

PVC-U PIPES: OPTIMAL EXTRUSION CONDITIONS FOR A 100+ YEAR DESIGN LIFETIME - PART II

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SHORT SUMMARY

It is well known that the extrusion conditions decisively affect the degree of gelation of PVC-U and through it, the physical characteristics, and properties of PVC-U pipes for the transport of pressurized water. The design lifetime of PVC-U pipe will, consequently, also be affected. The choice of optimal extrusion conditions can lead to an extension of the design lifetime to more than 100 years by keeping the initial design stress values and design coefficients unchanged.

KEYWORDS

PVC-U pressure pipes, onset temperature by DSC, Lower Prediction Level, Service Design Lifetime.

ABSTRACT

In order to generate public data demonstrating that pressure PVC-U pipes can be serviced for 100+ years, an investigation project has been launched in 2020 by CEIS and PVC4Pipes to clarify the relationships between the processing and QC testing conditions, and the design lifetime of PVC-U 250 pipes. Calcium-based 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 180°C is high enough to achieve a MRS250 classification. A moderate increase (+5°C or +10°C) of the extrusion temperature leads to a smooth decrease in the slope of the regression curve at 20°C and, therefore, to an increase of the predicted LPL100y values. Higher processing temperatures ($\geq 195^\circ\text{C}$) 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 to 100+ years. The resulting benefit for the prescribers and designers of pressurized water supply networks is to design their networks for 100+ years the use of design stress and design coefficient values typically used for a design lifetime of 50 years.

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 systems 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.

NOMENCLATURE

DSC: Differential Scanning Calorimetry; *LPL*: Lower Prediction (stress) Limit; *MRS*: Minimum Required Strength.

EXPERIMENTAL

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

1. RAW MATERIALS AND RECIPE MIXING CONDITIONS

The pipe compound used in this study consists of 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 levels in p.h.r. (per hundred resin) are depicted in 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 based one-pack stabilizer package including Grey RAL 7011 pigment, developed by the well know Spanish stabilizer Asua Products S.A. in Sondika, near Bilbao.

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, Krauss Maffei 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. 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.

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 @23°C. 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.

5. LONG_TERM HYDROSTATIC PRESSURE TESTS

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 stress 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 (20°C)

(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	1503/1515/1597
08/04b/190°C	7.4/6.2/6.3	120/114/131	400/453/476	4178/4448/4673 ^(c)
09/05b/195°C	6.2/9.7/5.4	78/127/138	273/308/371	3424 ^(c) /7390 ^(c) /7390 ^(c)

Table 3: Long term pressure test program (60°C)

(a) TEPFFA control point; (b) Test stopped before rupture; (c) On test.

6. MRS EVALUATION: LPL_{50y} & LPL_{100y} PREDICTIONS

MRS evaluations and LPL_{50y} & LPL_{100y} predictions are performed according to ISO 9080:2003, with Pipeson© Analyzer licensed software.

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. As consequence of previous works [3] [4] we focus on results from pipe samples extruded with melt temperatures $\geq 185^\circ\text{C}$.

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)

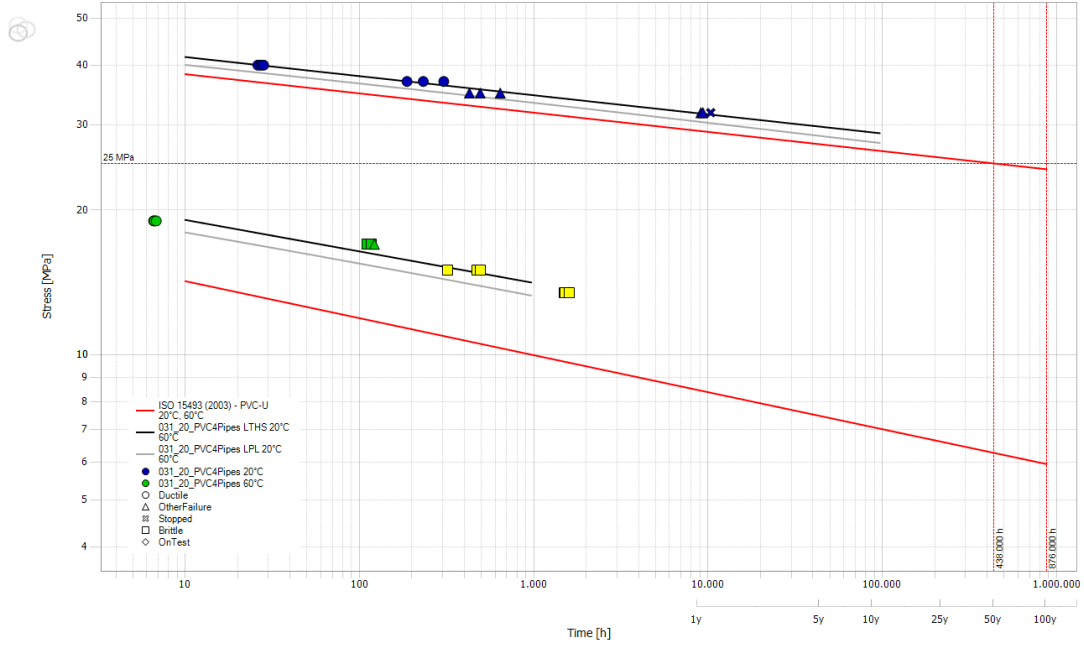


Figure 1: References PLA0031/20.07 & PLA0090/23.03b (185°C).

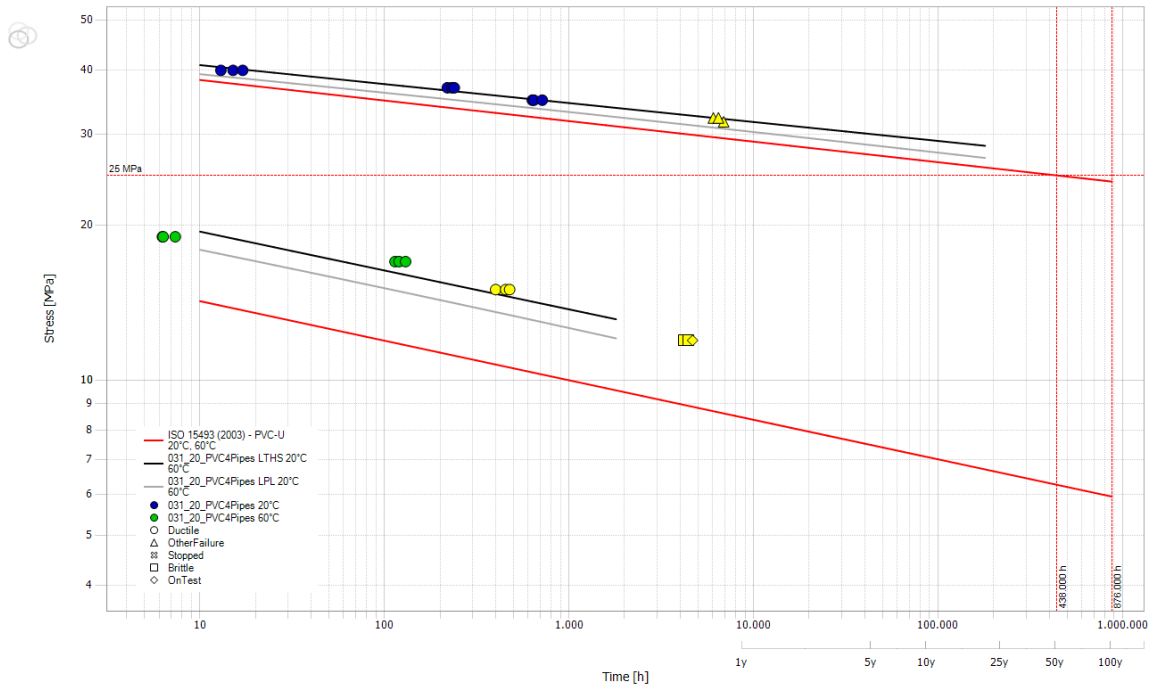


Figure 2: References PLA0031/20.08 & PLA0090/23.04b (190°C).

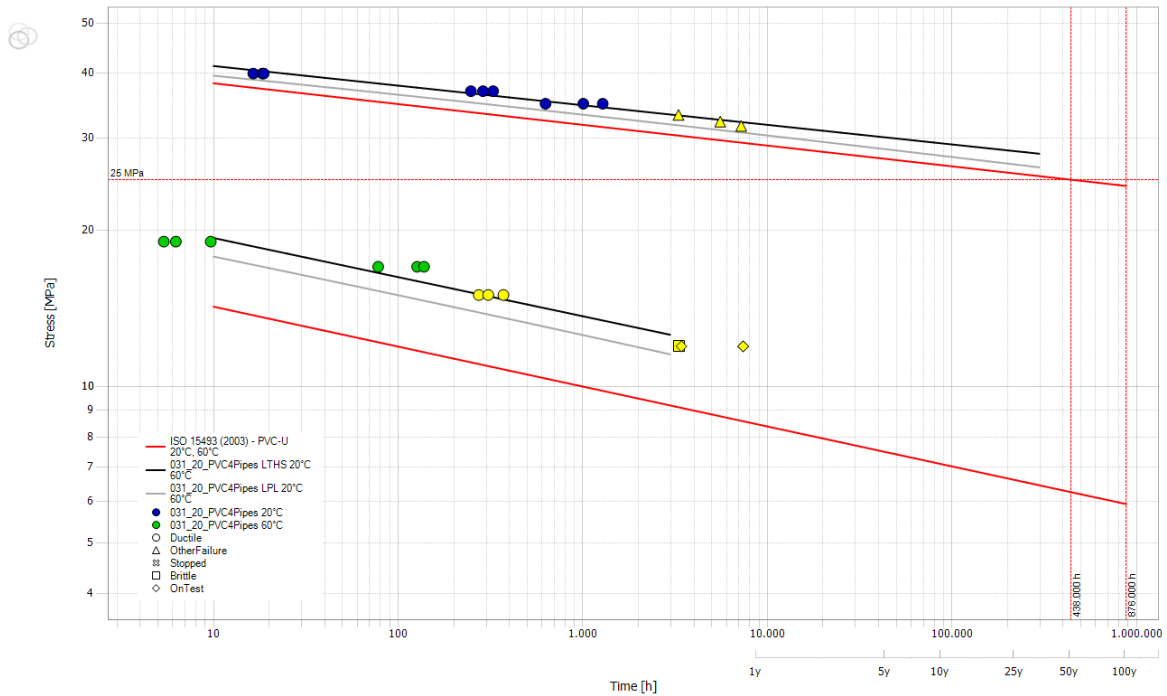


Figure 3: References PLA0031/20.09 & PLA0090/23.05b (195°C).

The slope of the predicted regression curves drops when melt temperature increases resulting in higher LPL50y & LPL100y predicted values as clearly shown in Table 4.

Melt Temp. (°C)	PLA0031/20 Sample	PLA0090/23 Sample	LPL50y (MPa)	LPL100y (MPa)
185	07	03b	25,827	25,060
190	08	01b, 04b	26,067	25,337
195	09	02b, 05b	25,971	25,217

Table 4: LPL50y and LPL100y predictions.

A combined regression curve with all individual values (Melt Temperatures 185°C, 190°C & 195°C) are plotted in $\log(\sigma)$ vs $\log(t)$ graph generated with the Pipeson© Analyzer software in Figure 4. LPL50y & LPL100y extrapolation limits now extended beyond 500000 hours, and the ongoing long-term pressure test will help us to make more accurate predictions of the currently available LPL100y estimations.

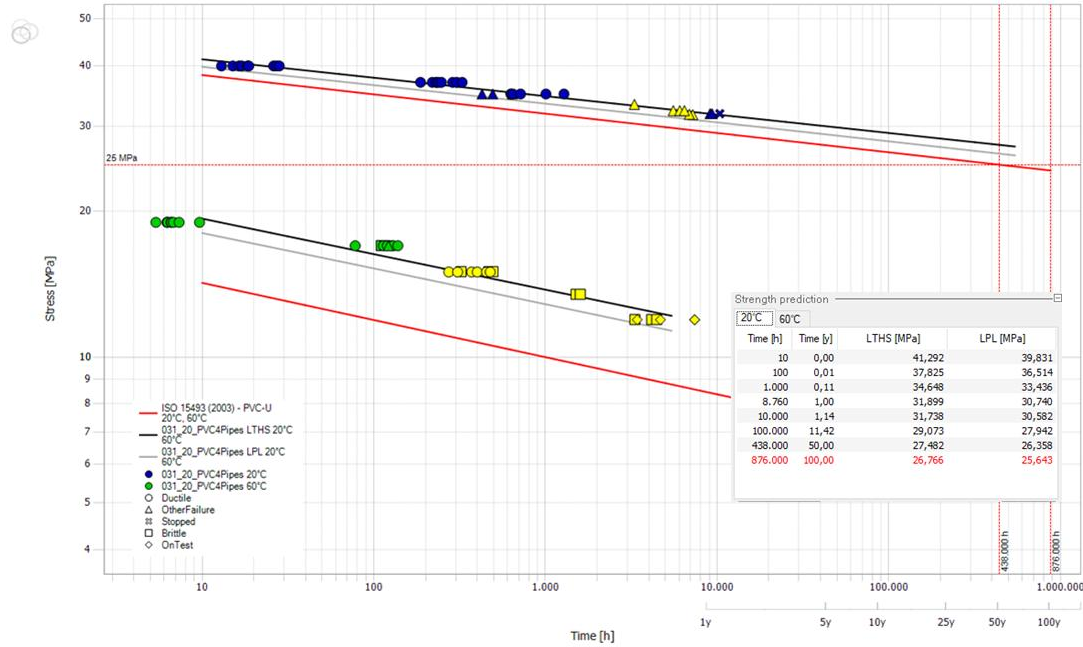


Figure 4: References PLA0031/20 & PLA0090/23 (Melt Temperatures: 185°C, 190°C & 195°C).

The pressure resistance of PVC-U pipes is well known to be related to gelation degree achieved during extrusion process [1] [2] As described elsewhere [3] [4] a strong relationship between the predicted LPL's vs the 'B-onset' temperature and the Gelation Degree (%), both measured by DSC following ISO 18373-1:2007 is clearly evidenced (Figures 5 and 6).

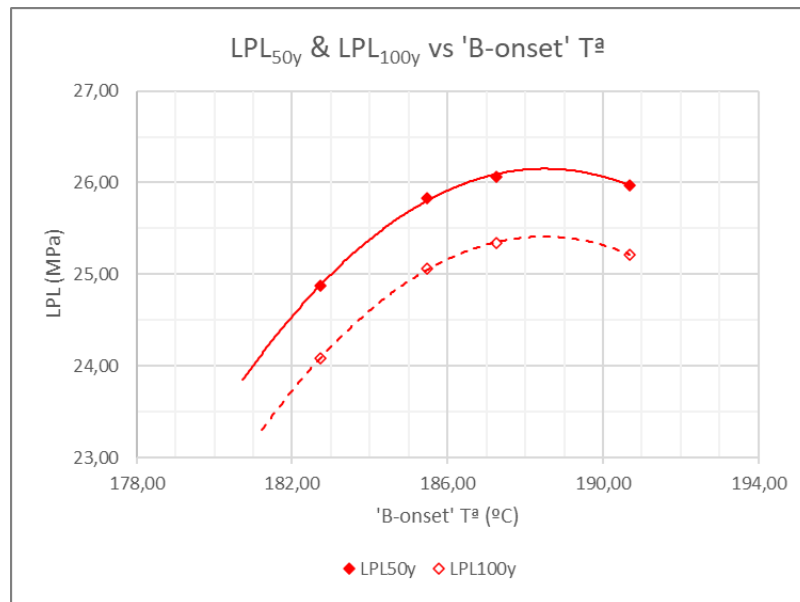


Figure 5: LPL vs. 'B-onset' temperature by DSC.

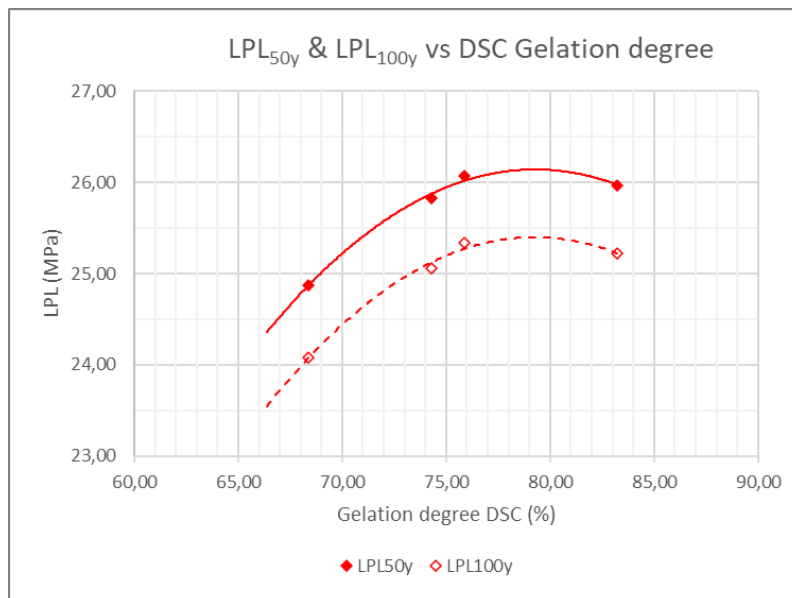


Figure 6: LPL vs. Gelation Degree by DSC.

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. The findings of this study can be summarized as follows:

1. An adequate Calcium based stabilized recipe made with PVC $\pm$ 67 k-value, processed with conical or parallel twin screw extruder, could achieve a LPL100y $\geq$ 25 MPa when the melt temperature is at least of 185°C (Figure 4).
2. A 'B-onset' temperature $\pm$ 180°C is required to achieve a the MRS 250 classification but, to achieve a LPL100y prediction above 25 MPa a 'B-onset' temperature of $\geq$ 185°C is needed (Figure 5).
3. DSC gelation degree of 75% is the minimum needed to achieve a LPL100y 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 (Figure 6).

ACKNOWLEDGMENTS

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REFERENCES

- [1] Pimentel Real LE, Joao IM, Pimenta SI and Diogo HP, 2018, Polymer Testing, 70, 481.
- [2] Benjamin P., 1980, Journal Vinyl Technology, 2, 254, 1980.
- [3] Lahoz J., 2022, Proc. PVC4Pipes Conference, Bologna, Italy.
- [4] Lahoz J., 2023, Proc. 21st Plastic Pipes Conference, Lake Buena Vista, FL, USA.

ANNEX A

TABLE A1

EXTRUSION TRIAL 26/02/2020 (PLA0031/20)

PVC-U DN140x5.4mm PN10		Time/Test Reference				
Cincinnati Milacron CM80 SC		9:12	10:54	12:40	14:03	15:37
Parameter	Unit	05 (175°C)	06 (180°C)	07 (185°C)	08 (190°C)	09 (195°C)
Dosing screw speed	rpm	18.6	21.1	23.5	26.4	29.2
Screw speed	rpm	8.1	8.1	9.0	9.8	11.1
Charge	%	73	81	76	77	74
Vacuum	bar	-0.9	-0.9	-0.9	-0.9	-0.9
Zone 1 Extruder	°C	150	148	175	185	183
Zone 2 Extruder	°C	165	164	166	175	186
Zone 3 Extruder	°C	160	159	163	170	179
Zone 4 Extruder	°C	155	155	162	166	169
Zone 5 Extruder	°C	152	152	153	161	162
T melt	°C	174/6	180/1	184	190/1	194/5
P melt	bar	178/9	203	189	199/202	189
Zone 1 Die-head	°C	161	162	168	164	165
Zone 2 Die-head	°C	176	175	178	179	179
Zone 3 Die-head	°C	190	190	190	190	189
Zone 4 Die-head	°C	180	180	186	181	183
Zone 5 Die-head	°C	199	196	198	194	200
Zone 6 Die-head	°C	202	196	205	200	207
Pipe-meter-weight	kg/m	3.310	3.298	3.334	3.322	3.322
Output	kg/h	160,8	184.0	202.0	227,2	249,1
Line speed	m/min	0.81	0.93	1.01	1.14	1.25

TABLE A2
EXTRUSION TRIAL 16/05/2023 (PLA0090/23)

PVC-U DN140x5.4mm PN10		Time/Test Reference				
Krauss Maffei KMD 2 130 26D				12:20	16:50	18:30
Parameter	Unit			03b (185°C)	04b (190°C)	05b (195°C)
Dosing screw speed	rpm			35,0	38,6	46,9
Screw speed	rpm			17,3	17,3	21.0
Charge	%			68	63	65
Vacuum	bar			-0.9	-0.9	-0.9
Zone 1 Extruder	°C			163	185	197
Zone 2 Extruder	°C			160	185	194
Zone 3 Extruder	°C			160	185	191
Zone 4 Extruder	°C			160	179	188
Zone 5 Extruder	°C			160	173	175
Zone 6 Extruder	°C			155	163	165
Die-Head Adaptor	°C			155	160	160
T melt	°C			188	194	197
P melt	bar			221	195	198
Zone 1 Die-head	°C			165	165	165
Zone 2 Die-head	°C			175	176	177
Zone 3 Die-head	°C			192	192	192
Zone 4 Die-head	°C			184	184	184
Zone 5 Die-head	°C			200	200	200
Zone 6 Die-head	°C			205	205	205
Pipe-meter-weight	kg/m			3,229	3,248	3,255
Output	kg/h			252,6	279,2	339,2
Line speed	m/min			1.30	1.43	1.74

