MANUFACTURING OF CARBON/POLYAMIDE BEAM BY VACUUM ASSISTED PULTRUSION

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Abstract
The objective of this study is to test the effects of different parameters in minimizing the void content of unidirectional carbon fibre reinforced polyamide rods made with pultrusion. A custom pultrusion apparatus was designed and instrumented. Thirty thermocouples allow controlling the temperature of each section of the pultrusion apparatus. Two load cells measure the pulling force and an encoder record the pulling speed. The pultrusion apparatus has a vacuum module between two impregnation dies. The vacuum pressure is applied to eliminate entrapped air in the composite. Commingled yarn of carbon fibres and bio-based polyamide fibres were used. Rods of 4.78mm in diameter were produced with different parameters including the preheating method, the preheating temperature, the impregnation dies temperatures and the pressure level in the vacuum die. Samples were taken from the manufactured rods, they were polished and observed with a microscope. Image processing indicates that a contact preheater made of heating pins produced rods with 3.8% of volume void content on average while a contactless preheater produced rods with 7.4% of volume void content. In addition, a vacuum pressure in the vacuum module helped reduce the void content by 3.2% on specimen manufactured without a contact preheater.

1. Introduction

Thermoplastic composites present interesting properties when compared to thermoset composites. They have improved impact and chemical resistance, they also allow post forming and welding [1]. Pultrusion of thermoplastic matrix composite has been studied since the 1990's [2,3]. Yet, 25 years later, thermoplastic pultrusion is still not widely used in the industry. The high melt viscosity of the thermoplastic resins makes the impregnation of high fibre content composite a significant challenge [1,4,5]. Precursors with lower impregnation distance like pre-consolidated tapes, commingled yarns and towpregs helps improve the impregnation of composite [1].

Figure 1 presents a common pultrusion apparatus. The traction system feeds the reinforcement fibres into the preheating module where it is usually heated up to a temperature near its melting point (Tm) [6]. Preheating the fibres reduces the time needed to melt the polymer in the pultrusion die [3]. Bechtold et al. [7] studied a more efficient way to preheat the fibres. They have shown that a contact preheater made of heating pins in contact with the precursor produced a better impregnation.

After being preheated, precursors enter the pultrusion die. It is usually made of a tapered region followed by a straight region. In the pultrusion die the thermoplastic melts and coalesce around dry fiber bundles. The excess of polymer flows backward in the dies as the cross section of the tapered region decreases. The head loss of this backward flow is generating a pressure that allows fibre bundle impregnation [8]. Air contained into the fibre bundles can only exit along the fibres fed into the pultrusion dies. These air

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evacuation channels can be blocked by fiber misalignment or uneven resin impregnation. This remaining air can act against the impregnation pressure and prevent bundle impregnation [8]. After exiting the pultrusion die, the composite is fed into the cooling die where the polymer solidifies. It has been observed that a contact cooling die produced better surface finish than air or water cooling techniques [6].

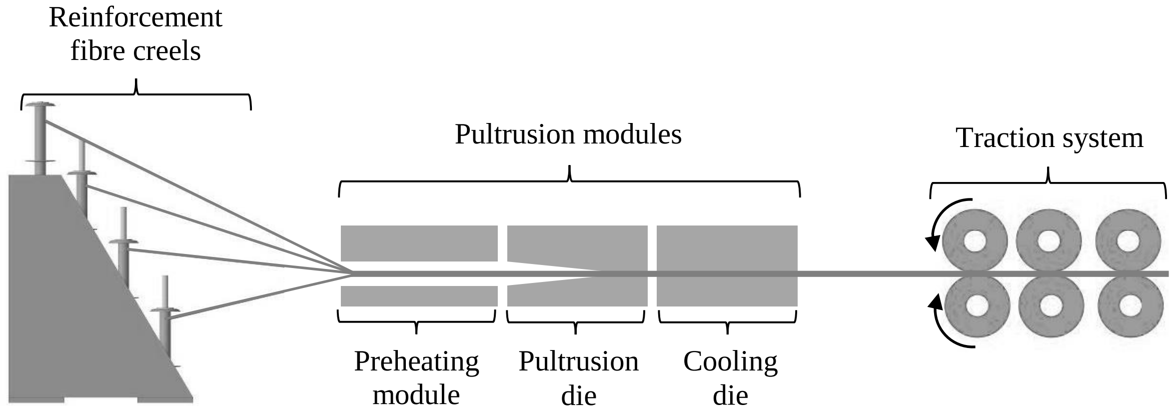

Figure 1. Schematic view of a common thermoplastic pultrusion apparatus.

In order to reduce the void content in the manufactured parts, a vacuum module along with a second impregnation die were added to the pultrusion apparatus. The addition of a vacuum module could reduce the void content in the composite by evacuating entrapped air in fibre bundles before they impregnate in the second pultrusion die. The goal of the study is to manufacture carbon fibre/polyamide rods having a diameter of 4.78 mm using pultrusion. The parameters studied are the preheating method (contactless or contact preheater), the preheater's temperature, the impregnation die temperature and the vacuum level in the vacuum die.

2. Materials and Methods

2.1. Materials

The materials used are commingled yarns of carbon fibres and polyamide fibres (Dualon, Karijene Inc., Japan). The properties of the yarns are presented in Table 1 while Table 2 present the properties of the polyamide fibres (Lexter, Mitsubishi Gas Chemical Inc., Japan).

Table 1. Commingled yarns properties

Property	Manufacturer datasheet values
Fibre volume content (%)	49.5
Fineness [12k, 6k] (tex)	[1300, 650]
Density (g/cm^3)	1.47
Tensile strength (MPa)	1723
Elastic modulus (GPa)	104

Table 2. Polyamide fibres properties

Property	Manufacturer datasheet values
Melting point (°C)	215
Glass-transition temperature (°C)	63
Density (g/cm^3)	1.13
Process temperature (°C)	210-250

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2.1. Pultrusion equipment

The pultrusion apparatus used is made of a creel, pultrusion modules and a pulling system. Yarn precursors are wound onto 23 bobbins and placed onto the creel. Yarns are tensioned by adjusting the friction between the bobbins and the creel. This tension keeps the yarns straight before they enter the pultrusion modules. The yarns pass through six different pultrusion modules made of P20 tool steel and depicted in Figure 2. The modules are mounted on two rails, allowing removing and reordering the different modules as needed. The contactless preheater is a 300 mm long open chamber that is heated by two 725 W finned strip heaters. Note that two thermocouples are used in this module, one is between the two heaters and is used as the control thermocouple. The other one is curved down towards the carbon fibres to acquire the temperature of the air close to the fibers. The contact preheater consists of three 400 W heated cylinders of a diameter of 19 mm. The precursor yarns go under the first pin, over the second and under the third pin of the contact preheater to achieve a good contact between the fibres and the pins. The pultrusion die 1 and 2 both have a cylindrical shaped cavity. Both dies have a tapered zone of 76 mm in length with an angle of 5°. This zone is followed by a 20 mm long straight zone. Pultrusion die 1 has a final diameter of 5 mm while pultrusion die 2 has a final diameter of 4.78 mm. This cross section difference of 9.5 % allows overfilling the second pultrusion die with resin. The vacuum module is positioned between the two pultrusion die. It consists of a chamber where a vacuum can be maintained. The backflow in the tapered sections of the two pultrusion die seals the rod opening making the chamber airtight. The other potential leaks pathways are sealed with sealant tape. The yarns are pulled through the pultrusion modules using the traction system. It consists of 3 pairs of opposing rollers between which the composite rod is squeezed. The superior rollers are actuated by an electric motor; the inferior ones are free wheels.

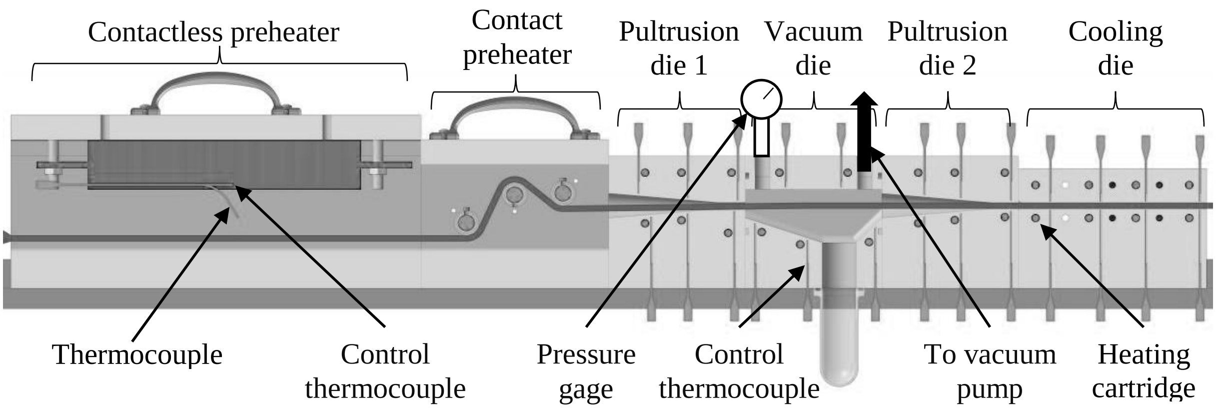

Figure 2. Schematic view of a transversal cut of the pultrusion modules.

The temperature control of the system is achieved by 30 thermocouples inserted into blind holes that are 2 mm away from the interior surface of the different pultrusion dies. A rotary encoder monitors the pultrusion speed. Two loadcells are also placed between the traction system and the pultrusion apparatus to measure the pulling force. These sensors are connected to a computer through the data acquisition module (USB2416, Measurement Computing). A LabVIEW interface monitor and control all sensors and heating cartridges.

2.3. Pultrusion experiments

Six different sets of parameters were tested. The varied parameters were contactless preheater's temperature, contact preheater configuration, pultrusion die temperatures and level of vacuum in the vacuum die. The nominal fibre volume content of 57% was obtained with 23 yarns of 12k commingled

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fibres and one yarn of 6k commingled fibres. The nominal pulling speed was 50mm/min for all the experiments. When no cooling die was used, air streams were used to cool the pultruded rod at pultrusion die 2 exit. Table 3 summarizes the temperature used for the experiments performed. Pultrusion die 1, vacuum die and pultrusion die 2, all had the same set points during the experiments. They are reported in the impregnation system column. The specimens named with a “C” were manufactured with a contact preheater. Specimens named with an “L” were manufactured without contact preheater and without cooling die. The “V” is added to specimens pultruded under vacuum.

Table 3. Temperature set points and vacuum state used for the realised experiments.

Specimen names	Contactless preheater (°C)	Contact preheater (°C)	Impregnation system (°C)	Cooling die (°C)	Vacuum
C1	240	215	250	185	Off
C2	200	200	250	185	Off
C3	200	205	275	185	Off
C4V	230	225	275	185	On
L1	230	-	270	-	Off
L2V	230	-	270	-	On

2.4. Characterization method

The samples generated were polished and observed under a microscope (Metallovert, Leitz). Pictures at a magnification of 200 X mapped the whole section. The cross-section images were analysed by image processing (ImageJ, open source) [9]. The fibre volume content (V_f) and the void content (V_v) were then obtained by selecting gray level threshold above and below which the pixels are considered as part of a fibre or a void. The area ratio of the selected pixels corresponds to void and fibre contents. Three samples were analysed for every parameter sets except for specimen C3 and L1 where only two samples were analysed.

3. Results and Discussion

3.1. Temperature inside the composite

The heat transfer between the pultrusion dies and the pultruded part was characterized by inserting a thermocouple into the composite during the manufacturing of specimen L2V. Figure 3 presents a graph of the temperatures measured by this thermocouple and the position of the thermocouple in the rod.

The temperature inside the precursor fibre bundle reached a peak of 155°C in the contactless preheater. The poor heat transfer inside the fibre bundle caused a difference of 75°C below the set point. The temperature inside the fibre bundle decreased by 7°C after its maximum temperature was reached. The temperature loss is attributed to a small gap between the preheater’s cover and the pultrusion die 1, which allowed warm air to exit the preheater. The temperature at which the composite enters in the pultrusion die is important because a colder polymer will have a lower viscosity and this will reduce the impregnation of the fibre bed.

The precursor’s temperature reached the die temperature around 60 mm after the entrance of the die in both die cases. This delay is attributed to the fact that the precursors are not in good contact with the tapered die walls. During the manufacturing of specimen L2V, the temperature set point for the impregnation system was 270°C. It was observed that the temperature inside the rod reached a peak of

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284°C in both pultrusion dies. This temperature overshoot is attributed to an improper insertion of the thermocouple in their blind holes.

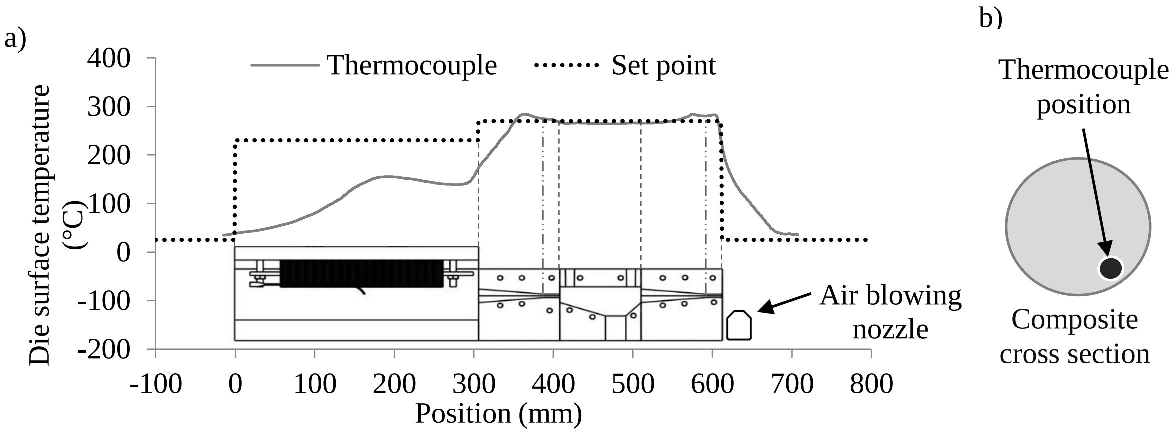

Figure 3. a) The temperature inside the composite in the pultrusion apparatus. b) The approximate position of the thermocouple in the rod.

3.2. Constituent content and pulling force

Table 4 summarizes the measurements of the pulling force, the absolute pressure in the vacuum die, the fractional fibre volume content ratio (V_f) and the fractional void volume content ratio (V_v) for each experiment. The pulling force and absolute pressure were averaged for the residence time of the specimen in the pultrusion apparatus. The V_f and V_v were averaged for the three samples taken from each parameter set, excepted for C3 and L1 where only two samples were averaged. The values are presented along with their standard deviation.

Table 4. Pulling force, absolute pressure in the vacuum die, fibre volume content and void volume content obtained for each specimen.

Specimen names	Pulling force (N)	Absolute pressure (kPa)	V_f (%)	V_v (%)
C1	444	Patm	38±6	2.7±0.5
C2	443	Patm	35±4	4.6±1.1
C3	287	Patm	45±2	3.4±0.5
C4V	416	13	38±4	4.4±1.2
L1	270	Patm	35±7	7.4±0.7
L2V	240	20	36±3	4.2±1.1

The pulling force is similar for the C1, C2, and C4V specimens. Specimen C3 presents a low pulling force. During this experiment, it was frequent to see the rod stick to the cooling die walls and stop being pulled. To solve this, the cooling die was slightly opened to release the friction, therefore lowering the pulling force. L1 and L2V present the lowest pulling force observed. This is explained by the absence of a cooling die. It has been observed that the cooling die creates an increase of pulling force.

The measured V_f present some variations between specimen. While the expected V_f was 57%, it is believed that the fibre breakage in the pultrusion die has contributed in lowering the final number of fibres in the manufactured part. Fibre breakage is further discussed in section 3.3. The lowest V_v

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measured is on specimen C1 manufactured with a contact preheater. The highest V_v is observed on specimen L1 which was not manufactured with a contact preheater. The impregnation system's temperature and the contactless preheater temperature did not have an impact on void content. Figure 4 presents the void content of the specimens against various process parameters.

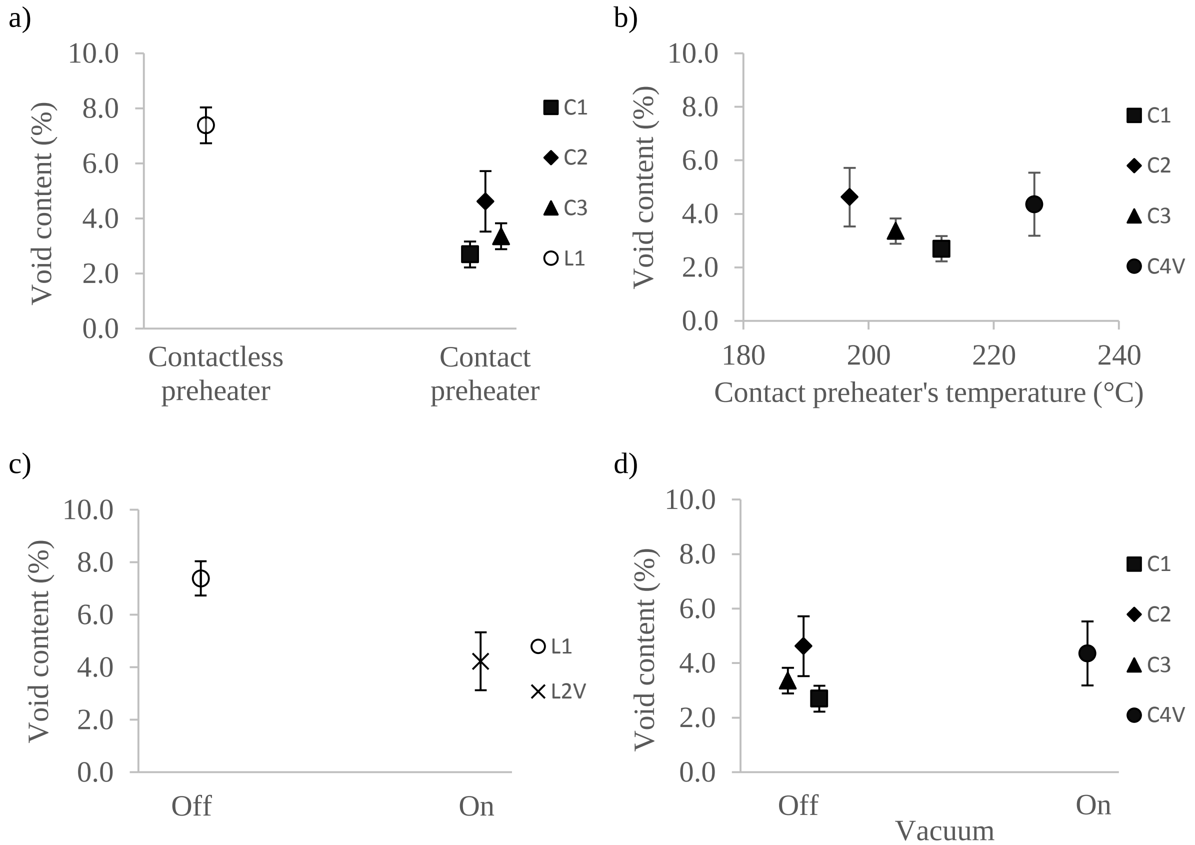

Figure 4. Void content in the specimens compared to different process parameters. a) Void content in specimens manufactured with or without a contact preheater. b) Void content against the contact preheater's average temperature. c) Void content compared to the presence of vacuum when no contact preheater is used. d) Void content compared to the presence of vacuum when a contact preheater is used.

Figure 4a suggests that the usage of a contact preheater allows reducing the void content in the specimens. This is consistent with the results of Bechtold et al. [7]. When comparing the contact preheater's temperature, it can be seen in Figure 4b that a contact preheater's temperature increase tends to decrease the void content. However, specimen C4V has the highest contact preheater's temperature but still presents a void content similar to C2. The optimal contact preheater's temperature might be near Tm. Further experiments will be conducted to verify this hypothesis.

During the manufacturing of specimens C1 and C4V, it was observed that the temperature of at least one of the three contact preheater's pins was over Tm. When passing over that pin the resin melted and flowed through the fibres. The pins also spread the fibres into a flat tape of small thickness allowing a good heat transfer throughout the composite but also reducing the impregnation distance. Therefore, the contact preheater also acted as a pre impregnator when its temperature was above Tm. Observations are concordant with what Miller and Gibson reported on pin impregnation of thermoplastic composites [10]. Unfortunately, because the manufactured part is of cylindrical shape, the flat pre impregnated tape was

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too wide and had to fold over itself to enter in the cylindrical die. That created misaligned fibres and voids between fibres bundles near those misaligned fibers.

Figure 5 shows representative areas of the pultruded beam cross sections for all experiments. It is observed that the usage of a contact preheater at temperatures close to Tm changed the type of voids present in the composites. Specimen C1, C3, and C4V, manufactured with a contact preheater, present voids between fibre bundles (macro-voids). Specimen L1 and L2V, manufactured without a contact preheater, present voids inside the fibre bundles (micro-voids). Although specimen C2 was manufactured with a contact preheater, the voids present in its section are inside the fibre bundles. Specimen C2 was manufactured with the lowest contact preheater's temperature.

It can be seen from the graph in Figure 4c that the addition of vacuum pressure in the vacuum die improves the impregnation for specimens that are not preheated with a contact preheater. Micrograph of samples L1 and L2V (Figure 5) presents voids within the fibre bundles. On L1, the voids are in clusters whereas in sample L2V the voids are many small voids in the same region. This observation supports the idea that the vacuum obtained between the two pultrusion dies helped evacuate some of the entrapped air in the fibre bundles. The air leaving the bundle in the vacuum die would reduce the volume of the void.

Figure 4d shows that vacuum pressure does not improve the impregnation of specimens preheated by a contact preheater. The preheater configuration used in this study created fiber misalignment. The voids in composites manufactured with a contact preheater are attributed to misaligned fibers.

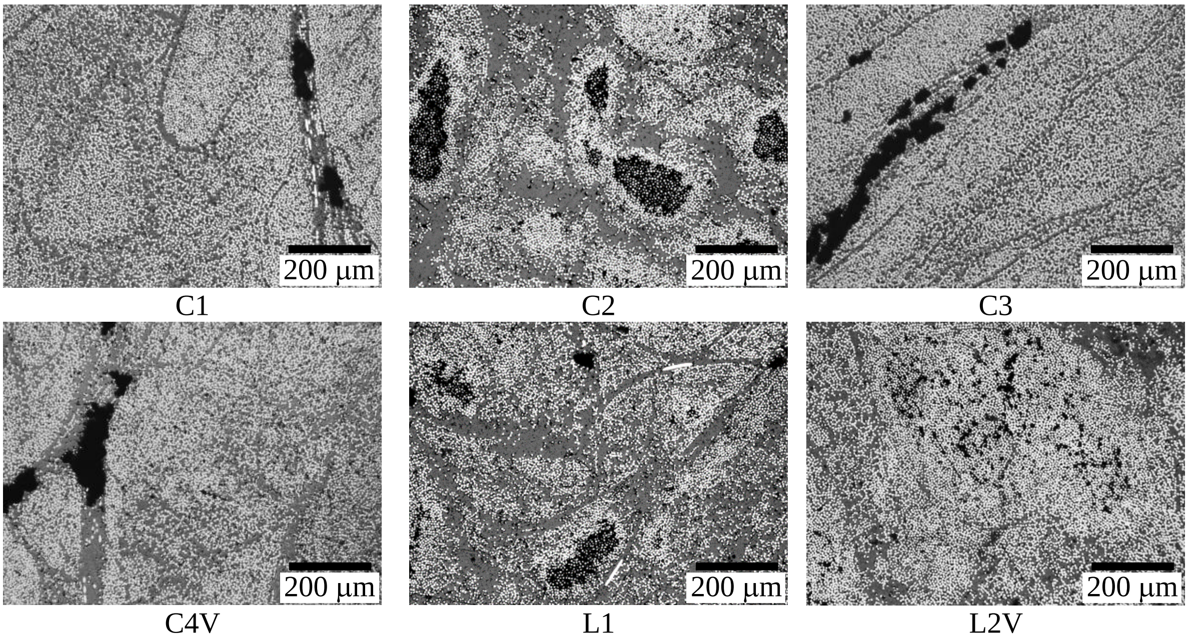

Figure 5. Micrographs of manufactured specimens. The photos were taken in zones showing typical void type for the observed sample. The fibres appear in white, the matrix is in gray and the black areas are voids. The different experiments correspond to the following: specimen C1 (contact preheater), specimen C2 (low temperature contact preheater) specimen C3 (contactless preheater), specimen C4V (contact preheater and vacuum), specimen L1 (contactless preheater) and specimen L2V (contactless preheater and vacuum).

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3.3. Fibre breakage

A recurrent problem faced was the breakage of fibres in the impregnation dies. Every experiment conducted were interrupted after the pultrusion of several meters. It was observed that the backflow on both pultrusion dies was constituted of excess resin as well as broken fibres. Broken fibres in the melted matrix created more friction and broke more fibres. After a while, the manufactured rod would not have enough material to fill the die cross section. This problem prevented us from making mechanical testing of the samples. The fibre volume fraction would have had more impact on the results than the other process parameters. This problem will be further assessed in future work.

4. Conclusion

An innovative system with two impregnation dies and a vacuum chamber was designed and manufactured. Carbon fibre and Polyamide composites rods with fiber volume content of 40% were manufactured using pultrusion. The impact of the preheating method was studied and it was observed that a contact preheater reduced the void content of manufactured composites. Void contents went from 7.4% to 3.8% on average. Samples manufactured without a contact preheater presented a decrease in void content when vacuum was applied in the vacuum die. It seems that a vacuumed environment helps reduce the size of micro voids but has no effect on reducing macro voids. Further work will need to be performed to confirm the impact of the vacuum pressure in the vacuum die on the void content of the manufactured composites. The fibre breakage will also be assessed and solved in order to analyse the mechanical properties of the future manufactured rods.

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