and a detailed understanding of these physical forces interact with fluid dynamics to optimize resin distribution and quality in CRTM [1]. A significant challenge in CRTM processes is monitoring the transverse flow of the resin in the thickness direction of a dry preform, where a fully saturated liquid zone transitions to an unfilled, non-saturated fabric layer. This scenario is complex to investigate in the case of a thermoplastic matrix as it can be influenced by multiple factors such as gravity, pressure gradients, the permeability, temperature gradient and capillary effects within the fabric's microstructure. Analytical models like Darcy's Law may not adequately capture the anisotropic and non-linear behavior of resin flow. Advanced computational fluid dynamics (CFD) and real-time experimental monitoring

are still needed for better simulation of these dynamics, ensuring even saturation of the resin throughout the fibrous preform and mitigating potential dry spots. Both numerical and experimental strategies require a precise understanding of permeability variation, fiber alignment, and the interactions between the resin and fibers, as well as the physical length-scale of such interactions. The scientific question addressed by the current study in relation to composite manufacturing focuses on the experimental microstructure characterization for evaluation of impregnation quality, specifically the saturation degree of intra-bundle spaces post-manufacturing. The goal is to assess the potential of a new manufacturing protocol termed simplified CRTM to generate transverse-dominated flow of a thermoplastic matrix through a non-woven fabric of glass fibers.

For microstructure characterization, both destructive and non-destructive imaging methods can be employed to conduct qualitative and quantitative analyses of the microstructure of residual porosity in polymer matrix composites. Each specific imaging technique provides physically-anchored information about the microstructure, encoded in multidimensional spatial arrays known as “images”. A comprehensive survey of commonly used imaging techniques for microstructure characterization in polymer matrix composites illustrates the diversity of techniques employed. Optical microscopy (OM) leverages electromagnetic radiation in the visible spectrum to resolve macro- and meso-scale structural details. Although effective for surface inspections, it is constrained by the diffraction limits of visible light, limiting its utility for extensive samples length scales when fine microstructural details are required. For example, Kabachi et al. [2] employed OM to inspect cross-sections of fiber bundles and their deformation under load. Similarly, Liu et al. [3] applied OM to analyze porosity in carbon fiber reinforced epoxy prepregs, quantifying pore size and distribution influenced by varying pressures. More advanced OM protocols, such as those used by Ishida et al. [4], were employed to visualize in-situ resin flow and impregnation dynamics in thermoplastic composites during compression processes, providing insights into bulk material behavior under applied stress. For higher resolution and depth of analysis, Khaled et al. [5] utilized Scanning Electron Microscopy (SEM) in addition to OM for structural analysis. SEM employs a focused beam of high-energy electrons to interact with sample materials at the atomic level, generating various signals that reveal information about surface topography and composition. This electron-sample interaction allows for detailed analysis of microstructures down to a few nanometers, far beyond the capabilities of OM. Polarized Light Microscopy, utilized by Li, Yixian, et al. [6] employs polarized light to enhance contrast in birefringent materials, such as certain types of fiber composites. This technique exploits the differential absorption of polarized light by the material to enhance visual contrast and reveal hidden details crucial for analyzing fibre orientation and matrix organization. Fluorescence Microscopy, though less commonly reported for polymer composites, uses the excitation of fluorophores within the material by specific wavelengths, typically in the UV range. The subsequent emission at longer wavelengths is captured to produce high-contrast images of specific components of the matrix or fibers within the composite. Recent advancements include X-ray microscopy, which employs short-wavelength X-rays to penetrate composite materials, providing volumetric imaging capabilities without the physical sectioning required by traditional microscopic techniques. This method is essential for identifying internal defects and understanding the three-dimensional distribution of fibers and voids within composites. Ayadi et al. [7] explored the capabilities of standard laboratory-scale absorption-based micro-computed tomography (μ CT) and helical μ CT, which use X-ray computed tomography to create cross-sectional images of composites non-destructively. μ CT provides high-resolution (between 2 and 6 μ m), three-dimensional images from X-ray linear attenuation coefficients, allowing for detailed internal characterisation and quantification of porosity, fiber alignment, and crack propagation within composites.

Based on a non-exhaustive literature review, various imaging techniques have been shown to be effective for both qualitative and quantitative inspection of composite microstructures. However, several issues remain ambiguously addressed, including: (i) While multiple imaging techniques are available, studies typically do not provide comparative analyses of the same region of interest (ROI) under different imaging techniques. (ii) Most research relies on statistical comparisons of quantitative results obtained from the respective techniques, often omitting any quantification of uncertainty. (iii) The analysis of partially impregnated composite samples using 2D destructive imaging techniques necessitates extensive surface preparation, such as polishing. Unfortunately, the quality of these prepared surfaces is poorly documented within the existing literature. (iv) While the examination of a

region of interest may require observation of the entire thickness of composite samples, there is often a lack of consideration regarding the trade-off between the field of view, the smallest detectable detail, and the achievable resolution. In this study, the second main scientific aspect to address is about the efficiency of multi-modal imaging approach based on 2D destructive techniques for impregnation quality analysis using extensive field acquisitions with polarized light microscopy, FM, and SEM.

This study evaluates the impregnation quality based on microstructural characterization of thermoplastic matrix composites manufactured through a Simplified CRTM process, which uses a stack of thermoplastic films that liquefy under processing conditions to create a low-viscosity matrix. By controlling compaction ratios, the study examines how polymeric resin saturates a woven glass fibre and polypropylene matrix, aiming to understand resin distribution and localize porosity. Additionally, multiple imaging techniques—including Polarized Light Microscopy, Fluorescence Microscopy, and Scanning Electron Microscopy—are employed and correlated to enhance the resolution and accuracy of evaluations for porosity and fibre alignment through multi-modal imaging. The integration of these techniques provides a comprehensive assessment of the composites' internal structure, contributing to the optimization of a CRTM process designed for a transverse flow dominated scenario of impregnation. During the impregnation phase, displacement-controlled compaction is used, and the progression of fluid flow depends on the coupling between mechanical deformation of the preform, fluid pressure, and applied temperature, assuming isothermal conditions. As compaction reduces the thickness of the fibre bed, fibre volume fraction increases, thus decreasing permeability [2]. This, along with the high viscosity of the matrix, makes impregnation challenging, leading to potential defects like unfilled regions or trapped air bubbles that may compromise the integrity and mechanical properties of the final product.

2. Materials and Methods

2.1 Materials

The matrix material used in the current study was a commercially available polypropylene (PP) thermoplastic, PPC13442 from Total®, with a density of 0.905 g/cm³, a melting temperature of 165 °C, and a melt flow index of 100 g/10 min. The PP was supplied as pellets and converted into thin films via thermocompression, resulting in films with an average thickness of 0.572 mm (±0.032 mm). The reinforcement used was a unidirectional (UD) woven glass fiber (GF), type 8089 HM from Chomarat®, characterized by a density of 2.55 g/cm³ and an areal weight of 1054 g/m². Rectangular UD plies, each 375×375 mm², were manually cut from larger sheets.

2.2 Methods

2.2.1 Fabrication of composite plates:

Composite plates were fabricated as flat, rectangular specimens of 375×375 mm². The glass fiber preform included six UD plies in a [0/90]₃ stack. This study assessed the efficacy of multimodal 2D imaging techniques in evaluating meso scale PP impregnation into the fiber bundle microstructure through two different manufacturing configurations. Configuration 1 is based on film stacking based on alternation between GF plies and PP thin sheets (Fig.1.a) with no applied pressure after adjusting the height of the mold's cavity. The film stacking based approach is used to allow the observation of the microstructure of undeformed fiber bundles. Configuration 2 is a simplified CRTM version, where the solid-state PP sheets are all placed below the GF stack (Fig.1.b). Upon heating, the PP melted, forming a liquid pool. Despite the high viscosity of PP, which minimized capillary effects and made them insufficient for initiating polymer flow into preform gaps, mechanical pressure during compression facilitated the matrix impregnation inter and intra the GF bundles. Impregnation quality varied due to the trade-off between the mechanical deformability of the preform and decreased permeability, which could lead to residual porosity, including dry zones or trapped air bubbles, or PP shrinkage post-cooling.

2.2.2 Manufacturing platform:

Composite plates were produced using an industrial-scale press (Pinette PEI®) capable of exerting up to 120T of force with control over force (respectively displacement) with a precision of ± 4kN (respectively of ± 0,1 mm). The press features programmable cycles for temperature, displacement, and force, ensuring consistent manufacturing conditions. The manufacturing process included a ten-segment program designed to heat, then sequentially compacting the preform, and then cool the material systematically. The first two segment heat the mold, followed by six segments of staged compaction of 5 minutes interval each to impregnate the reinforcement and achieve the desired thickness. The two final

segments cool the plates while maintaining mold height. During this study four plates were manufactured under controlled compaction ratios (Cr) as expressed in Eq.1. The focus will be limited to two of the plates from the batch having a Cr of 0% and 41% respectively.

$$CR = \frac{h_{ref} - h_{final}}{h_{ref}} \quad (1)$$

Where h_{ref} is height of the plate manufactured using configuration 1 and h_{final} is height of consecutive plates manufactured using configuration 2.

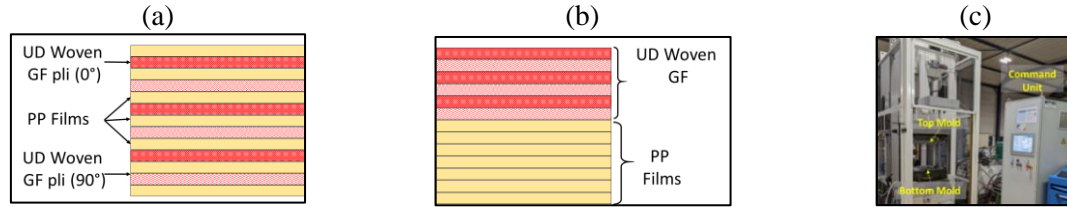


Figure 1. (a) Configuration 1, (b) configuration 2 and (c) Manufacturing Pinette PEI press

2.2.3 Sample preparation: Polishing of partially impregnated composite plates

The manufacturing of composite plates often results in variable microstructure characteristics due to different levels of resin impregnation, leading to residual porosity and varying saturation levels across the preform. Traditional surface preparation techniques, such as mechanical polishing, may inadvertently damage the microstructure, particularly in areas with loosely impregnated fibers in the 0° and 90° orientations. To address these challenges, a four-step polishing protocol was developed to minimize damage while preparing the composite surface for image-based analyses. The first step corresponds to an initial embedding of the sample by using a mixture of EpoFix from Struers®, a commercial embedding resin, blended with EpoDye, a fluorescent dye from Struers®. This step helps secure the sample and fluorescent epoxy infiltrates all open porosities. The second step corresponds to gradual polishing using abrasive discs of 500, 1200, and 4000 grit on an automated polishing machine (TegraMin, Struers®). Parameters such as force, rotational speed, and duration were precisely controlled to minimize fiber damage of the partially impregnated samples. The third step consists of a secondary embedding for the exposed surface following the polishing step, by applying for a second time the fluorescent dye-enriched epoxy to the exposed surface areas to stabilize fibers and seal porosities exposed during the first polishing. The end step consists of final polishing and for mirror finish using 500, 1200 and 4000 grit discs to remove excess resin while preserving the fluorescent layer, particularly around transversely (90°) oriented fiber bundles. Additional finishing steps include using 6µm and 3µm diamond particles to refine surface quality and enhance the circularity of fibers, particularly in the 0° oriented bundles (Fig.2.a).

2.2.4 Microstructural Characterization Using Multi-modal Imaging Techniques

The characterization of microstructural features in partially impregnated composite materials requires revealing detailed information about material composition, structural integrity, and the presence of defects such as porosities and dry zones. To this end, the current study focused on relatively classical laboratory scale techniques for a comprehensive suite of imaging modalities designed to address these challenges. A numerical microscope (Axio Zoom V16, Zeiss®) equipped with both white and UV light sources and numerical camera for collecting polarized light microscopy and fluorescence microscopy. Its automated displacement-controlled stage and X80 magnification lens allowed for extended field imaging across multiple fiber bundle cross-sections throughout the sample's thickness (Z-direction). A scanning electron microscope (JCM6000, Jeol®) operating in Backscattered Electron Detector – Composition mode (BED-C mode) was used at X200 magnification to collect extended field images. The lack of an automated stage necessitated a manual method for capturing overlapping images, which are later stitched numerically to generate a comprehensive view of the extended microstructure.

2.2.5 Detailed analysis techniques

- Polarized light microscopy (PLM) was used to control the state of the polished surfaces as it augments contrast in anisotropic materials. PLM can reveal subtle differences in material properties, such as birefringence, that are indicative of the orientation, integrity of fibers and of the polished surface.

This method excels in identifying areas with structural anomalies like scratches or rough zones, which are critical for assessing the quality of the polishing process (Fig.2.b).

- Fluorescence microscopy (FM) was used in the current study to enhance the visualization of porosity areas (i.e. dry zones and open air pockets) within composites by exciting fluorophores with UV light. These molecules emit light at a different wavelength, enabling the distinction of stained regions that indicate porosities or dry zones (Fig.2.b).
- Scanning electron microscopy (SEM) in BED-C Mode is used for detailed compositional analysis of the polished surface of the composite samples. This technique involves directing an electron beam onto the sample surface, where heavier elements (such as glass fiber) cause more electrons to scatter back. The BED-C mode captures these backscattered electrons to generate high-resolution images that provide contrast based on atomic number variations. This method is especially effective for mapping the distribution of different phases (i.e. robing, PP matrix and glass fiber) within the samples (Fig.2.b).



Figure 2. (a) Surface state evolution following during first polishing (step 2) and following mirror finishing (step 4). (b) Images of the same surface polished at 4000 grit following the first robing step and observed using OM, PLM, FM and SEM.

2.2.6 Multi-modal imaging approach:

The integrative use of multi-modal imaging techniques provides a robust framework for characterizing the microstructure of partially impregnated composite samples. PLM assesses surface quality and identifies defects such as scratches, crucial for evaluating the polishing process. FM enhances visualization of internal inconsistencies, such as uneven impregnation and porosities, by exciting fluorophores that emit light at specific wavelengths to highlight these features. SEM based on BED-C mode delivers precise compositional mapping, essential for analysing glass fibers. This multi-modal approach allows for a comprehensive analysis, each technique providing unique insights that contribute to a detailed assessment of the microstructure, encoded in a 2D spatial array where each voxel represents specific physical properties.

2.2.7 Extended field analyses: Micro/Meso/Macro length scales:

Extended field acquisition is a valuable technique that involves creating a grid of elementary images, which are subsequently stitched together to provide an expansive and detailed view of the microstructure. This approach enables the visualization of a large area while maintaining high resolution, crucial for detailed local analysis and accurate segmentation of the microstructure.

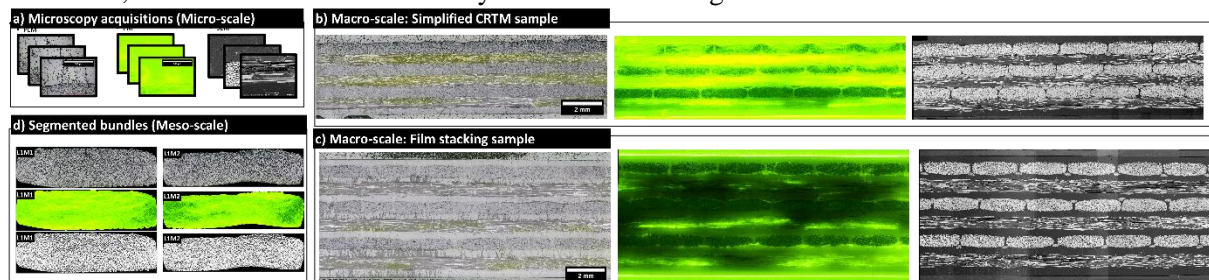


Figure 3. (a) Localized images from extensive field analysis. (b) Extended field images of the composite produced using simplified CRTM. (c) Extended field images of the composite produced using film stacking. (d) Illustrations of fiber bundles extracted from the extended field images.

In this study, the technique was specifically employed to include at least six full bundle cross-sections and the entire thickness of the composite plate, ensuring comprehensive coverage. The resolution was carefully calibrated in line with the magnification levels of the employed imaging methods. A standard magnification of X80 was utilized with the Zeiss microscope to clearly visualize individual fibers, while a higher magnification of X200 was applied in SEM analyses using the Backscattered Electron Detector – Composition mode (BED-C) to capture finer structural details. This strategy ensures that both macroscopic and microscopic features of the composites are captured with clarity and precision, facilitating a thorough investigation of the material's properties.

2.2.8 Multi-modal imaging post-processing workflow

The considered workflow relied on collecting extended field images based on PLM, FM and SEM. As the same microscope was used for PLM and FM, the registration operations (which corresponds to the alignment of the extended field images) of PLM and FM images were limited. More precaution was attributed to resizing the extended-field SEM images and then applying a rigid registration method based on superposition of control points between the SEM and the polarized light microscopy images. By combining the images obtained from FM and SEM, useful information such as inter/ intra bundle residual porosity and matrix localization, bundle shape, single fiber cross section can be obtained to carry out qualitative and quantitative analysis. All image processing operations relied on open source Fiji/ImageJ software. In a second step, a correlative imaging technique was applied by combining the extended-field images to generate a synthetic extended-field image of more detailed structural information. For this purpose pixel classifications were conducted on SEM extended field images to extract 5 classes representing the glass fibers, PP, Robing epoxy, unfilled spaces and pixels at the edges of glass fibers (Fig.4). A five class pixel classification was also performed on FM extended field images where the degree of intensity of green color was associated to the concentration of the fluorescent agent. Five levels of fluorescent concentrations were identified where level 0 corresponded to absent of fluorescent agent of contrast and level 4 corresponded to the highest level corresponding to local pixel saturation. All classification operation of pixels were conducted using machine learning approach based on random forest classifications and using the open software Illastik [8], [9].

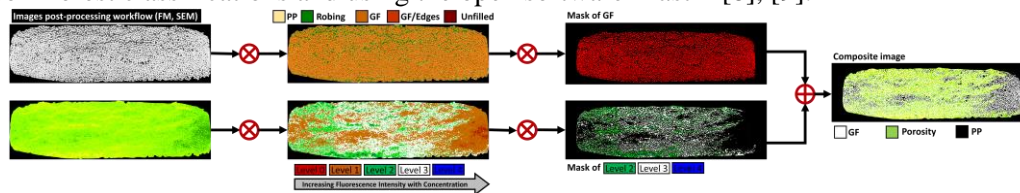


Figure 4. Correlative imaging workflow

3. Results: Quantitative Analysis of Composite Materials

3.1 Area Fraction Quantification

The quantitative analysis of glass fiber area fractions in the composite plates revealed distinct variations dependent on the manufacturing method and the specific layers within the composite structure. For plates produced via the simplified CRTM method, the glass fiber area fractions were quantified as follows: 49.1% in layer 1, 56.0% in layer 3, and 55.5% in layer 5. These values indicate on one hand a robust integration of the glass fibers within the composite matrix, primarily where the rich polypropylene (PP) zone is located. On the other hand, the definition of the fibre bundle contours in layer 1 are relatively complex due to the presence of loose fibers which complicates the definition of the contour of the fiber bundle at high precision. In contrast, the reference plate manufactured by the film stacking technique exhibited lower area fractions: 44.0% in layer 1, 43.7% in layer 3, and 42.1% in layer 5. The observed discrepancies in fiber area fractions are indicative of the total area range of the fiber bundles which are highly compacted in the case of simplified CRTM method while they remain undeformed in the case of the film stacking method. This pattern suggests either an inadequate impregnation or mechanical damages during polishing, particularly in the outer layers. However, according to the sample preparation procedure which is implicating two-step polishing and robing, mechanical damage of the surface is probable during the first polishing step but it is less significant during the second polishing step after application of the second robing which seals the surface damage.

Furthermore, the quantification of the area of glass fibers relies on SEM observations based on the BED-C mode which is not sensitive to fiber breakage as it relies on detecting the chemical composition of the surface. Indeed, based on PLM, a damaged single fiber appears as a jagged geometry but it appears as less fractured based on SEM observations (see Fig.5). Based on such observations the hypothesis of inadequate impregnation seems more coherent.

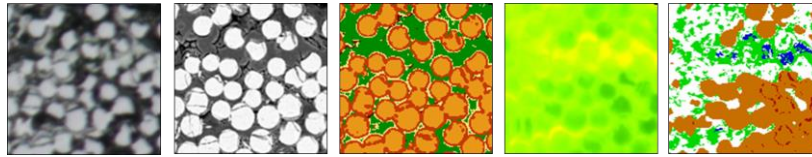


Figure 5. Micro-scale observations of a localized zone in a 0° oriented bundles

3.2 Porosity Analysis

The analysis of porosity levels across different layers of the composite plates provides insight into the structural integrity and potential defects arising from the manufacturing process. In the film stacking sample, which was manufactured at a 0% compaction ratio, the porosity within the fiber bundles of layer 3 was notably low, averaging at 0.02%. This minimal porosity suggests effective fiber impregnation due to the presence of the polymeric matrix above and below the UD plies. However, the porosity levels in the film stacking sample were higher across the layer 1 measuring 8.7% and 8.2% in layer 5 respectively. These higher levels compared to layer 3 potentially indicate the presence of surface defects such as scratches or damages incurred by the sample preparation operations. Further, based on the FM image of this sample, it is noticed that there was an excessive removal of the second robing layer which exposed again the partially impregnated zones and thus caused a higher damage level of the surface due to the destructive nature of polishing (see Fig.3). More detailed analysis within the simplified CRTM sample showed that porosity levels were dependent on the layer position relative to the PP location, with layer 1 exhibiting 25.2% (± 5.9), layer 3 showing 14.4% (± 7.4) and layer 5 indicating 1.7% (± 0.5).

3.3 Comparative Porosity and Area Fraction Analysis

The comparative analysis between layers further substantiates the influence of manufacturing techniques on the structural characteristics of the composite plates. For instance, the lowest glass fiber area fractions and highest porosity levels in the outermost layers of the simplified CRTM sample underscore the challenges in achieving uniform material distribution and integrity throughout the composite structure. Additionally, the progressive decrease in PP area fractions from 42.8% (± 1.5) in layer 5 to 25.7% (± 6.8) in layer 1 during the simplified CRTM process suggests a transverse flow of the polymer, which could be affecting the overall compaction and consistency of the material across the plate.

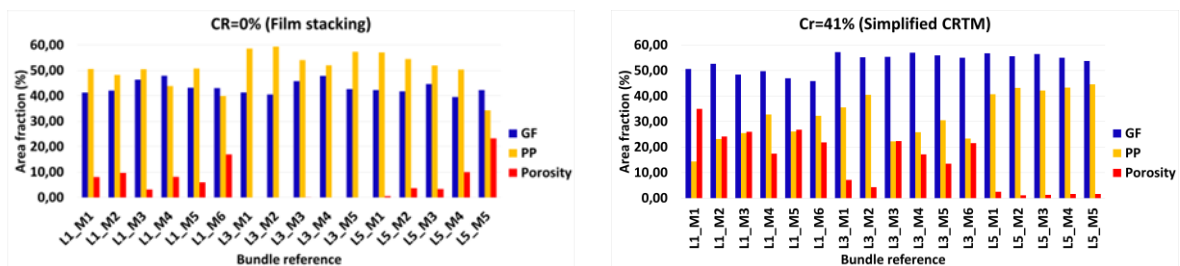


Figure 6. Area fraction distribution of the identified bundles from the samples

4. Conclusions

This study provides valuable insights into the structural quality and efficacy of thermoplastic matrix composites manufactured via simplified CRTM compared to traditional film stacking methods. It highlights CRTM's transverse flow during fiber fiber impregnation quality. A critical aspect of this research is the utilization of multi-modal imaging techniques, which have shown considerable potential in identifying and quantifying these variabilities in porosity and fiber distribution in partially impregnated composite materials. These imaging techniques have proven crucial for a detailed assessment of composite microstructures, offering a deeper understanding of the material

characterization that standard single-mode imaging might miss. Future perspectives involve leveraging the capabilities of multi-modal imaging to refine and optimize the CRTM process further. By integrating advanced imaging data into the manufacturing protocol, it is possible to achieve greater control over the impregnation process, reducing defects and enhancing the uniformity and quality of the final composite products. This approach will not only improve the structural integrity of composites but also expand the practical applications of CRTM in industries requiring high-performance materials. Continued advancements in imaging technology and its integration into CRTM processes hold the promise of pioneering developments in composite manufacturing.

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