Securing a 100+ year design lifetime for Pressure PVC-U Pipes through Optimized Extrusion and QC Testing Conditions

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Abstract. In order to generate public data demonstrating that pressure PVC-U pipes can be designed for 100+ year, an investigation project has been launched in 2020 by CEIS and PVC4Pipes. This project aims to clarify the relationships between the processing and QC testing conditions, and the design lifetime of PVC-U 250 pipes. Ca/Zn stabilized pressure pipes were extruded with different processing temperatures and characterized by their DSC onset temperature, DSC degree of gelation and resistance to dichloromethane. Their long-term hydrostatic strength was predicted using the methods described in ISO 9080. The main desired outcome of this project was to set up a correlation between the extrusion temperature and the 97.5% Lower Prediction Level of the stress that a PVC-U pipe can withstand after 100 years (LPL100y). The results of this study show that an extrusion temperature of 180ºC is high enough to achieve a MRS250 classification. A moderate increase (+5ºC or +10ºC) of the extrusion temperature leads to a smooth decrease in the slope of the regression curve at 20ºC and, therefore, to an increase of the predicted LPL100y values. Higher processing temperatures (≥195ºC) result in only marginal increases in LPL100y, with clear risk of material degradation during processing. The increase of LPL100y allows the initial MRS250 classification to be extended up to 100+ years. The resulting benefit for the prescribers and designers of pressurized water supply networks is to design their networks for 100+ years by using of design stress and design coefficient values typically used for a design lifetime of 50 years.

1. Introduction

For decades, PVC pipes have helped to provide access to clean water and sanitation all over the world at affordable costs. Being easy to install and highly durable, the PVC pipes have enabled efficient irrigation even in remote areas and have helped to address global issues such as soil erosion and water scarcity.

Over the years, it has become evident that the life expectancy of plastic pipe system for the transport of pressurized water made of PVC-U can far exceed the 50-year design lifetime secured by the product standards, by maintaining its original design conditions.

Being part of a comprehensive project, whose objective is to highlight the close relationship between processing temperature, gelation degree and long-term pressure resistance of PVC-U pipes, the present study, focuses on processing conditions that allow them to reach a 100+ year design lifetime.

2. Nomenclature

DSC Differential Scanning Calorimetry
LPL Lower Prediction (stress) Limit
MRS Minimum Required Strength

3. Experimental

The pipes were prepared and tested according to the below described process:

3.1. Raw materials and recipe mixing conditions.

The pipe compound used in this study, consists in a mixture of Poly (vinyl chloride) (PVC), mineral filler (chalk) and a one-pack Ca/Zn stabilizer package, and includes pigments to achieve desired color (iron grey, RAL7011). The components of the mixture are pre-weighted and added into a two-stage hot

(vertical) & cold (horizontal) mixer where mineral filler and additives are first well dispersed into the PVC powder that heats up to 120°C, and then slowly cooled down to 55°C. The ready-to-use compound (premix) is not sieve-screened prior to silo storage.
The references of used raw materials and their respective dosage level in p.h.r. (per hundred resin) are depicted in the Table 1.

Component	Reference	Supplier	Dosage level (p.h.r.)
S-PVC (K66-68)	Solvin 267 RC [a]	Ineos Inovyn Ltd.	100
Filler (coated chalk)	Lubricarb 3TS [b]	Minera del Santo Angel S.L.	6.0
Additives (incl. pigment)	Cores SZ 7011[c]	Asua Products S.A.	3.2

Table 1: PVC-U pipe compound composition and dosage level.

(a) S-PVC Solvin 267 RC is a well-known extrusion grade homopolymer with a bulk density of 0.580 g/cc and a viscosity index of 67 manufactured in the Ineos Inovyn Ltd.'s site at Martorell, near Barcelona.
(b) The filler Lubricarb 3TS is a coated calcium carbonate with a D50 of 2.6µm, obtained from the limestone quarries of Minera del Santo Angel, S.L. near Sevilla.
(c) Cores SZ 7011 is a Ca/Zn one-pack stabilizer package including Grey RAL 7011 pigment, developed by the well know Spanish stabilizer Asua Products S.A. in Sondika, near Bilbao.

3.2. Extrusion Test parameters

After a storage period longer than 24 h in a silo, the elaborated premix is being fed to the extruder: a conical twin screw extruder (CM-80-SC, Milacron Inc.) with gravimetric dosage unit (SAVEOMAT, Inoex GmbH) in the first trials (PLA0031/20) and a parallel twin screw extruder (KMD-2-130/26D, KraussMaffei GmbH) with gravimetric dosage unit (SAVEOMAT M2, Inoex GmbH) in the last tests (PLA0090/23). The extruder adaptor is fitted with a pressure and temperature sensor probe (Gefran, spa) located in the melt stream. A standard spider type die-head LC250, (extrusion range DN140-250mm) is selected to shape the pipes.
All pipes are extruded to comply with a S12.5 (C=2.0) DN 140x5.4mm PN10 classification, as defined in ISO 1452-2:2009. The respective extrusion conditions used for the pipes tested in this paper, are detailed in Table A1& A2 of Annex A.

3.3. Extrusion Pipe Samples

For the present project, each extrusion test reference has been identified with their respective melt temperature: 175°, 180°, 185°, 190° and 195°C as measured by the temperature sensor probe. From each extrusion test, several 6-meter-long pipe samples have been selected. Each pipe sample has been clearly identified with their test reference number and extrusion melt temperature.

3.4. DSC Evaluation

A complete Differential Scanning Calorimetry (DSC) analysis has been performed following ISO 18373:2007. Four samples have been taken at 0°, 90°, 180° and 270° positions around the pipe circumference. Thin disk obtained by a hole punch of 20±10 mg is prepared. Each disk is encapsulated in an aluminum pan with perforated & crimped cover to reduce sample movements while melting.

DSC analyses have been carried out in a Perkin Elmer DSC4000 instrument with analysis software Pyris V.10.1.0.0412. Temperature scans runs from 35°C to 240°C at 20°C/min heating speed and N2 purge gas flow @23ºC. The processing temperatures of the pipes were determined by measuring the B-Onset temperatures as described in ISO 18373-2:2007. Gelation degrees were determined as described in reference 1.

3.5. Long-term hydrostatic pressure test

The equipment involved in hydrostatic pressure testing includes an IPT 1721-061 pipe tester (range up to 100 bar, ± 2 %), two IPT 1751-1761 liquid baths (range 20°C & 60°C, ± 2°C, respectively) and diverse DN140mm end-cups sets IPT 1732-0140, all from Institut für Prüftechnik Gerätebau GmbH & Co. KG.

A comprehensive pressure test program has been planned in accordance with ISO 1167-1:2006 & ISO 1167-2:2006. Table 2 and Table 3 show the respective pressure testing program at 20°C and 60°C with the rupture times of test specimens. Pressure stresses has been selected to achieve rupture times distributed over at least three decades in time. Two TEPFFA and one ISO 1452-2:2009 control points are included in the testing program.

Pipe Sample	Pressure testing rupture times (h)			
Ref / °C	20°C/40MPa	20°C/37MPa	20°C/35MPa(a)	20°C/31.87MPa(b)
05/175°C	32.8/33.4/32.7	149/275/283	498/630/656	9388(c)/9388(c)/9102(c)
06/180°C	20.3/22.5/20.4	213/241/312	476/502/524	9388(c)/9388(c)/9216(c)
07/185°C	26.1/27.1/28.1	187/304/232	427/493/642	9093/9328/10359(c)
08/190°C	17.0/13.0/15.1	232/219/239	632/645/716	6446/6051/6883
09/195°C	18.7/16.4/18.4	288/248/227	628/1006/1281	3298/5550/7208

Table 2: Long term pressure test program (20ºC)
(a) TEPFFA control point; (b) ISO 1452-2:2009 1000h control point; (c)Test stopped before rupture.

Pipe Sample	Pressure testing rupture times (h)			
Ref / °C	60°C/19MPa	60°C/17MPa	60°C/15MPa	60°C/11.95MPa(a)
05/175°C	8.8/7.0/6.5	160/135/133	257	1000(b)/1000(b)/1000(b)
06/180°C	6.5/7.7/8.8	89/55/102	665	1000(b)/1000(b)/1000(b)
07/*03b*/185°C	6.6/6.6/6.8	116/122/110	*321/470/493*	*1514/1513(c)/1513(c)*
08/*04b*/190°C	7.4/6.2/6.3	120/114/131	*400/453/476*	*3228(c)/3281(c)/3281(c)*
09/*05b*/195°C	6.2/9.7/5.4	78/127/138	*273/308/371*	*2528(c)/2888(c)/2888(c)*

Table 3: Long term pressure test program (60ºC)
(a) TEPFFA control point; (b) Test stopped before rupture; (c) On test

MRS evaluations and LPL50y & LPL100y predictions are performed according to ISO 9080:2003, with Pipeson© Analyzer licensed software.

4. Results and Discussion

The graphical extrapolation obtained for each reference evaluated, are plotted in log(σ) vs log(t) graphs generated with the Pipeson© Analyzer software. The tables embedded in Figures 1 to 3, show the LPL_{50y} & LPL_{100y} predicted values at 20°C for their respective reference.

As consequence of previous works [3] [4] we focus on results from pipe samples extruded with melt temperatures ≥185°C.

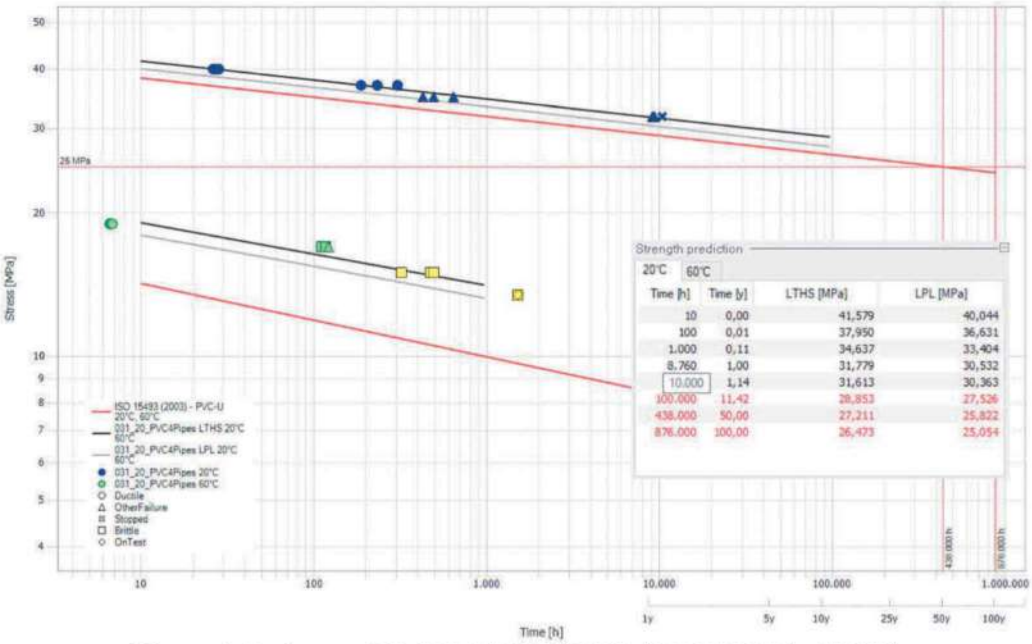


Figure 1: Reference PLA0030/20.07 & PLA0090/23.03b (185°C)

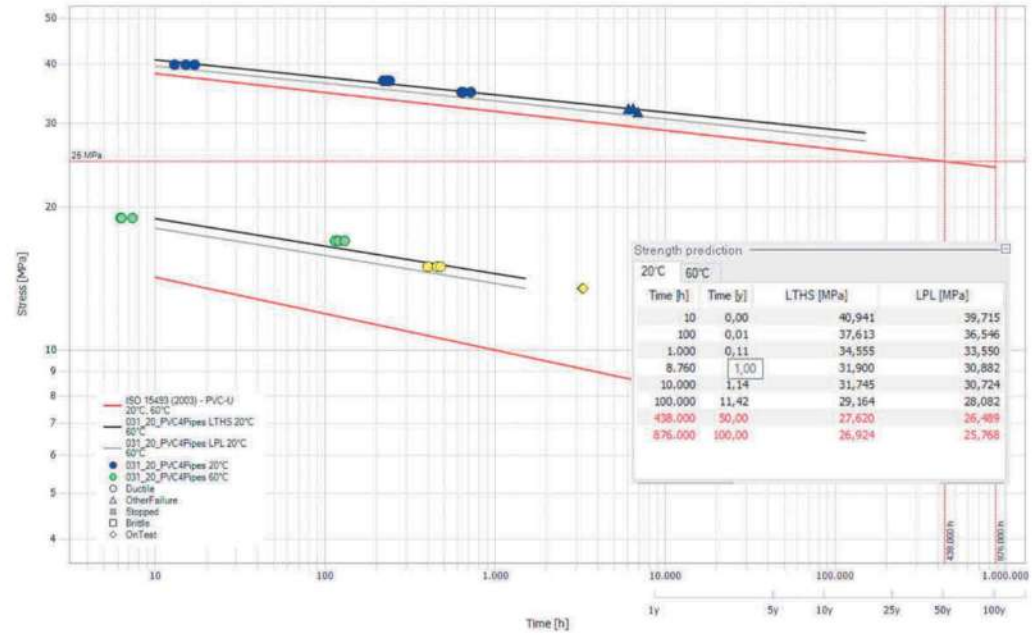


Figure 2: Reference PLA0030/20.08 & PLA0090/23.04b (190°C).

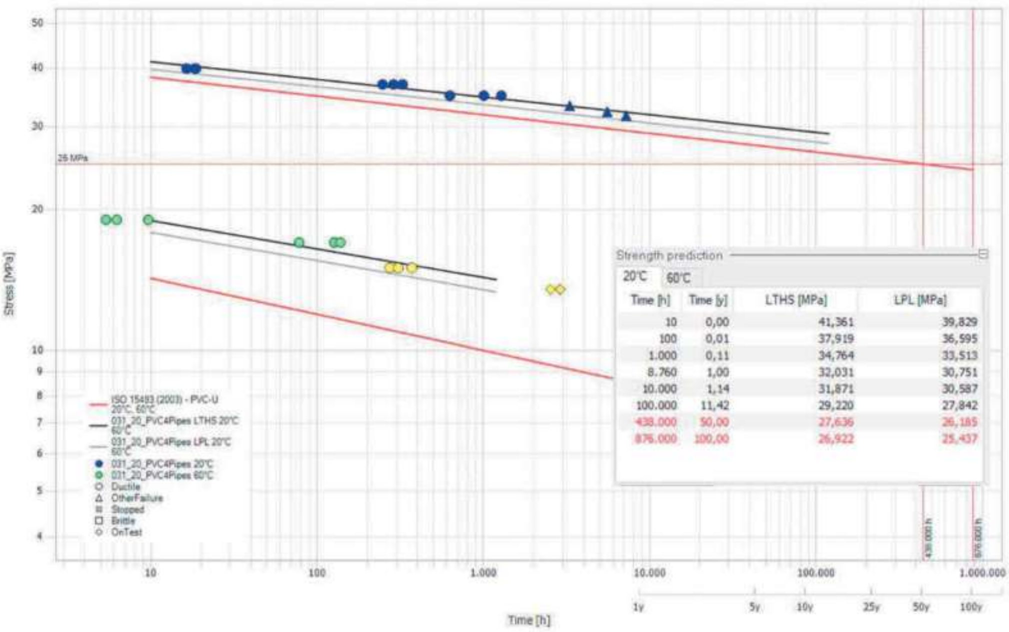


Figure 3: Reference PLA0030/20.09 & PLA0090/23.05b (195ºC).

Yellow highlighted values in the Figures 1, 2 & 3 correspond with hydrostatic long-term pressure test rupture times performed on new 2023 extrusion pipe samples ref. 03b, 04b & 05b (with identical recipe but processed on twin-screw parallel extruder). Those new rupture times fits perfectly with previous regression curves derived from rupture times of pipe samples ref. 07, 08 & 09 (processed with twin-screw conical extruder on 2020). In both cases the PVC resin was the same (Solvin S 267 RC from Inovyn) and very close k-value analysis (67,0 in the former lot vs. 66,75 in the last one).

The slope of the predicted regression curves drops when melt temperature increases resulting in higher LPL_{50y} & LPL_{100y} predicted values as clearly shown in Table 4.

Melt Temp. (ºC)	PLA0031/20 Sample	PLA0090/23 Sample	LPL_{50y} (MPa)	LPL_{100y} (MPa)
185	07	03b	25,82	**25,05**
190	08	04b	26,49	25,77
195	09	05b	26,18	25,44

Table 4: LPL_{50y} and LPL_{100y} predictions.

LPL_{50y} & LPL_{100y} extrapolation limits now extended up to 10^5 hours and ongoing long-term pressure test will help us to make more accurate predictions of the currently available LPL_{100y} estimations.

The pressure resistance of PVC-U pipes is well known to be related with gelation degree achieved during extrusion process [1] [2] As described elsewhere [3] [4] a strong relationship between the predicted LPL's and the gelation degree measured by DSC, following ISO 18373-1:2007 is evidenced (Figure 4). A gelation degree higher than 70% is needed to achieve a LPL_{100y} prediction of 25 MPa. Further increases in gelation degree beyond 80%, only lead to marginal gains in LPL's.

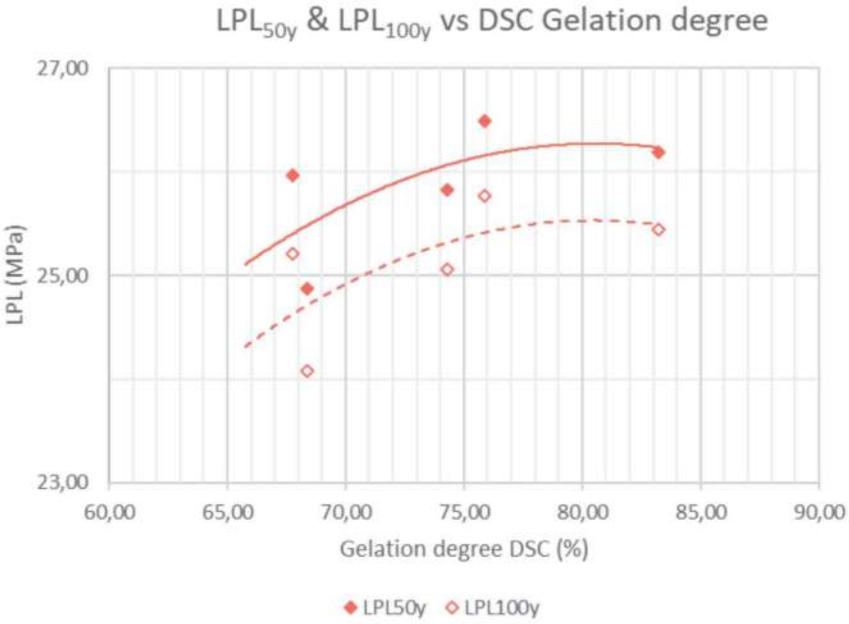


Figure 4: LPL vs Gelation degree by DSC.

The above correlation could also be evidenced between the LPL's and B-onset temperatures (Figure 5). Estimated LPL_{50y} increases from 25 MPa up to 27 MPa with B-onset temperature because a decrease in the slope of regression curve. B-onset temperature higher than 180°C is required to achieve a MRS 250 classification. Achieving an estimated LPL_{100y} above 25 MPa requires a B-onset temperature of at least of 185°C. Only marginal gains on LPL_{50y} & LPL_{100y} are obtained when increasing onset temperatures above 190°C and risk of premature failures because PVC degradated black spots increases (Picture 01).

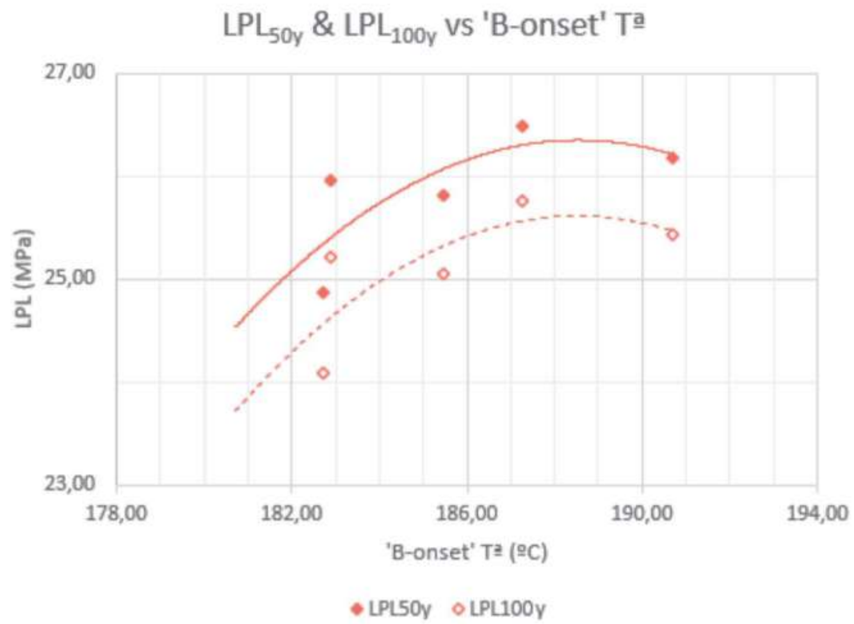
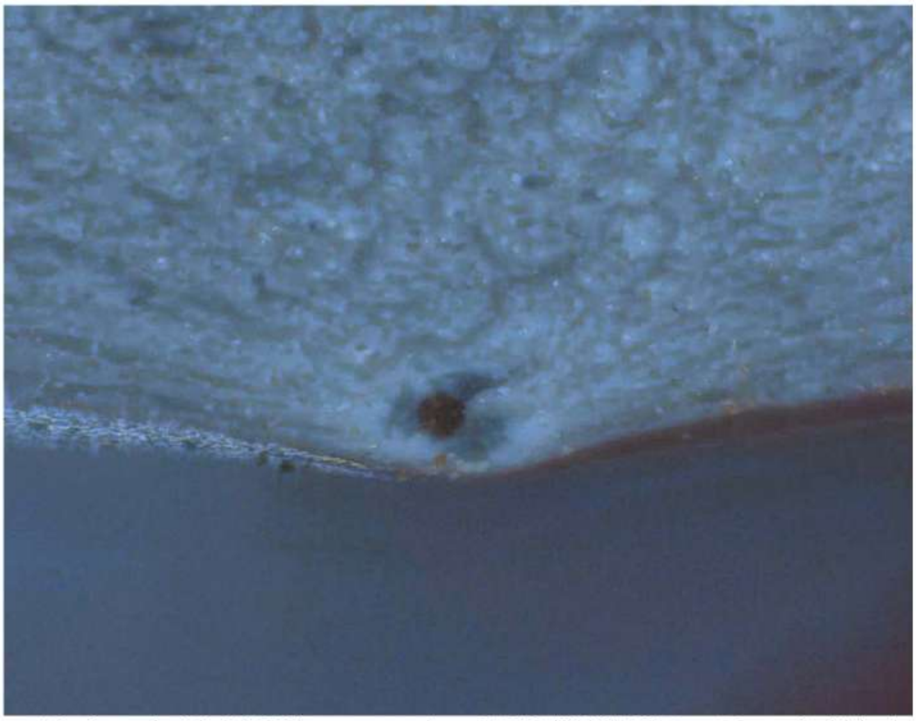


Figure 5: LPL vs B-onset temperature by DSC.


Picture 01: Sample 09 (195ºC), rupture time 5550h/20ºC/32Mpa. Degraded black spot.

It has also been shown possible to correlate the B-onset temperature with the mass temperature of the melt at the extruder/die head adaptor (Figure 6), despite an offset related to the type of temperature probe used that should be adjusted for each recipe/extruder/probe.

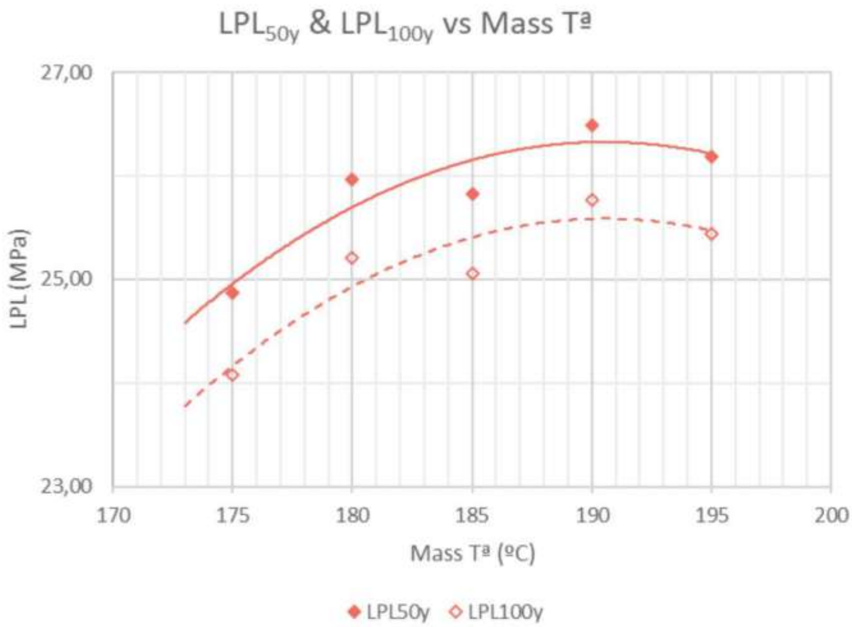


Figure 6: LPL vs Melt temperature.

5. Conclusions

Long-term pressure test rupture times of PVC-U pressure pipes extruded in different conditions have been evaluated according to ISO 9080:2003 with Pipeson© Analyzer software and their estimated Lower Prediction Limits have been correlated with both DSC gelation degree and B-onset temperature.

An adequate Ca/Zn recipe made with PVC ±67 k-value, processed with conical or parallel twin screw extruder, could achieve a LPL_{100y} ≥25Mpa when the melt temperature is at least of 185ºC.

DSC analysis (B-onset temperature, gelation degree) is a powerful tool for Quality Control of both extrusion and testing conditions.

Despite a B-onset temperature of 180°C is required to achieve a the MRS 250 classification, to achieve a LPL_{100y} prediction above 25 MPa needs a B-onset temperature of at least of 185°C. DSC gelation degree of 70% is the minimum needed to achieve a LPL_{100y} estimation of 25 MPa. Further increases in gelation degree beyond 80%, only lead to marginal gains in LPL's and increases risk of PVC degradation.

References

[1] Pimentel Real LE, Joao IM, Pimenta SI and Diogo HP, 2018, Polymer Testing, **70,** 481.
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[3] Lahoz J, 2022, Proc. PVC4Pipes Conference (Bologna).
[4] Lahoz J, 2023, Proc. 21st Plastic Pipes Conference (Lake Buena Vista).