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Development of a new U 200 pultrusion die to produce GRP composite profiles using different thermosetting resins

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Abstract

In this work, a steel heated pultrusion die was designed, developed and manufactured to produce U200 glass fibre reinforced thermosetting matrix (GRP) profiles [1]. The finite element analysis (FEA) was used to predict and optimise the developed die heating by using cylindrical electrical powered cartridges. To assess the new die performance it was mounted in the 120 kN pultrusion line of the Portuguese company Vidropol SA and used to produce continuously U200 profiles able to meet all requirements specified for the E23 grade accordingly to the European Standard EN 13706: 2002 [2].

After setting up the type, orientation and sequence of layers in laminate, orthophthalic, isophthalic and bisphenolic unsaturated polyester as well as vinylester resins were used to produce glass fibre reinforced U 200 composite profiles. An appropriated catalyst system was selected and the processing variables optimised for each case, namely, pultrusion pull-speed and die temperature.

Finally, the produced U200 profiles were submitted to visual inspection, calcination and mechanical tests, namely, flexural, tensional and interlaminar shear strength (ILSS) tests, to assess their accomplishment with the EN 13706 requirements.

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1. Introduction

Pultrusion has been and still remains as one of most attractive composite processing techniques, perhaps one of those presenting major growth of the whole composites industry. Currently, non-structural and structural composite pultruded profiles are being increasingly applied in several relevant markets, such as: construction, particularly where corrosive conditions are presented, electric, marine and transportation markets [3]. The excellent mechanical properties,

electrical, acoustic and thermal insulating characteristics, high corrosion resistance and integrity and low weight are main reasons for the success of pultrusion profiles in the market.

In this work a new heated die was developed and constructed to allow the Portuguese manufacturer Vidropol SA producing by pultrusion U 200 glass reinforced composite profiles for being employed in structural applications. After optimising the glass fibres impregnation/guiding and feeding system in the equipment and defining the profile laminate stacking that allow achieving the desired mechanical properties, the new die was mounted in the 120 kN pultrusion equipment from Vidropol SA for testing. Different thermosetting matrices were also used to

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manufacture the U shaped 200 glass reinforced profiles and corresponding processing window conditions defined. Finally, the U 200 produced profiles were tested to assess their accordance with the requirements of the E23 grade defined in the European Standard EN 13706:2002 in order ensure their successfully launch in the commercial market.

2. Pultrusion processing conditions

2.1. Equipment

A 120 kN pultrusion machinet from Vidropol SA was been used in this work (see Fig.1).

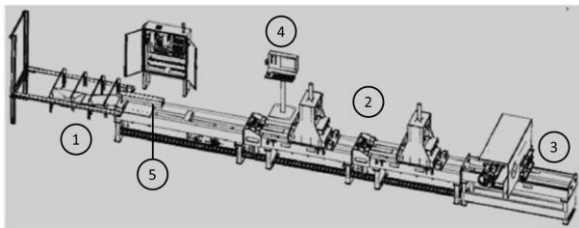


Fig. 1. Schematic drawing of pultrusion machine from Vidropol SA

As the above figure shows, the equipment is composed by five main systems: 1) fibres impregnating, guiding and feeding one; 2) pulling; 3) final cutting; 4) main command panel and, finally, 5) the profile die.

2.2. Adjusting the fibres impregnating/guiding system

Before beginning the production of any new pultrusion profile a new impregnating/guiding/feeding must be built to ensure the impregnated fibres take approximately the shape of te profile cross section nearby the pultrusion die entrance. In this work, such operation was complex due the large dimensions of the cross section of the U200 profile to be pultruded. Larger cross sections mean to use higher number of glass fibre roving strand yarns and of another fibre layers.

As may be seen in figure 2, a solution was achieved by placing two perforated polyamide (PA) plates before the steel die entrance to guide and shaping the different fibre strands and layers to the die geometry.

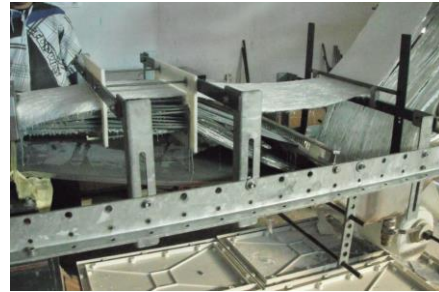


Fig. 2. Two PA perforated plates placed near the steel die entrance

To ensure the adequate impregnation of glass fibres it the guiding system was also improved and adapted between the glass rovings storage shelves and the liquid resin impregnation basin entrance (see Fig.3).



Fig. 3. guiding mechanism used at the resin impregnation basin

2.3. The U200 heated die developed

The dimensions of the U 200 glass fibre reinforced profile to be produced may be seen in figure 4.

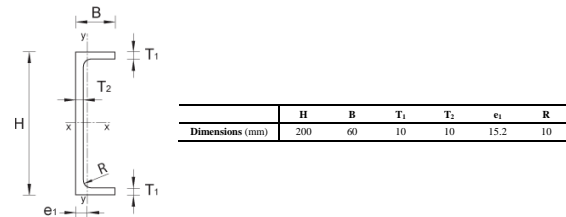


Fig. 4. Dimensions of the U 200 profile

Figure 5a) exhibits one of the extremities of the 1400 × 290 × 137 (mm) steel die designed. All internal die surfaces have been hard chrome plating treated and 28 CHC M10 screws, accordingly to DIN 912, were used to fasten tightly the upper and bottom parts. Such screws were calculated in order to support a maximum pressure of 13.8 bar in the interior of the die [3]. It was also ensured that the die supported by its extremities was able to withstand its own weight

plus the sum of the weight of all heating cartridges and composite material distributed along its entire 1400 mm length without overpassing a vertical bending displacement of 0.01 mm (see Fig. 5b)). In such case, the maximum vertical displacement, y_{max} ; was calculate as:

$$y_{max} = \frac{5 \cdot p \cdot L^4}{384 \cdot E \cdot I} \quad (1)$$

where:

- p is the weight distributed along all the die length (N/m)
- L is the total die length (L=1.400 m)
- E is the Young modulus of steel (E= 210×10⁹ Pa)
- I is first moment of inertia of the die cross section (m⁴)

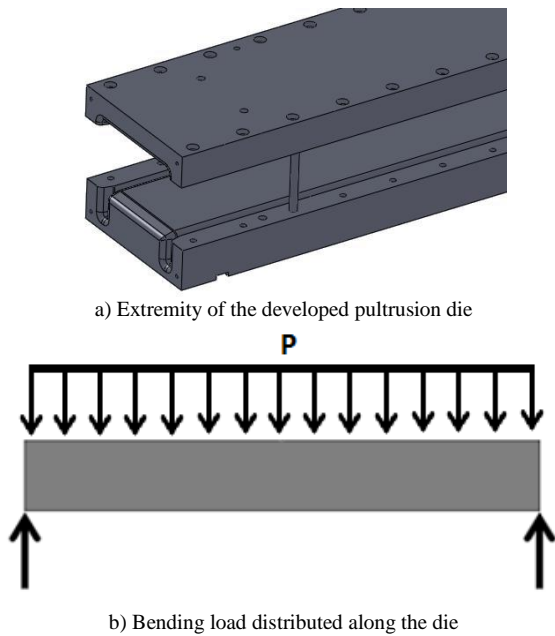


Fig. 5. Developed steel die: a) Extremity of the developed pultrusion die; b) Bending load distributed along the die

Finite element analysis (FEA) was used to optimise the die heating system composed by an assembling of cylindrical electrical cartridges. The software SolidWorks® [4] was used in the simulations considering the heat transfer problem in transient regime and the following assumptions: i) all adjacent superficies are in perfect contact, ii) the die presents a simplified geometry and, iii) the temperature distribution along time is supposed to be constant and

similar to the one defined in the work carried out by Xiao Lin Liu, et al. [5]. Additionally, the die was supposed to be at the initial temperature of 125°C, a convection coefficient of 10 W/(m²·K) has been taken and the external room temperature was considered 25°C.

A tetrahedral (4 nodes) mesh with 89233 elements was used in the model in order to decrease calculation time (see figure. 6).

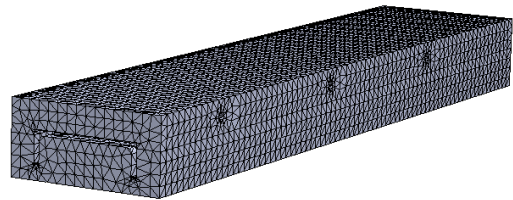


Fig. 6. Tetrahedral (4 nodes) mesh used in the FEM simulations

The positions where the thermocouples and the different cylindrical heating cartridges were placed in the new die are shown in figure 7. Each colour of the figure corresponds to one of the heating cartridge groups having the characteristics defined in table 1.

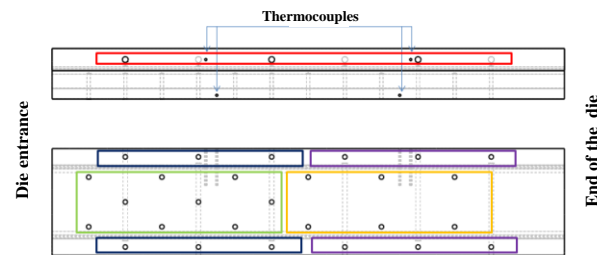


Fig. 7. Positions of the heating cartridges and thermocouples

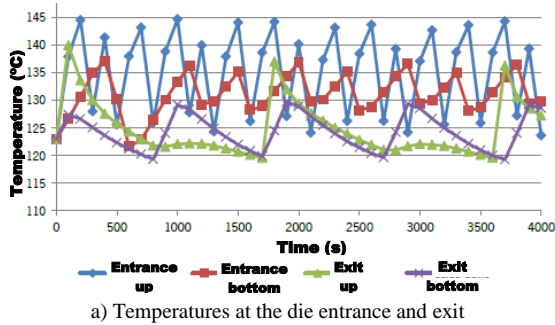
Table 1. Groups of the electric cartridge shown in Fig. 7

Cartridge group	Power (W)	Diameter (mm)	Length (mm)
Red	1000	16	250
Orange	400	12.5	70
Blue	500	12.5	90
Violet	400	12.5	90
Green	500	12.5	70

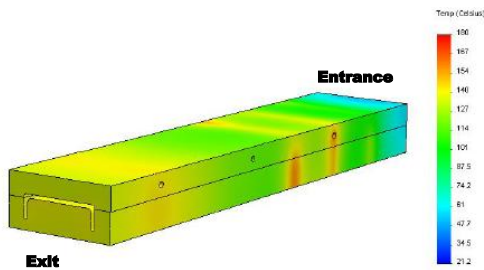
Figure 8 exemplifies two typical simulations made by using the above model. The simulation shown in Fig. 8a) presents the evolution of the die temperature near the zone where the four thermocouples were installed, two in upper part and the other two in its bottom part of the die, during a 4000 s heating and to allow maintaining temperatures in the range of

130°C - 135°C and 120°C - 125°C on the die entrance and exit, respectively. As it may be seen, the cartridges mounted nearby die entrance die switched automatically with much higher frequency “on” and “off” than those installed near the exit of the die due to the constant feed of new cold material.

Figure 8b) exhibits the temperature in the die after 2000 s (37.6 min) of heating time. The lower temperatures that may be seen in the die entrance side also reflect the continuous feeding of new cold material to the die.



a) Temperatures at the die entrance and exit



b) Die temperature after a 37.6 min of heating

Fig. 8. Simulation of the temperature evolution on the U200 die: a) Temperatures at the die entrance and exit; b) Die temperature after a 37.6 min of heating

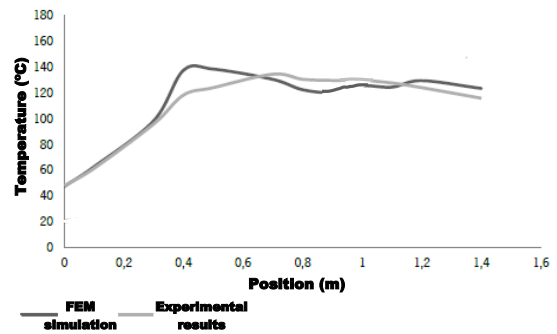
2.5. Validating the heating die simulations

As soon the new developed die became available, it was mounted in the pultrusion equipment for testing (see figure 9a)). Digital thermocouples were placed on the upper part of die to measure the temperature evolution along its length and assess the heating die FEM predictions. The tests were conducted at the pull speed of 400 mm/min using the orthophthalic polyester Resipur® 2208 from Resiquimica SA combined with two initiators, the Peroxan® PB % and Peroxan® BP Pulver 50w from Pergan, in mass contents of 1% and 2%, respectively. Furthermore, calcium carbonate was also added to the resin in a mass content of 5%

As figure 9b) shows, the evolution of temperature obtained experimentally along the die length is quite similar to the one predicted from FEM simulations. An average difference of 4% was obtained between results and, only slightly higher temperatures were predicted by FEM on the die, in the range from 0.35 m and 0.6 m, with a maximum difference of 16% nearby the 0.4 m.



a) Die mounted on the pultrusion equipment



b) Comparison between simulations and experimental results

Fig. 9. Comparison between the temperatures obtained experimentally and from FEM simulations: a) Die mounted on the pultrusion equipment; b) Comparison between simulations and experimental result

Additionally, it was observed a maximum difference of about 6% between the temperature measured on the U profile manufactured (138 °C) and that one (147 °C) predicted in the simulations carried out in the work carried out by Xiao Lin Lui et al [5]. Thus, it was possible to conclude that all experimental results obtained seemed to be in good agreement with the predictions from FEM simulations.

3. Laminate design

Almost all final properties of pultruded fibre reinforced profiles become determined by the laminate chosen, i. e. type, sequence and fibre content and orientation in each layer. In this work, the laminate of the U 200 glass reinforced profiles was mainly

selected to ensure they accomplish the requirements, namely the mechanical ones, established for the E23 grade defined in the European Standard EN 13706: 2002 [2].

The following 10 ply symmetrical laminate [Mat 450 / plain fabric 500 / unidirectional roving (0°) / plain fabric 500 / Mat 50]_s, which uses 260 glass rovings of 9600 Tex divided by two unidirectional layers, has been selected. Accordingly to the theoretical predictions made, such laminate was able to reach the minimum tensile modulus value of 23 GPa in the longitudinal direction of fibre reinforced profile as it was required by the EN 13706: 2002 for a profile belonging to the E23 grade.

In fact, an Young modulus of 27,7 GPa was predicted in the longitudinal direction for the full section of the GRP profile to be produced with the above laminate, by using the rule of mixtures and well-known equations from the micromechanics/macromechanics of composite layers and assuming the properties of raw materials and layers depicted in Table 2 [3, 6-9]. Such predicted value for the longitudinal modulus assumes that the Mat, plain fabric and unidirectional roving layers will occupy, respectively, volume contents of approximately 31%, 20% and 49% in all full section of the GRP profile.

Table 2. Raw materials and layer properties based on manufacturers datasheets and experience

Component	Young Modulus (GPa)		Density (kg/m ³)	Fibre volume content (%)	Fibre mass content (%)
	Fibre direction	Transverse direction			
Glass fibres	70.0 ^a	-	2550 ^a	-	-
Orthophthalic polyester Resipur [®] 2208	3.0 ^a	3,0 ^a	1130 ^a	-	-
Layer of unidirectional glass fibres (0°)	43.4 ^c	5.5 ^c	2010 ^c	62 ^c	78.5 ^b
Layer of Mat 450 g/m ²	7.35 ^c	7.35 ^c	1528 ^c	28 ^c	47.0 ^b
Layer of plain fabric 500 g/m ²	20.3 ^c	20.3 ^c	1954 ^c	58 ^c	75.5 ^b

^a From manufacturer datasheets; ^b Values based in previous tests & experience; ^c Calculated values

Additionally, the prevision made assumes that fibre mass and volume fractions of 61.8 % and 48.5 %, respectively, will be obtained in the final GRP profile. These values were in good agreement with those ones already determined in other GRP profiles with different transversal sections produced in similar conditions using the same equipment. It must be also

mentioned that for the selected laminate it was also possible to predict a Young modulus of 9.0 GPa in transversal direction of the GRP profile cross section, which is value higher than the minimum one of 7 GPa figure (required by the EN 13706: 2002 standard [2]).

4. Thermosetting matrices

In addition to the commonly used orthophthalic polyester resin, the possibility of using three new different resins was studied in the present work. Such resins were two unsaturated polyesters, one isophthalic (Resipur[®] 5103, from Resiquimica SA) and the other bisphenolic (Atlac[®] 382 from DSM), and a vinylester one (Atlac[®] 580 from DSM).

Such study was been done in three stages: i) the first one involved the choice of the catalyst system more appropriated for each resin, ii) the second concerned in defining the exact proportions that each catalyst component must be added the resin to achieve its full and adequate cure and, iii) the validation of the selected catalyst system was finally made in a third stage.

4.1. Selecting catalyst systems

In the pultrusion process two different basic types of initiators are usually used in the thermosetting resins cure [3]: i) one is the so-called “kicker”, which is more reactive and provides the faster beginning of the cure reaction at the lower temperatures existing on die entrance and, ii) the other, so-called “finisher”, has lower reactivity and is able to ensure a full and much smooth cure of the GRP profile inside the die at higher temperatures. In this work, two different catalyst systems based on the use of different “kickers” and “finishers” were studied for each of the new three thermosetting resins to be used.

In the case of the Resipur[®] 5103 isophthalic unsaturated polyester one of the catalyst systems was based on the Peroxan[®] BP-Pulver 50 W (Dibenzoyl peroxide), as “kicker”, and the Peroxan[®] PB (tert-butyl peroxybenzoate), as “finisher”, both initiators from the Pergan GmbH. Such catalyst system was the same that was already used with success to produce GRP profiles based in orthophthalic unsaturated polyester matrices. The other catalyst system tested with this resin the same “kicker” was selected (Peroxan[®] BP-Pulver 50 W) but the above mentioned “finisher” was replaced by the initiator Trigonox[®] C (ter-butyl peroxybenzoate) from the AkzoNobel.

Both catalyst systems studied for the Resipur® 5103 isophthalic polyester were also tested in the case of the bisphenolic polyester resin Atlac® 382.

Finally, in the case of the vinylester resin two catalyst systems were studied: i) the first one applied with the other two resins using the Peroxan® BP-Pulver 50 W and Peroxan® PB, as “kicker” and “finisher”, respectively and, ii) a catalyst system usually considered more adequate to vinylester resins based in the following initiators: the Andonox Pulcat AW-PF (methyl isobutyl ketone) and the Norox TBPB (tertiary-Butyl Peroxybenzoate), both from the Syrgis Performance Initiators AB, as “kicker” and “finisher”, respectively.

To select the more appropriate catalyst system to be used with each resin SPI gel tests at 82°C, according to the ASTM D 7029 – 09 standard [10], were performed to determine the values of the gel and cure times and exothermal pic associated to each system. Then, such determined values were compared with those obtained for the already approved catalyst system used with success in the production of GRP profiles based on the orthophthalic polyester resin Resipur® 2208.

Table 3 shows the results obtained from the SPI gel tests made.

By comparing the results obtained with the new resins to the already used orthophthalic polyester one, the following catalyst systems were selected to produce the U200 GRP profile because of closer results presented: i) initiators Peroxan® PB and the Peroxan® PB–Pulver 50 W for the isophthalic polyester Resipur® 5103; ii) initiators Trigonox C and Peroxan® PB –Pulver 50 W for the bisphenolic polyester Atlac® 382 and, iii) initiators Andonox Pulcat AW-PF and Norox TBPB for the vinylester resin Atlac® 580.

Additionally, it was also possible to conclude that, as expected, the catalyst system based on the Trigonox® C and Peroxan® BP Pulver 50 w doesn't work as curing system with the vinylester resin.

4.2. Optimising the selected catalyst systems

In a second stage, the exact level content of the “kicker” and “finisher” initiators to be added to each new resin was investigated. First, based in experience, testing and manufacturer recommendations minima and maxima levels for the content that initiators could be added to each new specific resin were established. The maxima and minima limits considered in this work for the content that any of the selected initiators

might be added to each specific resin are summarised in Table 4.

Table 3. Results obtained from the SPI Gel Tests at 82°C [10]

Resin	Catalyst system	Exo-thermal pic (°C)	Gel time (min)	Curing time (min)
Orthophthalic polyester Resipur® 2208	1% Peroxan®PB + 2% Peroxan®PB-Pulver 50 W	169.2	2.4	2.7
Isophthalic polyester Resipur® 5103	1,5% Trigonox C + 2% Peroxan® BP Pulver 50 w	173.2	3.0	4.0
	1.5% Peroxan®PB + 2% Peroxan®PB-Pulver 50 W	175.1	2.8	4.0
Bisphenolic polyester Atlac® 382	0.5% Trigonox C + 1% Peroxan® BP Pulver 50 w	181.0	6.9	8.3
	0.5% Peroxan®PB + 1% Peroxan®PB-Pulver 50 W	171.6	10.2	12.0
Vinylester Atlac® 580	1.5% Trigonox C + 5% Peroxan® BP Pulver 50 w	-	>30	-
	1.2% Andonox Pulcat AW-PF + 0.4 Norox TBPB	164,0	5.3	7.4

Assuming these values a design of SPI gel testing experiments (DOE) at 82°C [11] was built by combining maximum and minimum values of the initiator contents settled for each new resin. Table 5 summarise results obtained from this collection of tests.

Thus, obtained results were analysed in order to minimise the gel and curing times in the case of the isophthalic and bisphenolic polyesters and vinylester resins, respectively. By using these criteria the following catalyst systems were selected to produce the U200 GRP profiles:

- i) To add the maximum contents of both initiators (1.5% Peroxan® BP + 5% Peroxan® BP Pulver 50 w) to the isophthalic polyester Resipur® 5103. In such case, it will be expected to obtain values of 2.1 min and 178,9 °C as gel time and exothermal pic, respectively.
- ii) to use also the maximum contents of the initiators (2% of Trigonox® C+5% Peroxan® BP Pulver 50 w) in the bisphenolic polyester resin Atlac® 382. In such case, it will be expected to obtain values of 2.9 min

and 193,5 °C as gel time and exothermal pic, respectively.

iii) Finally, it was decided to use a catalyst system based on the application of 0,6% of Norox® TBPB + 1,2% of Andonox® Pulcat AW-PF to the vinylester resin Atlac® 580. In this case

Table 4. Maximum and minimum content of initiators that could be added to each new resin

Resin	Maximum and minimum initiator content (%)									
	"Kickers"				Finishers					
	Peroxan®P B-Pulver 50 W		Andonox® Pulcat AW-PF		Peroxan®P B		Trigonox® C		Norox® TBPB	
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
Isophthalic polyester Resipur® 5103	2	5	-	-	1	1.5	-	-	-	-
Bisphenolic polyester Atlac® 382	2	5	-	-	-	-	1	2	-	-
Vinylester Atlac® 580	-	-	1.2	2.4	-	-	-	-	0.4	0.6

Table 5. Curing variables obtained from the SPI Gel tests made with the selected catalyst system

Resin	Catalyst system		Exothermal pic (°C)	Gel time (min)	Cure time (min)
	Kicker	Finisher			
Isophthalic Resipur® 5103	2% Peroxan® BP Pulver 50w	1% Peroxan® BP	182.3	4.2	-
		1.5% Peroxan® BP	184.9	3.8	-
Bisphenolic Atlac® 382	5% Peroxan® BP Pulver 50 w	1% Peroxan® BP	184.6	2.4	-
		1.5% Peroxan® BP	178.9	2.1	-
Vinylester Atlac® 580	2% Peroxan® BP Pulver 50 w	1% Trigonox® C	183.6	6.4	-
		2% Trigonox® C	204.6	4.6	-
Vinylester Atlac® 580	5% Peroxan® BP Pulver 50 w	1% Trigonox® C	193.5	4.0	-
		2% Trigonox® C	198.7	2.9	-
Vinylester Atlac® 580	1.2% Andonox Pulcat AW-PF	0.4% Norox® TBPB	155.1	-	7.4
		0.6% Norox® TBPB	135.8	-	7.1
Vinylester Atlac® 580	2.4% Andonox Pulcat AW-PF	0.4% Norox® TBPB	164.3	-	6.4
		0.6% Norox® TBPB	172.5	-	5.8

The validation of the previously optimised catalyst systems has been done in this third stage by repeating SPI gel tests at 82°C in accordance to the ASTM D 7029 – 04 standard. Results obtained seem to be similar to the expected ones and then reliable and suitable to allow using the resins and respective catalyst systems selected on the production of the desired U200 GRP profiles.

5. Optimising the pultrusion process

The optimisation of the pultrusion process was achieved in order to manufacture the U200 GRP profiles in good conditions at maximum production rate. This was done by selecting three temperature range at the die entrance and end, measuring the temperature on the produced profile at the die exit, its Barcol hardness and verifying if all the visual and dimensional level of defects observed on the final manufactured U200 profiles were in accordance with the requirements and tolerances imposed by the European Standard EN 13706-2: 2002.

Monitoring the temperature on the profile at the die exit it is a good measure of the degree of cure achieved. Temperatures on the profile higher than the ones in the die means cure it was not complete done inside of the die. The Barcol hardness measured on the profile is another good indicator of the degree of cure reached. According to EN 13706-2: 2002 standard and experience Barcol hardness values lower than 80% of the figures given by the resin manufacturer means uncured profiles that should be rejected or subject to post-cure treatment.

From the work carried out it was concluded that the U200 GRP should be produced in thee following pultrusion conditions accomplished the requirements of the EN 13706-2: 2002 standard:

- i) Using the orthophthalic polyester Resipur® 2208 as matrix: 140 °C and 130 °C as temperatures of die entrance and exit, respectively, at the pull speed of 400 mm/min.
- ii) Using the isophthalic polyester Resipur® 5103 as matrix: 140 °C and 130 °C as temperatures of die entrance and exit, respectively, at the pull speed of 300 mm/min.
- iii) Using the bisphenolic polyester Atlac® 382 as matrix: 140 °C and 130 °C as temperatures of die entrance and exit, respectively, at the pull speed of 200 mm/min.
- iv) Using the vinylester Atlac® 580as matrix: 160 °C and 150 °C as temperatures of die entrance and exit, respectively, at the pull speed of 200 mm/min.

6. Testing the U200 produced profile

Finally, the pultruded U200 GRP profiles based on the orthophthalic matrix were submitted to the batch of mechanical tests defined in the EN 13706-2: 2002 to verify their accordance to the requirements of that

European Standard. Table 6 shows the results obtained from those tests and compares them with the standard requirements.

Table 6. Comparison between results obtained, minima requirements and theoretical predict ones

Property	Test method	Required values	Experimental values	Theoretic predicted values
Elastic modulus in a full section flexural test (GPa)	Annex D, EN 13706-2	23	27.5 ± 3.4	27.7
Tensile modulus, axial direction (coupon test) (GPa)	EN ISO 527-4	23	32.7 ± 1.8	27.7
Tensile modulus, transversal direction (coupon test) (GPa)	EN ISO 527-4	7	7.0 ± 0.2	9.0
Tensile strength, axial direction (coupon test) (MPa)	EN ISO 527-4	240	369.4 ± 16.9	-
Tensile strength, transversal direction (coupon test) (MPa)	EN ISO 527-4	50	59.8 ± 4.0	-
Pin-bearing strength, axial direction (coupon test) (MPa)	Annex E, EN 13706-2	150	200.1 ± 1.8	-
Pin-bearing strength, transversal direction (coupon test) (MPa)	Annex E, EN 13706-2	70	202.4 ± 1.7	-
Flexural strength, axial direction (coupon test) (MPa)	EN ISO 14125	240	617.3 ± 16.9	-
Flexural strength, transversal direction (coupon test) (MPa)	EN ISO 14125	100	78.2 ± 9.5	-
Interlaminar shear strength, axial direction (coupon test) (MPa)	EN ISO 14130	25	28.1 ± 0.4	-

As it may be seen from previous table 6, the profiles accomplished with almost all mechanical requirements of the standard and presented characteristics in good agreement with theoretical predicted values, with exception of the flexural strength in the transversal direction where a value lower than the required one was obtained. This last result as well as the experimental result of the transversal tensile modulus obtained, which was very similar to the required one, denote the necessity of enhance a little bit more the mechanical properties of the profile in its transversal direction. This could be easily done by include two more glass plain fabric layers in the profile laminate.

7. Conclusions

A study concerning the production of a U200 GRP profile by pultrusion was developed in this work. A steel die was designed and manufactured and all processing conditions optimised. The production of the profile using four different matrices was also studied and the catalyst system for three new matrices established by using SPI gel tests.

The laminate of the GRP profiles to be produced was also designed to accomplish all production and market requirements, such as those established for the grade E23 of the European Standard EN 13706 : 2002.

Finally, produced profiles were also submitted to mechanical testing to verify their accomplishment to all requirements of the above mention European standard. In spite of the good results obtained and their good agreement with the theoretical predicted ones it was verify to be necessary enhancing a little bit more the transversal properties of the U200 GRP profile by introducing two more layers of glass fibre plain fabric in the laminate

Acknowledgements

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