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Thermoplastic Matrix Composites from Towpregs

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1. Introduction

In recent years, continuous fibre reinforced thermoplastic matrix composites have been successfully employed in the aircraft, military and aerospace industries due to the excellent properties (Brandt et al. 1993 & Nunes et al 2005a). In these and many other commercial engineering applications, they can replace other materials, such as thermosetting matrix composites. However, the high cost of the impregnation of continuous fibre thermoplastic composites, arising from the melting of the polymer or the use of solvents, still restricts their use in commercial applications. Hence, cost reduction largely depends on developing more efficient methods for impregnating fibres with high-viscosity thermoplastics and for processing final composite parts.

This chapter summarizes the development of new technologies to fabricate long and continuous fibre reinforced composite structures from thermoplastic matrix semi-products (towpregs and PCT – pre-consolidated tapes) for commercial and highly demanding markets.

The production of continuous fibre reinforced thermoplastic matrix towpregs and PCT's was done using a recently developed coating line (Nunes et al. 2008, 2010 & Silva, R.F. et al. 2008).

Using this prototype equipment, it was possible to produce glass fibre polypropylene (PP) and polyvinylchloride (PVC) towpregs for commercial markets and towpregs from carbon fibres and Primospire[®], an amorphous highly aromatic material developed by Solvay Advanced Polymers, for application in advanced markets (Nunes et al. 2005, 2009 & Silva, J. F. et al. 2010).

To process these thermoplastic pre-pregs into composite structures, conventional thermosetting equipments were adapted to fabricate thermoplastic matrix composites. Filament winding, pultrusion and hot compression moulding were the studied technologies. The mechanical properties determined on the final composites were compared with the theoretical predictions and have shown to be acceptable for the targeted markets.

As applications, filament wound pressure vessels prototypes for gas and incompressible fluids were produced from towpregs and submitted to internal pressure burst tests [Silva, J. F. et al. 2008 & Velosa et al. 2009]. These prototypes have accomplished all requirements of the applicable European standards.

2. Experimental

2.1 Powder coating equipment

The prototype powder coating equipment used to produce glass and carbon fibre reinforced towpregs is schematically depicted in Figure 1. It consists of six main parts: a wind-off system, a fibres spreader unit, a heating section, a coating section, a consolidation unit and a wind-up section. In order to produce the towpregs, the reinforcing fibres are wound-off from their tows and pulled through a pneumatic spreader. After, they are heated in a convection oven and so made to pass into a polymer powder vibrating bath to be coated. A gravity system keeps constant the amount of polymer powder. The oven of the consolidation unit allows softening the polymer powder, promoting its adhesion to the fibre surface. Finally, the thermoplastic matrix towpreg is cooled down and wound-up on the final spool.

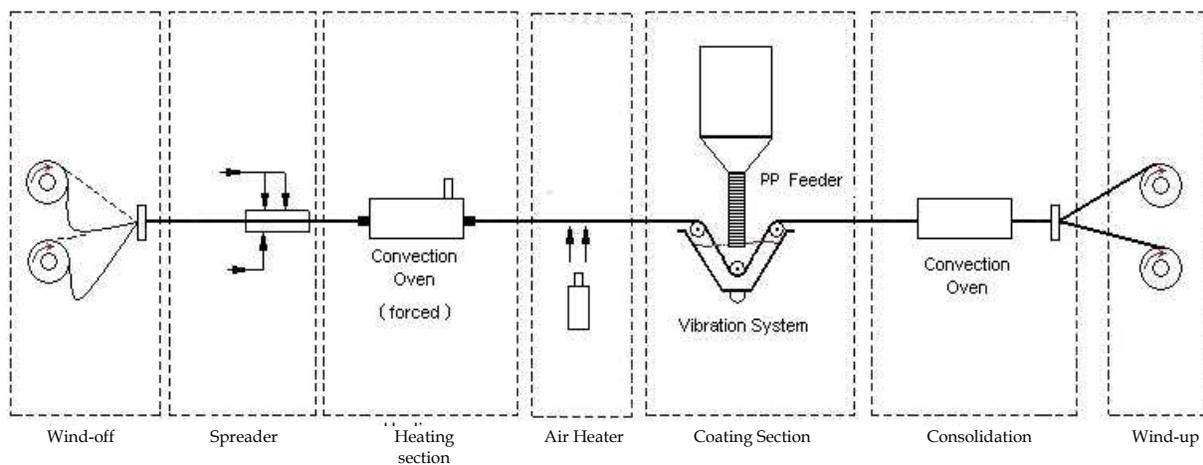


Fig. 1. Schematic diagram of the powder-coating line set-up

The photograph depicted in Figure 2 shows a general overview of the developed powder coating equipment.

2.2 Raw materials

2400 Tex type E glass fibre rovings, from Owens Corning, polypropylene, from ICO Polymers France (Icorene 9184B P), and polyvinyl chloride, supplied by CIRES (PVC - PREVINIL AG 736), powders were used to produce GF/PP and GF/PVC towpregs to be applied in common composite engineering parts. Table 1 summarises the most relevant properties of these materials.

Property	Units	Glass fibres	Polypropylene	PVC
Density	Mg/m ³	2.56	0.91	1.4
Tensile strength	MPa	3500	30	55
Tensile modulus	GPa	76	1.3	3.0
Average powder particle size	µm	-	440	150
Linear roving weight	Tex	2400	-	-

Table 1. Properties of raw materials used in towpregs for common applications



Fig. 2. Powder coating equipment

A new polymer developed by Solvay Advanced Polymers (Primospire® PR 120) and carbon fibre tows from TORAYCA (760 Tex M30SC) were used to produce towpregs for highly demanding markets. Table 2 presents the most relevant properties determined on these raw materials.

Property	Units	Carbon fibres	Primospire®
Density	Mg/m ³	1.73	1.21
Tensile strength	MPa	2833	104.3
Tensile modulus	GPa	200	8.0
Average powder particle size	µm	-	139.4
Linear roving weight	Tex	760	-

Table 2. Properties of the raw materials used in towpregs for advanced applications

This new polymer was also characterised in terms of other relevant properties, such as thermal, rheological and flame and smoke characteristics. The glass transition temperature (T_g) of the polymer was determined, by using a Diamond Pyris Perkin Elmer DSC, as 158.0 which is the value supplied by its manufacturer (158.0 °C).

The rheological characteristics of the Primospire® PR 120 were determined in oscillatory regimen using a parallel plate rheometer TA Instruments Weissenberg. The dependence of the elastic and dissipative moduli with the oscillatory frequency was obtained at 3 different

temperatures: 320 °C, 330 °C and 340 °C. Polymer discs with 25 mm of diameter produced by compression moulding were used in these tests. The Cox-Merz rule was used to establish the relation between the dynamic viscosity (function of the angular frequency) and the shear viscosity (function of the shear rate).

Figures 3 and 4 show the results obtained for the dependence of the viscosity on shear rate values at different temperatures using linear and logarithm scales, respectively.

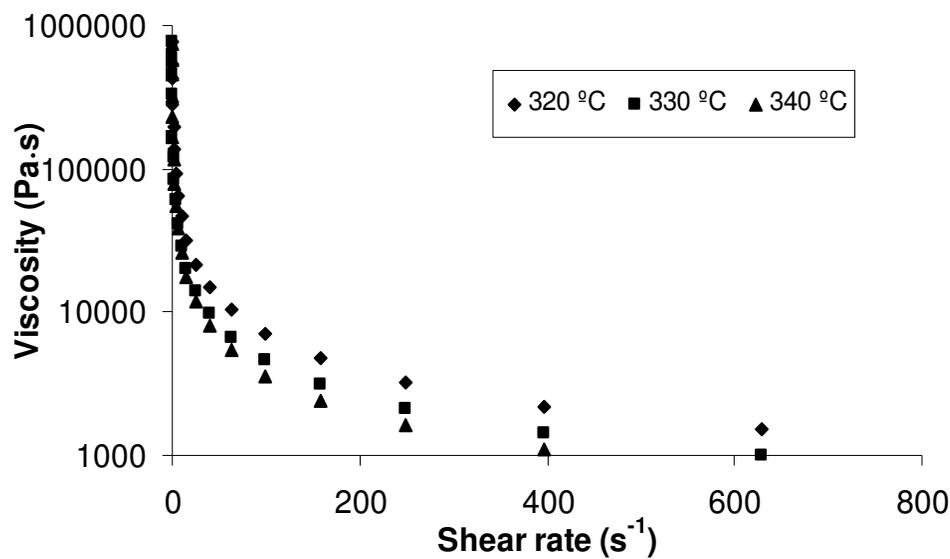


Fig. 3. Dependence of the Primospire® viscosity on shear rate at different temperatures

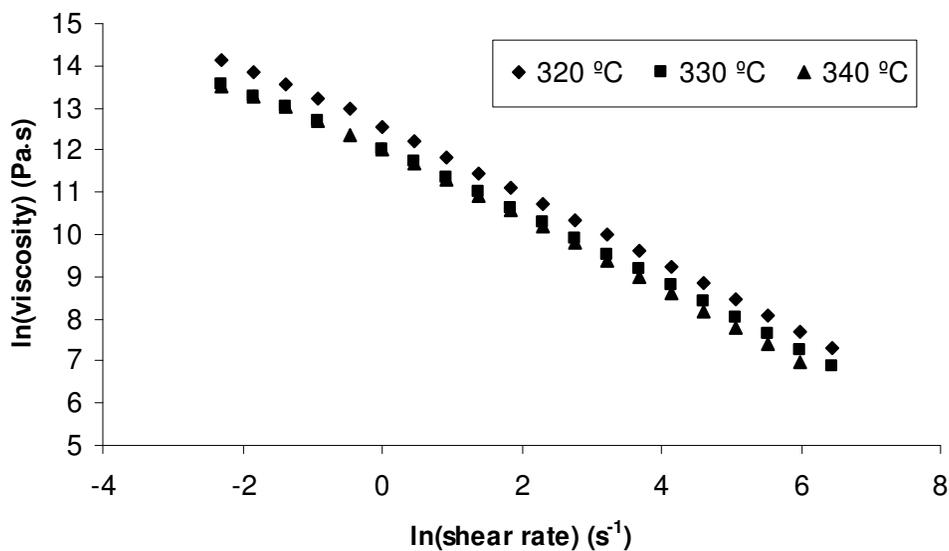


Fig. 4. Dependence of the viscosity on shear rate at different temperatures in a ln-ln scale

To obtain the flame and smoke characteristics of the Primospire® tests were carried out, according to ASTM E 1354:2004, in a cone calorimeter using a constant external heat flux of 50 kW/m² and an exhaust duct flow rate 0.025 m³/s. Heated compression moulded square plates with approximately 100 x 100 x 4 (mm) and 50g of weight were horizontally tested. Table 3 summarises the results obtained in the cone calorimeter tests.

Property	Units	Value		
Time to ignition	s	163		
Total heat	kJ	574		
Mass loss	g	21.8		
	%	44.1		
		Peak	Time (s)	Average
Heat release rate	kW/m ²	119.4	265	34.7
Effective heat of combustion	MJ/kg	45	370	26.3
Specific extinction area	m ² /kg	242.6	190	102.5
Carbon monoxide	kg/kg	0.6438	1740	0.2722
Carbon dioxide	kg/kg	1.54	310	0.78

Table 3. Cone calorimeter results (ASTM E 1354)

Table 4 compares the obtained Primospire PR120 fire characteristics with those of other current polymers and composites. As can be seen, the study material seems to exhibit excellent fire characteristics.

	MATERIAL	Time to ignition (s)	Heat release rate peak (kW/m ²)	Effective Heat of Combustion (MJ/kg)	Total heat release (MJ/m ²)	Residue (%)
Thermoplastics	Primospire PR 120	163	119.4	26.3	57.4	55.9
	HDPE ^{a)}	71	2021	43.8	-	-
	PC ^{a)}	125	725	19.5	-	-
	PA ^{a)}	86	1489	29.7	-	-
	POM ^{a)}	37	571	13.6	-	-
Thermosettings ^{b)}	PF (phenol/formaldehyde= 1:2)	102	174	-	23	76.6
	PF (phenol/formaldehyde= 2:1)	59	79	-	5	95.1
Composites ^{c)}	Graphite/phenolic (1103)	104	177	-	50	72
	Graphite/PPS (1085)	173	94	-	26	84
	Graphite/Epoxy (1093)	94	171	-	-	76
	Graphite/PEEK (1086)	307	14	-	3	98

Notes: a) (Panagiotou 2004); b) (Nyden et al. 1994); c) (Sorathia & Beck 1995)]

Table 4. Fire properties of current polymers and composites

2.3 Optimising the processing conditions of the towpregs

Figure 5 shows the polymer mass fraction of the glass fibre reinforced polypropylene (GF/PP) towpregs by varying the coating line oven temperature at different fibre pull-speeds. The polymer fractions were determined by cutting and weighting 1 m length of the towpreg strips produced in the coating line.

As expected, the polymer mass fraction decreased with increasing fibre pull-speed, maxima polymer depositions being obtained for oven temperatures range between 400 °C and 450 °C.

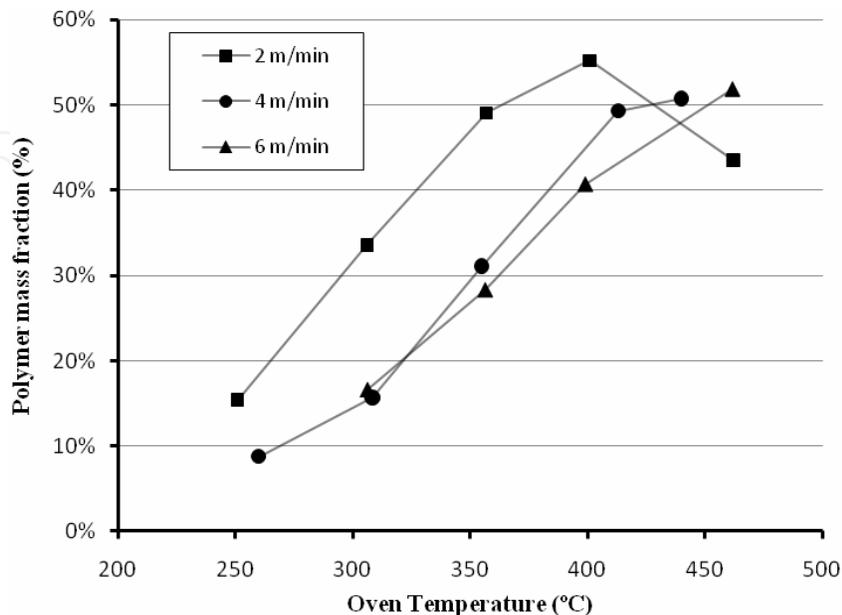


Fig. 5. Variation of the polymer mass fraction with oven temperature and fibre pull-speed

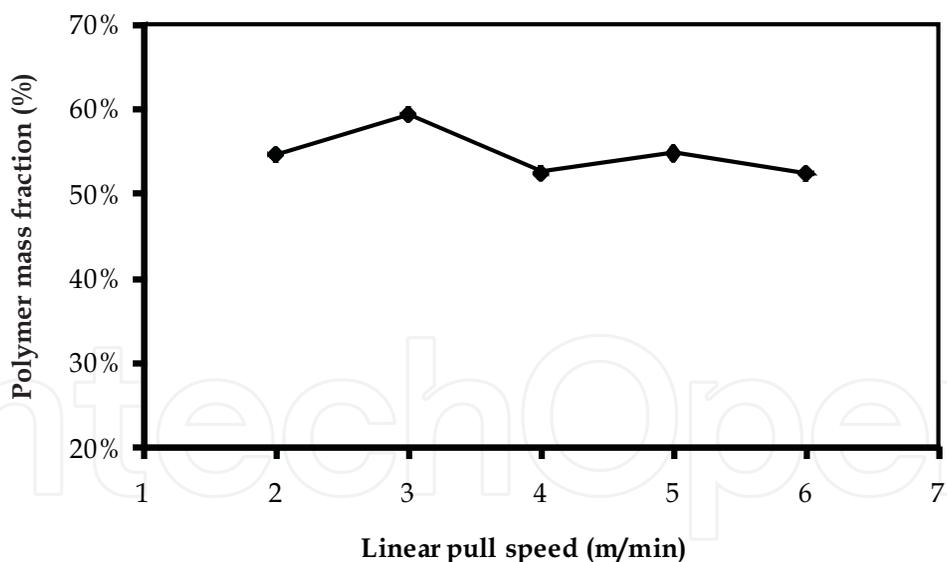


Fig. 6. Influence of production speed on the polymer content of the GF/PVC towpregs

Figure 6 presents the same type of results for the glass fibre reinforced polyvinyl chloride (GF/PVC) towpregs produced in the coating line using oven temperatures in the range between 260 °C and 315 °C. In this case, it was observed that only in such small range gap of temperatures it was possible to produce enough good GF/PVC towpregs. A deep decrease in the amount of polymer was verified when lower oven temperatures were used and considerably polymer degradation (great changes in PVC colour) was observed at higher oven temperatures.

As it may be seen, a good and almost constant level of PVC mass content was obtained by using fibre pull-speeds between 2.0 and 6.0 m/min.

Figures 7 to 9 show the variation of the polymer mass fraction in the Primospire[®]/Carbon towpregs with fibre pull speeds at three constant oven temperatures. It may be concluded that the polymer mass fraction decreases with the fibre pull speed at the lower oven temperature (600 °C). At the higher oven temperatures, the amount of polymer in the towpregs seems to keep an approximately constant value of 40% at all fibre pull speeds.

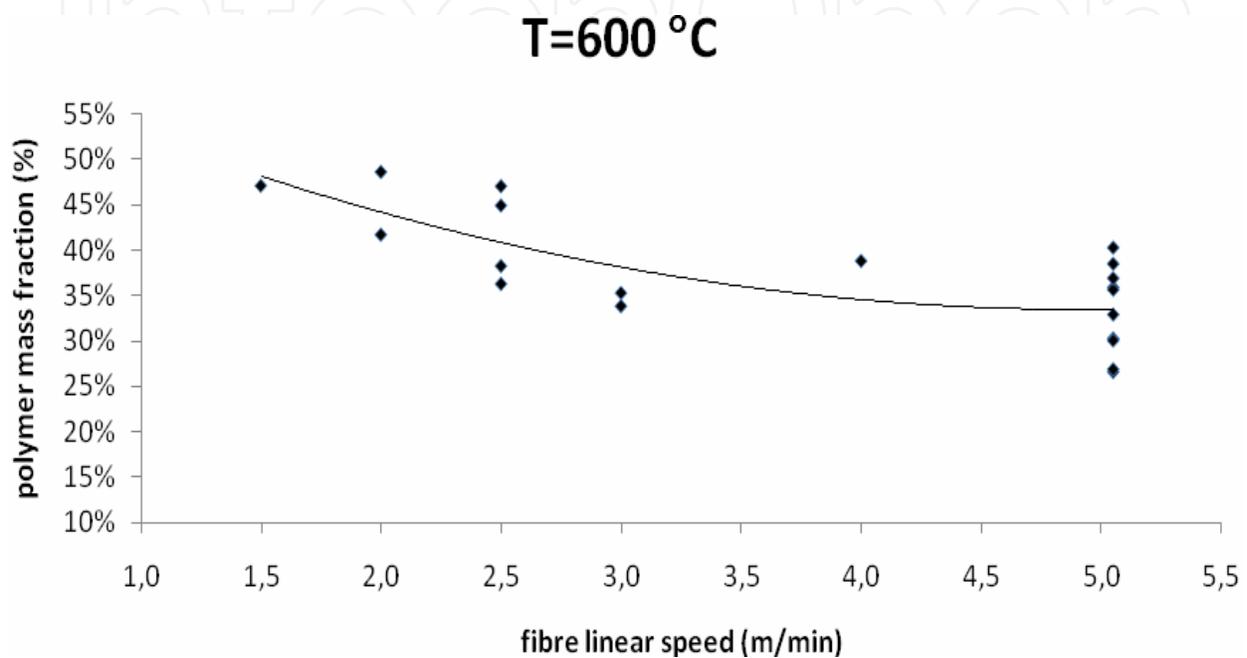


Fig. 7. Polymer mass fraction variation with fibre pull speed for 600 °C oven temperature

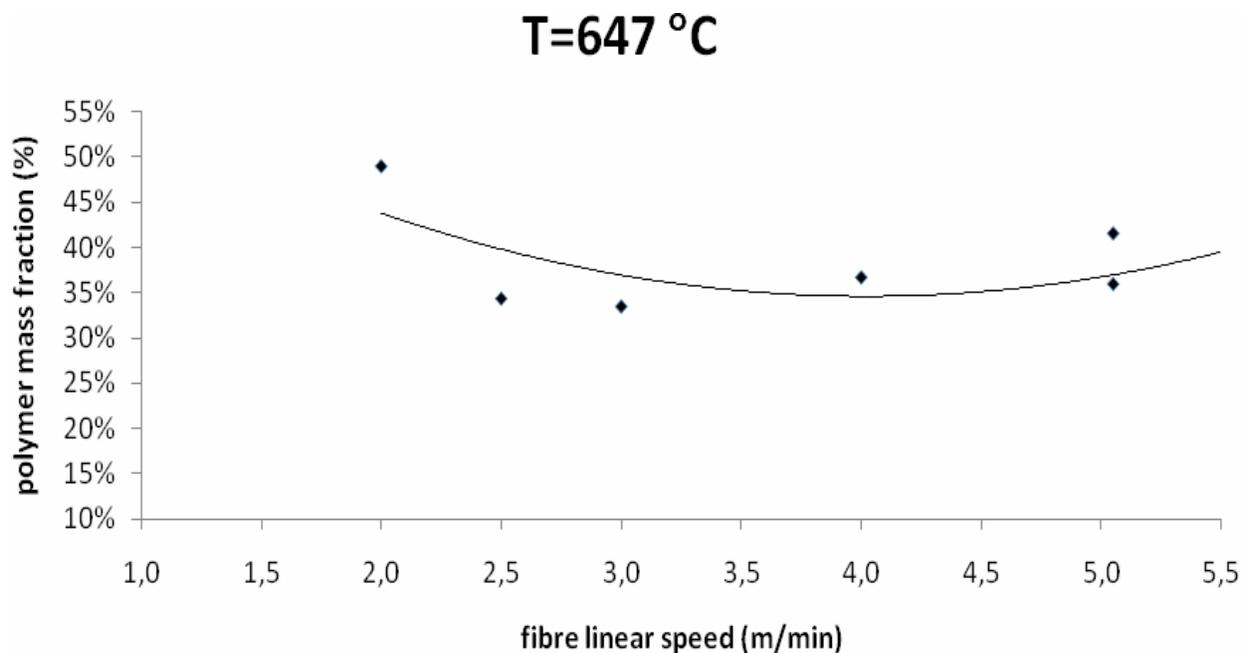


Fig. 8. Polymer mass fraction variation with fibre pull speed for 647 °C oven temperature

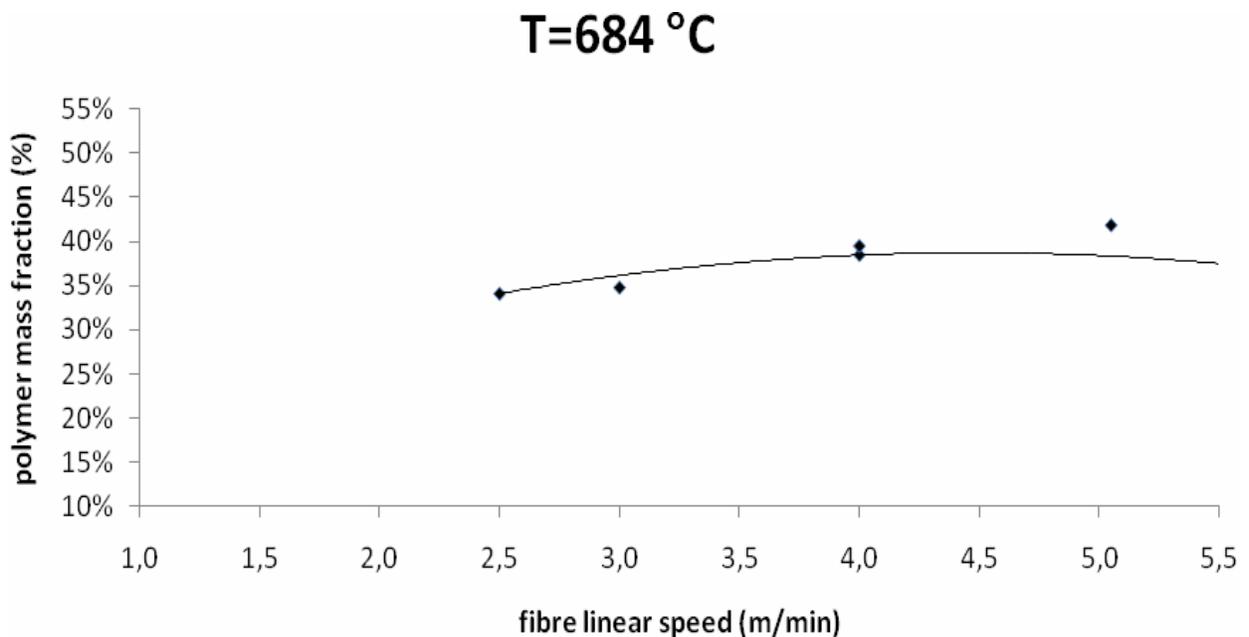


Fig. 9. Polymer mass fraction variation with fibre pull speed for 684 °C oven temperature

2.4 Characterization of towpregs by SEM

Several samples of the GF/PP towpregs were analysed under a Nova NanoSEM 200 scanning electron microscope to evaluate the polymer powder distribution and its adhesion to the fibres. Figure 10 shows a SEM micrograph of a towpreg sample produced in the dry coating line using an oven temperature around 400 °C and a fibre pull-speed of 4 m/min.

As it may be seen in Figure 10, at these optimised coating line operating conditions a good polymer melting and adhesion to glass fibres seems to have been achieved.

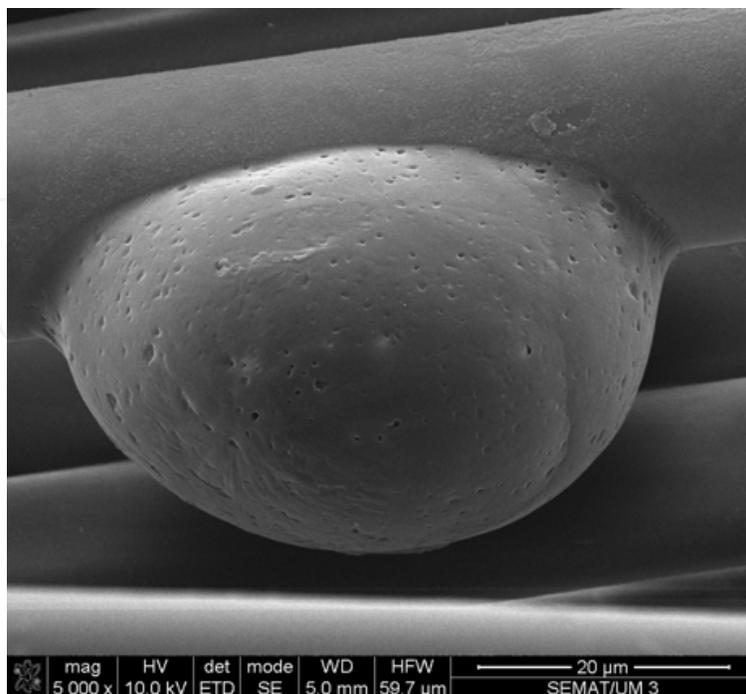


Fig. 10. SEM micrograph of GF/PP towpreg (magnification of 5000×)

A Leica S360 scanning electron microscope was also used to observe the typical aspect of the GF/PVC towpregs. As it is shown in the SEM micrograph depicted in Figure 11, a good adhesion was also obtained between PVC particles and glass filaments in the samples processed at the optimised oven temperatures from 260 °C to 315 °C.

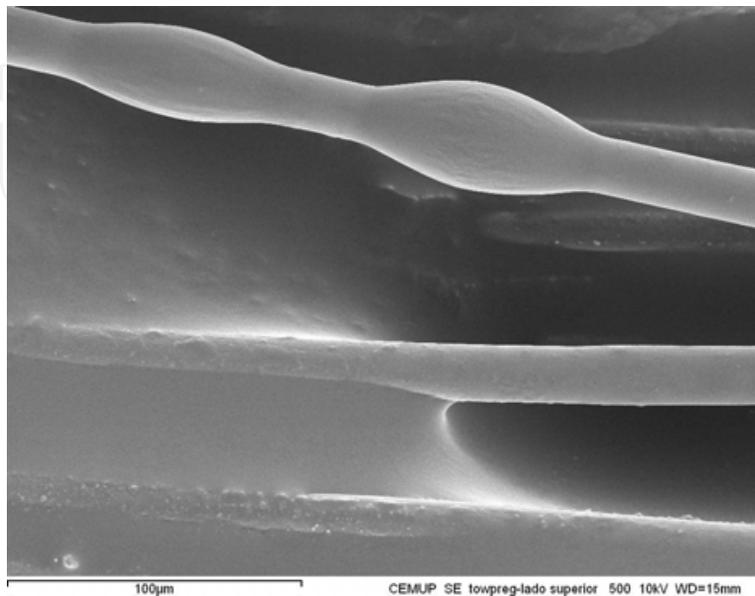


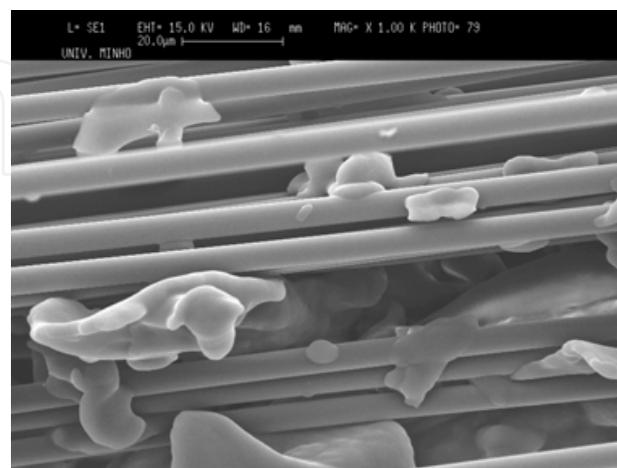
Fig. 11. SEM micrograph of GF/PVC towpreg (magnification of 1000×)

The same Leica S360 scanning electron microscope equipment was used to evaluate the polymer powder distribution and its adhesion to the fibres in several Primospire®/carbon towpreg samples. Figure 12 shows two representative SEM micrographs of samples produced using an optimised 650°C oven temperature in the dry coating equipment.

As can be seen, in the case of this highly demanding market towpregs most of the polymer particles exhibit bigger sizes than the fibre diameter and, even after heating, polymer particles present an irregular shape. It is also possible to observe a enough good degree of adhesion between fibres and polymer powder.



a) Magnification: 100×



b) Magnification: 1000×

Fig. 12. Micrographs of Primospire®/carbon towpreg under SEM

2.5 Composite processing technologies

2.5.1 Compression moulding

SATIM and MOORE hot plate presses with capacity of 400 kN were used to process the produced towpregs into composite plates by compression moulding using a technique described elsewhere (Klett et al. 1992). First, the towpregs were wound over a plate with appropriate dimensions and the resultant pre-form then conveniently placed in the cavity of a heated mould. After that, the press is closed, to obtain the desired pressure during the consolidation time. Then, the mould is cooled down to room temperature and, finally, the laminate composite plates are removed. Table 5 summarizes the compression moulding cycle parameters.

Composite	Temperature (°C)	Pressure (MPa)	Consolidation time (min)
GF/PP	250	20	15
GF/PVC	210	15	15
CF/Primospire®	320	20	30

Table 5. Compression moulding cycle parameters

In the case of CF/Primospire towpregs, plain woven fabrics were also produced from towpreg tows using a manual weaving loom. This pre-preg material has shown to be easier to process by compression moulding than unidirectional pre-forms.

2.5.2 Filament winding

Figure 13 depicts schematically the filament winding system developed to produce GF/PP pipes and plates from towpregs. This system was tested with a laboratory CNC 6 axes conventional PULTREX filament winding machine. The equipment consists on a pre-heating furnace, a hot-air heater and a pneumatic controlled consolidation roll.

Before being wound onto the mandrel, the GF/PP towpregs are guided, at controlled and constant tension, through the pre-heating furnace at the desired temperature. Final consolidation is achieved in the mandrel, at a required pressure, using a consolidation head,

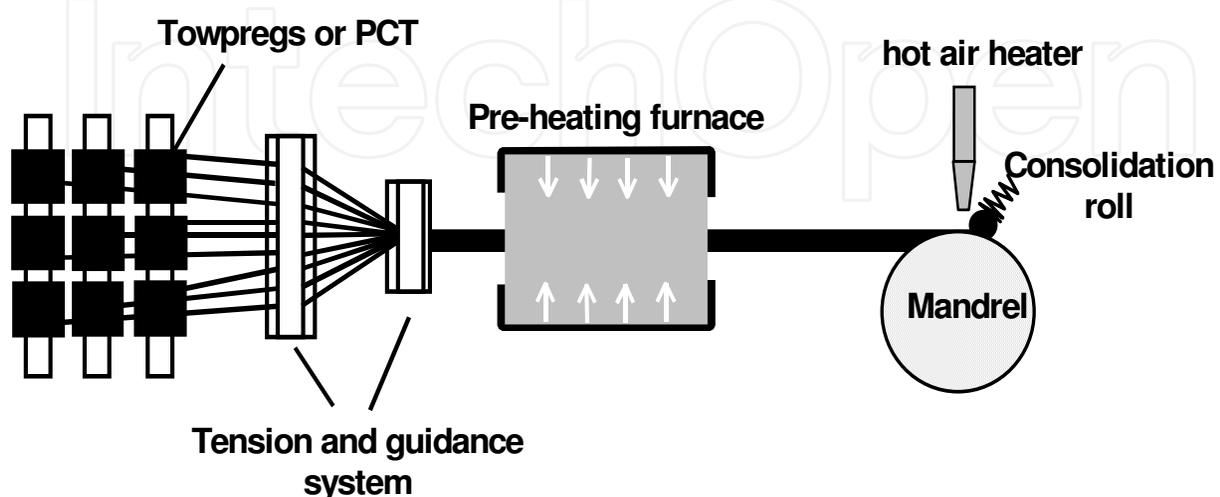


Fig. 13. Schematic representation of the filament winding system

assisted by a hot-air heater. A thermocouple allows the temperature to be adjusted during the consolidation.

GF/PP pipes with dimensions of $\varnothing 80 \times 2$ (mm) were produced using the typical filament winding conditions presented in Table 6.

Variable		Units	Value
Mandrel rotational speed		rpm	5-30
Temperature	Pre-heating	°C	200
	Air heater		300-350
Consolidation force		N	80-100
Tow tension		N	10

Table 6. Typical filament winding parameters

In the case of GF/PVC $\varnothing 80 \times 3$ (mm) filament wound pipes were produced by using a conventional wet fibre impregnation route. A low viscosity vinyl chloride homopolymer past obtained from an emulsion polymerization was used. By using this PVC type, it was only necessary to incorporate a heating system in the conventional filament winding machine eye-feed mechanism to process good quality continuous fibre reinforced pipes.

2.5.3 Pultrusion

A pultrusion head was used mounted on a conventional 60 kN pultrusion line. This head allowed the adaptation of the line, designed for thermoset matrix composites, to the production of continuous profiles made from thermoplastic matrix towpregs. The concept for the pultrusion head, as shown in Figure 14, includes three main parts: i) the pre-heating furnace; ii) the pressurization and consolidation die and, iii) the cooling die.

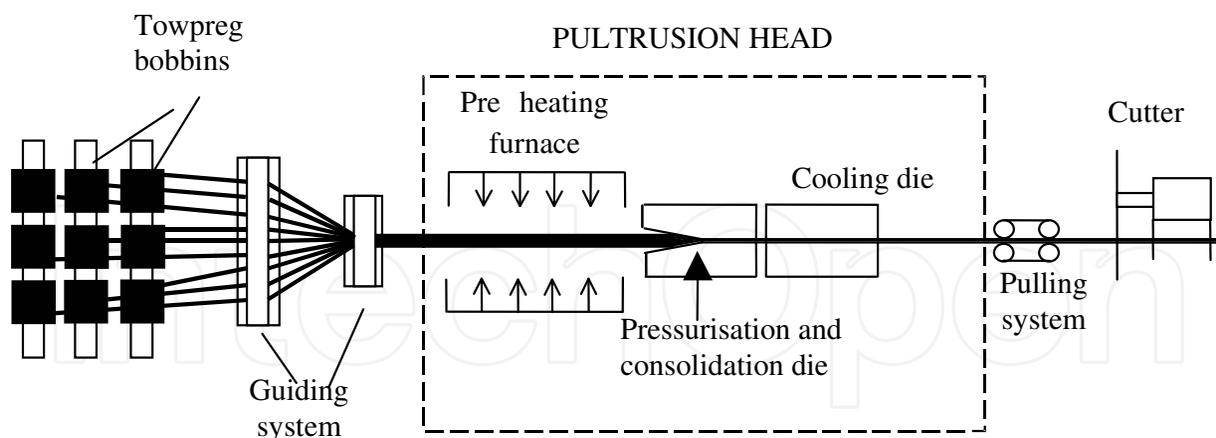


Fig. 14. Schematic diagram of the pultrusion line

The process involves three phases. First, the GF/PP towpregs are guided into the pre-heating furnace. Then, they pass through the first part of the pultrusion head where the consolidation occurs. The consolidated material then enters the cooling die where it cools down to a required temperature. Finally, after leaving the pultrusion head, the profile is cut to specified lengths. Table 7 reports the operating conditions typically used in tests with the pultrusion line.

Variable	Units	Value
Pultrusion pull speed	m/min	0.5-0.8
Pre-heating furnace temperature	°C	200-250
Die temperature	°C	300-320
Cooling die temperature	°C	60

Table 7. Typical pultrusion operating conditions

GF/PP U-shape profiles with $24 \times 4 \text{ mm}^2$ cross-section, 2 mm thick were fabricated, with well-defined forms and smooth surfaces.

2.6 Final composites mechanical properties

The fibre mass fraction, flexural and tensile properties of the continuous fibre reinforced composites fabricated by the different technologies were determined in accordance to ISO 1172, ISO 178 and EN 60, respectively. The split disk test method according to ASTM 2290 was employed to determine the circumferential strength and modulus on the filament winding pipes.

Table 8 summarises the final results obtained for CF/Primospire® composite specimens. The theoretical values presented in this Table were calculated from the raw materials properties by using the rule of mixtures (ROM).

Property		Units	Determined Values	
			Average	Stand. Dev.
Flexural modulus (Unidirectional composite)	Experimental	GPa	30.0	5.0
	Theoretical		103.8	
Flexural modulus (woven fabrics)	Experimental	GPa	26.8	2.2
	Theoretical		53.8	
Flexural strength (Unidirectional composite)	Experimental	MPa	124.3	15.0
	Theoretical		867.0	
Flexural strength (woven fabrics)	Experimental	MPa	160	56
	Theoretical		459.0	
Fibre mass fraction	Experimental	%	59.7	0.3
Fibre volume fraction	Calculated		51	

Table 8. Flexural properties of composites made from CF/Primospire® towpregs

As can be seen, the composites manufactured from the woven fabrics presented mechanical properties in better agreement with the theoretical expected ones than those reinforced with unidirectional fibres. The major causes for the differences found in these mechanical tests between the experimental and theoretical flexural stiffness and strength values have been attributed to a low fibre/matrix adhesion and also to fibre misalignments observed in the composite plates.

Figure 15 and 16 show the results from tensile and flexural tests, respectively, obtained from GF/PP towpregs produced in the coating line with different parameters and processed by compression moulding.

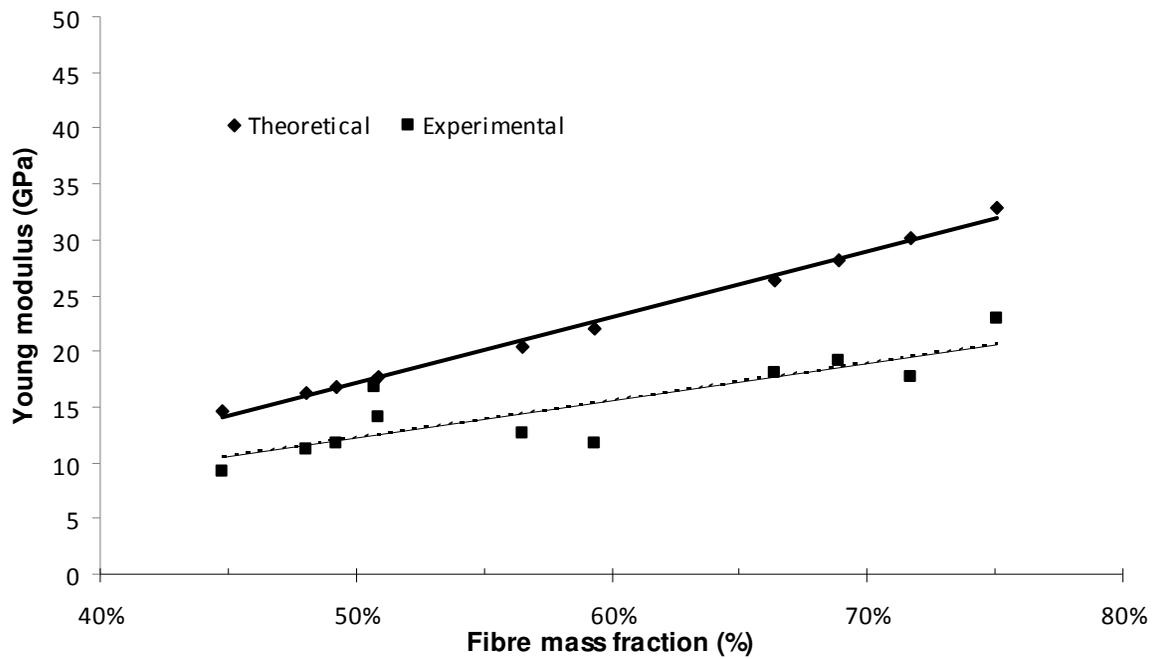


Fig. 15. Tensile test results from compression moulded GF/PP towpregs

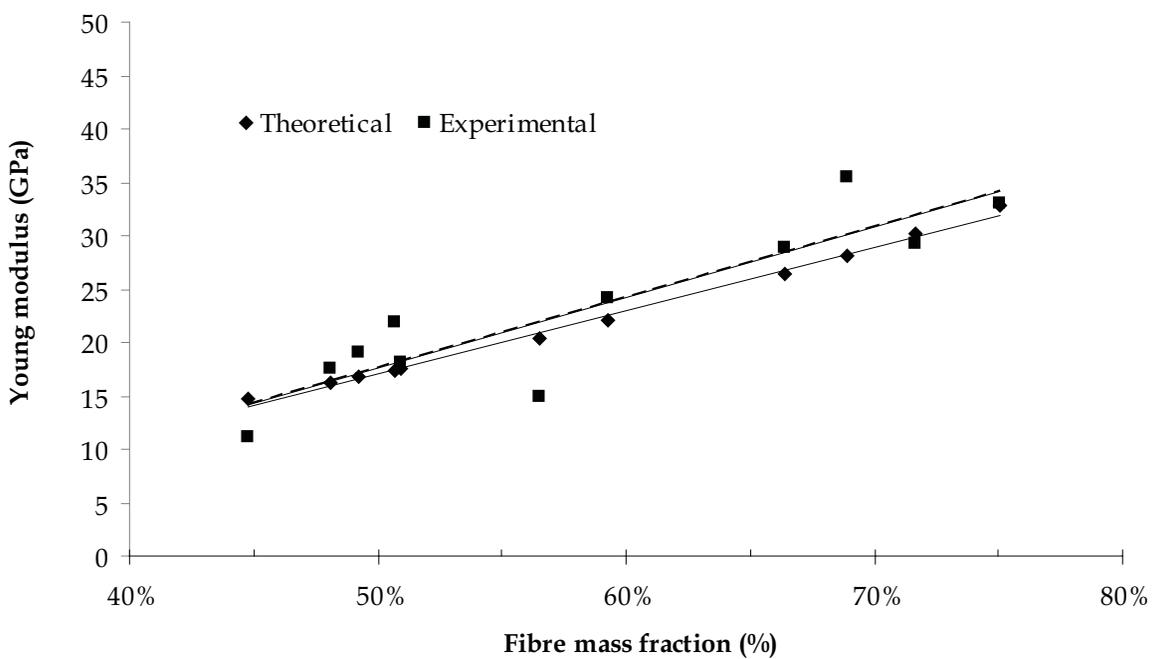


Fig. 16. Flexural test results from compression moulded GF/PP towpregs

As can be seen from the previous figures, the experimental results for the Young modulus are in accordance with the theoretical expected ones. Also, as expected, the value of that mechanical property increases with the fibre mass fraction.

The average (Av.) and standard deviation (SD) of all results from GF/PP towpregs consolidated by pultrusion or filament winding are summarised in Table 9.

As can be seen from Table 9, experimental strength results lower than the theoretical ones were obtained. In any case, such strength results seem to be compatible with the major

commercial applications expected for GF/PP composites. However, the experimentally obtained modulli results present good agreement with the theoretical ones.

Production technique	Kind of Data	Properties											
		Tensile strength (MPa)		Tensile modulus (GPa)		Flexural strength (MPa)		Flexural modulus (GPa)		Fibre mass fraction (%)		Fibre volume fraction (%)	
		Av.	SD	Av.	SD	Av.	ST	Av.	SD	Av.	SD	Av.	SD
Pultrusion	Determined	305	26	29.9	3.5	>117	4.3	22.5	0.3	78.4	1.4	56.2	2.8
	Theoretical	661.6	219	35.6	7.4	661.6	219	35.6	7.4				
Filament winding	Determined	431.0	37.6	31.0	2.8	-	-	-	-	80.2	1.5	59.0	2.8
	Theoretical	693.7	229	37.3	7.7	-	-	-	-				

Table 9. Mechanical properties of GF/PP composites

Tables 10 summarizes the experimental mechanical properties obtained on GF/PVC compression moulded plates and compares them with the theoretical ones predicted by the Classical Lamination Theory (CLT), by using the rule of mixtures and the raw materials properties shown in Table 1.

Property		Units	Determined values	
			Average	St. dev.
Flexural strength	Experimental	MPa	62.2	6.9
	theoretical		500.0	
Flexural modulus	Experimental	GPa	17.6	0.9
	Theoretical		26.7	
Fibre fraction	Mass	%	57.7	1.1
	Volume		42.7	

Table 10. Properties of composite plates made from towpreg

As may be seen, the composite flexural strength value is considerably lower than the theoretically expected one. This could be attributed, at least partially, to fiber misalignments found in the composite plates and fiber/polymer adhesion losses. In spite of the lower than expected flexural modulus values obtained, they may be considered sufficiently high to allow composites being applied in almost all commercial engineering applications.

Each GF/PVC pipe, produced by using the conventional wet fibre impregnation route previously described in the paragraph 2.5.2, was also tested in order to determine the circumferential tensile strength and fiber mass content accordingly to ASTM 2290 and EN 60, respectively.

For evaluating the consolidation quality, specimens with dimensions of $10 \times 7 \times 4 \text{ mm}^3$ were also cut from the filament wound pipes and submitted to interlaminar shear tests using a testing device based on the one described elsewhere (Lauke et al. 1992 & Nunes et al. 2005b).

After mounting this device in an universal INSTRON 4505 testing machine, simple supported specimens were submitted to shear tests using a cross-head speed of 1 mm/min.

Table 11 shows the experimental results obtained. Such results are also compared with the CLT theoretical predictions calculated in the above referred conditions. As may be seen, the strengths obtained in the GF/PVC filament wound pipes present a good approximation to the calculated theoretical values.

Property		Units	Determined values GF/PVC pipes	
			Average	St. dev.
Tensile strength	Experimental	MPa	114.7	9.5
	theoretical		236.3	
Interlaminar shear strength	Experimental	MPa	1.7	0.1
	theoretical			
Fibre fraction	Mass	%	31.7	2.1
	Volume		20.2	

Table 11. Properties of composite pipes made from PVC paste

3. Applications of thermoplastic matrix towpregs

Figures 17 to 22 show different applications successfully developed using the thermoplastic towpregs produced in this work. Figure 17 and 18 show a GF/PP pressure vessel with capacity of 0,06 m³ for incompressible fluids able to withstand an internal burst pressure up to 40 bar and a GF/PVC pipe having an internal diameter of 80 mm, respectively.



Fig. 17. Filament wound GF/PP pressure vessel processed from towpregs

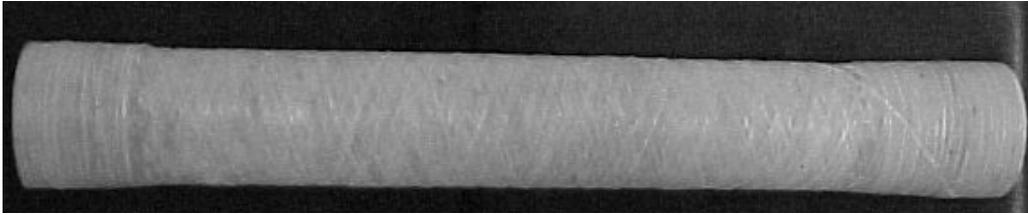


Fig. 18. Filament wound GF/PVC pipe

Figures 19 and 20 show a U-shaped $24 \times 4 \times 2$ (mm) GF/PP profile obtained by using the towpreg pultrusion and LFT compression moulded plates also processed from GF/PP towpregs. Such plates were stamped using cut towpregs mixed together at low shear stress to avoid fibre breakage.



Fig. 19. U-Shape GF/PP pultruded profile made from towpregs



Fig. 20. GF/PP LFT plates made from towpregs

Finally, a woven fabric manufactured from CF/Primospire[®] towpregs and suitable to be processed into a composite part by compression moulding is shown in Figure 21.



Fig. 21. Primospire/carbon woven fabric

4. Conclusions

The new powder-coating equipment has shown to be suitable to produce towpregs adequate for common and advanced engineering markets. From the tests made, it was found that all of those different towpregs can be easily and continuously produced at industrial production speeds between 2 a 6 m/min.

For common engineering markets glass fibre reinforced polypropylene and polyvinyl chloride matrix were studied. For these materials the optimised processing oven temperatures were in the ranges of 400°C to 450°C and 260°C to 315°C respectively.

Carbon fibre reinforced Primospire® towpregs were also studied envisaging possible applications in advanced composite structural markets. In such case, the optimised processing oven window was found to be located a much higher temperature range from 640 °C to 690 °C.

The mechanical properties of the composites processed from these towpregs by major different processing technologies were also found to be adequate either for structural as for common engineering applications.

This work also demonstrated the large potential of polymer powder deposition techniques to fabricate continuous fibre thermoplastic matrix towpregs that can be easily processed into composites with adequate engineering properties. By using efficient processing technologies different composite parts were already manufactured with success.

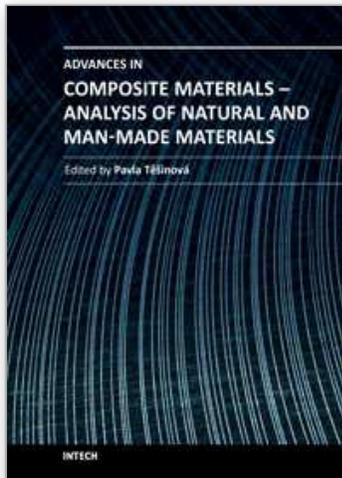
5. Acknowledgment

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Composites are made up of constituent materials with high engineering potential. This potential is wide as wide is the variation of materials and structure constructions when new updates are invented every day. Technological advances in composite field are included in the equipment surrounding us daily; our lives are becoming safer, hand in hand with economical and ecological advantages. This book collects original studies concerning composite materials, their properties and testing from various points of view. Chapters are divided into groups according to their main aim. Material properties are described in innovative way either for standard components as glass, epoxy, carbon, etc. or biomaterials and natural sources materials as ramie, bone, wood, etc. Manufacturing processes are represented by moulding methods; lamination process includes monitoring during process. Innovative testing procedures are described in electrochemistry, pulse velocity, fracture toughness in macro-micro mechanical behaviour and more.

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