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Ageing life of PA11 design for offshore flexible pipe

Kuang Ye *

***Corresponding Author:** Kuang Ye* Neptune Offshore Engineering Development Co. Ltd, China, +86 13752273060.

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Abstract:

Flexible pipes are now widely used in oil and gas industry in both riser and flowline applications. Long term performance of the pipes, especially internal pressure sheath ageing is concerned by people. PA material is usually used as an internal pressure sheath for flexible pipe and hydrolysis of PA material is limited when using. This report analysis different ageing critical using for PA ageing in flexible pipe design and give some recommendation for following design.

Keywords: PA11, ageing life, Flexible pipes, design.

Introduction

Flexible pipes are now widely used in oil and gas industry in both riser and flowline applications. Long term performance of the pipes, especially internal pressure sheath ageing is concerned by people. PA11 as one kind of material [1] have been used in flexible pipe for more than 50 years and hydrolysis of PA material is most concerned limited when it used at high temperatures [2-5].

1. Experiment Instrument and Fluid

Stainless steel vessels are used with internal volume is 1.5L.

Figure 2-1 Stainless vessel

The cover is equipped with two faucets, one enabling the introduction of gas at the bottom of the liquid by means of an open tube, the other allowing the purge of gas.

The cover is equipped with two faucets, one enabling the introduction of gas at the bottom of the liquid by means of an open tube, the other allowing the purge of gas.

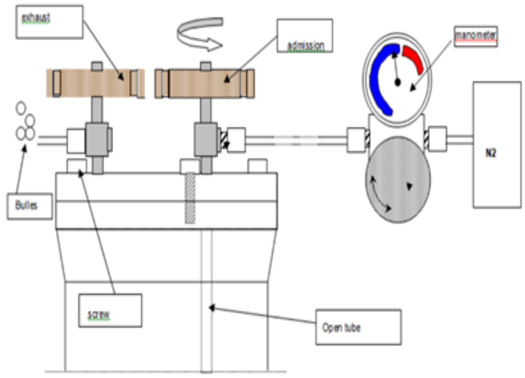


Figure 2-2 Experiment chart

Water： VOLVIC® water is used for its stable pH and low ion concentration.

Nitrogen: quality U from Air Liquide is used for oxygen removal. The concentration of O2 is very low (<1ppb).

The measurement of O2 concentration after 3 hours of bubbling proved that the concentration of O2 in water is less than 2ppm.

CO2: withless than 2ppm oxygen is used (spec API TR17<10ppm)[6]

2. Experiment Process

2.1 Oxygen Removal

For this type of ageing test, it is necessary to minimize the presence of oxygen in the water. This is done by bubbling of nitrogen (nitrogen type U from Air Liquide). Nitrogen is injected by an open tube in the bottom of the cell from the admission valve. It goes out by the exhaust valve in the top of the cell. A plastic tube connected to the exhaust valve is put in a vessel containing water in order to check visually the nitrogen flow. Nitrogen flow is stopped by closing the output valve first and then the input valve.

Nitrogen is bubbled during at least 3 hours.

15 vessels can be treated simultaneously.

2.2 Addition of CO2

A given quantity of CO2 is introduced in the vessels the same way as nitrogen.

The pH has been calculated from CO2 pressure, temperature and water composition based on a software called MICO (from Total).

For Volvic® water, the CO2 pressure [7] to be added is

24 bars for pH4

2 bars for pH5

The procedure for CO2 addition is as follows:

The exhaust valve is closed.

CO2 is introduced by the open tube until the target pressure .

The admission valve is closed.

After 10 minutes, the pressure is adjusted to get the target pressure. This is done 3 times.

The vessels are then installed in the oven for the aging phase.

2.3 Water Renewal

Due to the low solubility of plasticizer in water, it is necessary to change periodically water and to wash the samples and vessels to avoid an increase of pH and decrease of plasticizer desorption.

-Vessels are cooled down for at least 2h before handling. When at room temperature

- empty the vessel

- thoroughly rinse samples and vessel with water to remove all traces of plasticizer.

Figure 2-3 the ageing vessels

Do not use cleaning product (detergent or other) as it could result in pollution of the vessel.

A sponge or any other means of cleaning can be used to clean the internal surface of the vessels.

Before placing the vessel back in the oven to continue the ageing, procedures for filling , oxygen removal and CO2 addition occur again.

The renewal of the water is achieved at least at each time in the following table.

The times highlighted in yellow correspond to suggestions for sampling.

After ageing, elongation at break and CIV test was examined both and test method refers to ASTM D638 and API 17TR2.

Results & Discussions

Ageing material is cut from flexible pipe made by Neptune.

The ageing tests are carried out at 150°c, 140°c, 130°c, and 120°c respectively and test result as follows:

2.4 Sampling time

The sampling time at different test temperature are listed in Table 3-1 to Table 3-4

Table 2-1 Dates of sampling at 150°c

Temps (h)	Operation
0	Water injection
24	Water renewal
48	Water renewal + sampling
72	Water renewal + sampling
118	Water renewal + sampling
230	Water renewal + sampling

Table 2-2 Dates of sampling at 140°c

Temps (h)	Operation
0	Water injection
24	Water renewal
48	Water renewal
72	Water renewal + sampling
120	Water renewal
232	Water renewal + sampling
368	Water renewal + sampling
528	Water renewal + sampling

Table 2-3 Dates of sampling at 130°c

Temps (h)	Operation
0	Water injection
24	Water renewal
48	Water renewal
72	Water renewal
120	Water renewal + sampling
472	Water renewal + sampling
752	Water renewal + sampling
1035	Water renewal + sampling
1509	Water renewal + sampling
2005	Water renewal + sampling

Table 2-4 Dates of sampling at 120°c

Temps (h)	Operation
0	Water injection
24	Water renewal
48	Water renewal
72	Water renewal
120	Water renewal
192	Water renewal + sampling
288	Water renewal
424	Water renewal + sampling
680	Water renewal + sampling
913	Water renewal + sampling
1529	Water renewal + sampling
2145	Water renewal + sampling
2837	Water renewal + sampling

2.5 Tensile properties

The change of ultimate strength during ageing tests at different temperature is shown in Table 2-5.

Table 2-5 Change of ultimate strength during ageing tests

Ageing temperature	**Temps(h)**	**Ultimate Strength （MPa）**					
		1	2	3	4	5	Average
150°C	0	58	51	47	50	48	51
	48	37	34	35	20	24	30
	72	29	22	36	31	34	30
	118	30	46	32	34	43	37
	230	49	48	48	48	32	45
140°C	0	58	51	47	50	48	51
	72	31	26	24	19		25
	232	26	26	23	29	29	27
	368	32	28	43	31	21	31
	528	37	43	43	26	45	39
130°C	0	58	51	47	50	48	51
	120	43	32	33	42		37
	472	24	27	22	28	28	26
	752	28	24	17	25	24	23
	1035	30	18	20	30	29	25
	1509	34	20	31	28	33	29
	2005	35	31	45	33	46	38
120°C	0	58	51	47	50	48	51
	192	21	27	21	39		27
	424	17	36	34	18	16	24
	680	21	19	25	25	25	23
	913	25	25	20	19	18	22
	1529	30	25	29	30		28
	2145	29	43	26	27	43	33
	2837	42.4	34.35	43.4	35.6	38.7	39

The change of strain at yield during ageing tests at different temperature is shown in Table 2-6.

Table 2-6 Change of strain at yield during ageing tests

Ageing temperature	Temps(h)	Yield Strain （%）					
		1	2	3	4	5	Average
150°C	0	29	29	29	30	29	29
	48	41	41	40	40	40	40
	72	43	42	43	43	42	43
	118	46	46	46	46	47	46
	230	49	49	48	49	49	49
140°C	0	29	29	29	30	29	29
	72	35	36	36	35	-	36
	232	41	43	41	42	43	42
	368	43	44	44	44	45	44
	528	43	44	46	46	46	45
130°C	0	29	29	29	30	29	29
	120	34	34	34	33	-	34
	472	41	41	40	42	40	41
	752	45	43	43	44	43	43
	1035	45	45	45	44	46	45
	1509	46	46	47	47	47	46
	2005	49	49	49	50	48	49
120°C	0	29	29	29	30	29	29
	192	32	32	32	31	-	32
	424	38	38	38	38	38	38
	680	40	41	40	40	41	40
	913	43	43	43	43	43	43
	1529	44	45	44	44	-	44
	2145	44	-	43	42	44	43
	2837	-	-	-	-	-	-

The change of yield strength during ageing tests at different temperature is shown in Table 2-7.

Table 2-7 Change of yield strength during ageing tests

Ageing temperature	Temps(h)	Yield Strength （MPa）					
		1	2	3	4	5	Average
150℃	0	29	29	29	30	29	29
	48	41	41	40	40	40	40
	72	43	42	43	43	42	43
	118	46	46	46	46	47	46
	230	49	49	48	49	49	49
140℃	0	29	29	29	30	29	29
	72	35	36	36	35	-	36
	232	41	43	41	42	43	42
	368	43	44	44	44	45	44
	528	43	44	46	46	46	45
130℃	0	29	29	29	30	29	29
	120	34	34	34	33	-	34
	472	41	41	40	42	40	41
	752	45	43	43	44	43	43
	1035	45	45	45	44	46	45
	1509	46	46	47	47	47	46
	2005	49	49	49	50	48	49
120℃	0	29	29	29	30	29	29
	192	32	32	32	31	-	32
	424	38	38	38	38	38	38
	680	40	41	40	40	41	40
	913	43	43	43	43	43	43
	1529	44	45	44	44	-	44
	2145	44	-	43	42	44	43
	2837	-	-	-	-	-	-

The change of strain at break during ageing tests at different temperature is shown in Table 2-8. Graph for strain at break is illustrated in Fig. 2-4.

Table 2-8 Change of strain at break

Ageing temperature	Temps(h)	Elongation at break (%)					
		1	2	3	4	5	Average
150°C	0	350	324	299	312	307	318
	48	349	194	273	140	73	206
	72	53	33	60	70	61	55
	118	53	23	74	51	35	47
	230	21	34	22	28	57	33
140°C	0	350	324	299	312	307	318
	72	159	276	187	114	-	184
	232	72	75	60	79	51	67
	368	50	47	27	48	76	50
	528	40	26	35	61	31	38
130°C	0	350	324	299	312	307	318
	120	328	199	244	281	-	263
	472	96	149	95	96	73	102
	752	92	73	112	65	64	81
	1035	63	44	65	55	49	55
	1509	42	66	39	48	50	49
	2005	43	43	34	42	30	39
120°C	0	350	324	299	312	307	318
	192	230	124	110	282	-	187
	424	292	56	308	130	33	164
	680	214	55	101	45	134	110
	913	70	147	64	66	75	84
	1529	61	52	55	50	-	54
	2145	50	23	49	48	28	40
	2837	20	10	17	10	12	14

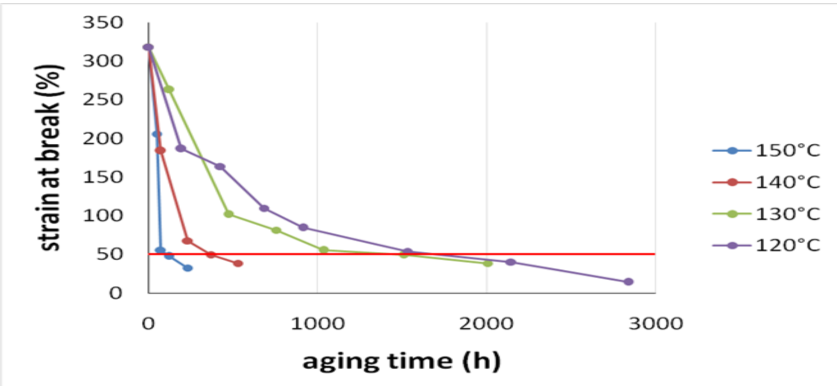
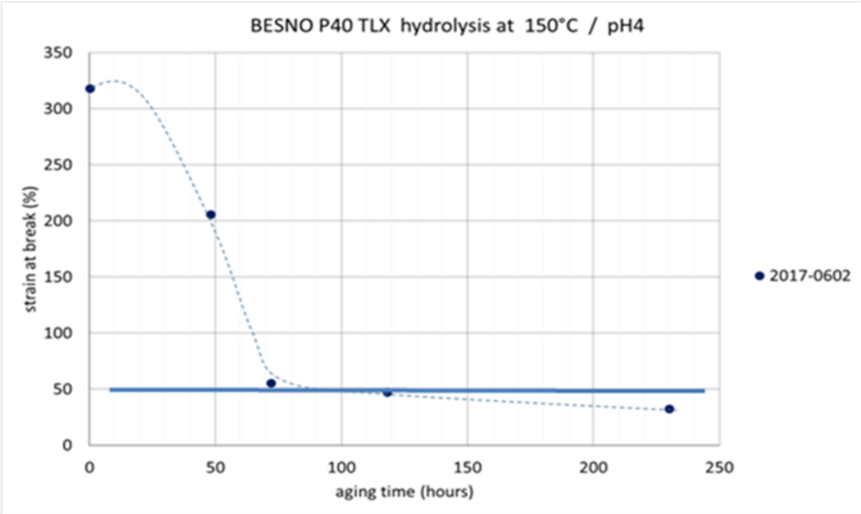
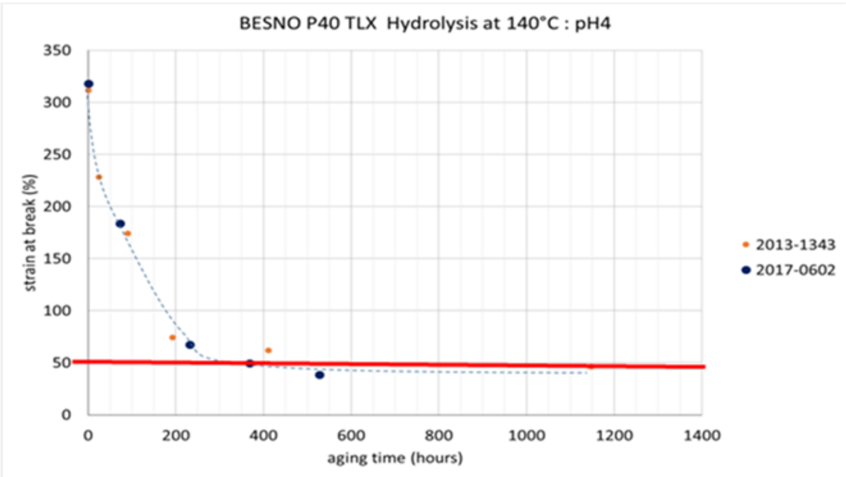


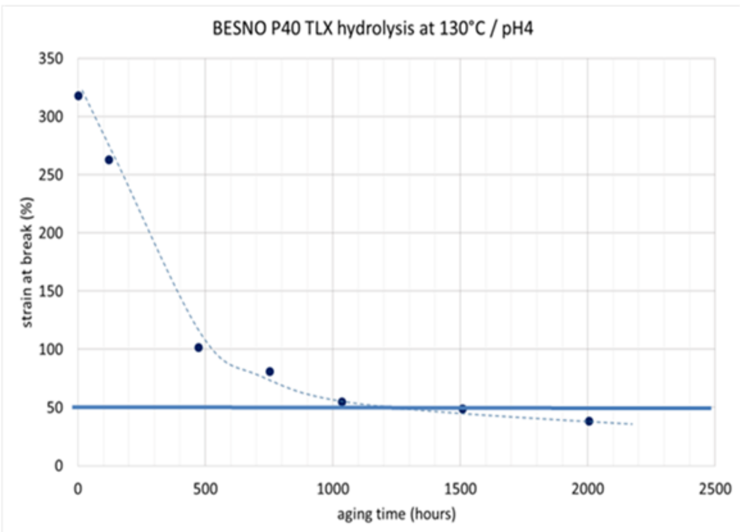
Fig.2-4-1 Graph for strain at break



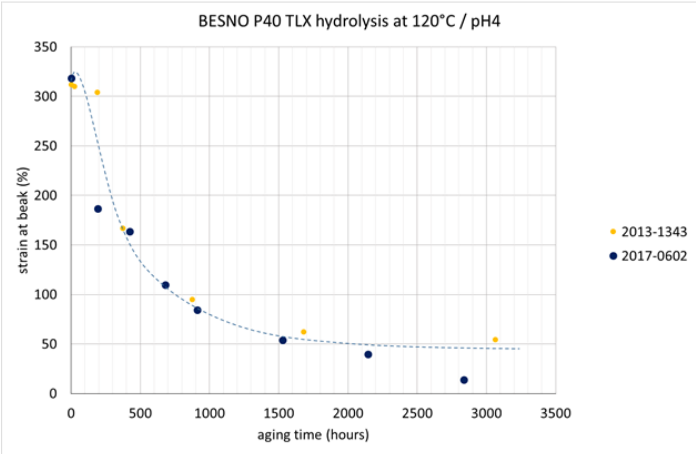
(a) Determination of life time at 150°C



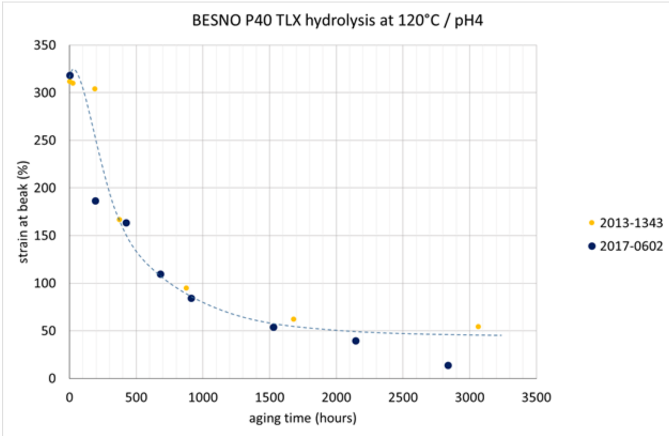
(b) Determination of life time at 140°C



(c) Determination of life time at 130°C



(c) Determination of life time at 130°C



(d) Determination of life time at 120°C

Fig. 2-4-2 Determination of life time at different ageing temperature

2.6 CIV results

The change of extractable during ageing test can be seen in Table 2-9 and Fig. 2-5. Content of extractable substance, which is plasticizer in PA11, drops down at the beginning of the tests due to the water renewal procedure.

In Table 2-9, viscosity 1 and viscosity 1 are inherent viscosity, which are measured with the concentration of m-cresol being 0.5% g/g. According to the API procedure, the concentration should be 0.5% g/cm3. The density of m-cresol being 1.034, the standard test method leads to a concentration of 0.48 g/g. Therefore, the CIV is calculated using the following equation.

$$CIV=\frac{Viscosity1+Viscosity2}{2\times 1.034\times(1-Extractable)}$$

Ageing temperature	Temps(h)	Extractable (%)	Viscosity 1	Viscosity 2	CIV
150°C	0	13.0	1.92	2.01	2.18
	48	6.6	1.15	1.17	1.20
	72	4.3	1.11	1.12	1.13
	118	4.0	1.06	1.08	1.08
	230	3.1	1.07	1.07	1.07
140°C	0	13	1.92	2.01	2.18
	72	5.5	1.27	1.29	1.31
	232	3.4	1.06	1.07	1.07
	368	3.2	1.07	1.08	1.07
	528	3.1	1.07	1.09	1.08
130°C	0	13	1.92	2.01	2.18
	120	4.4	1.37	1.38	1.39
	472	3.36	1.1	1.12	1.11
	752	3.28	1.12	1.13	1.12
	1035	2.91	1.1	1.1	1.1
	1509	3.15	1.12	1.13	1.12
	2005	2.64	1.12	1.14	1.12
120°C	0	13.00	1.92	2.01	2.18
	192	11.02	1.47	1.48	1.60
	424	3.51	1.24	1.25	1.25
	680	3.39	1.15	1.16	1.16
	913	2.77	1.13	1.13	1.12
	1529	2.62	1.11	1.11	1.10
	2145	2.47	1.10	1.11	1.10
	2837	3.47	1.08	1.08	1.08

Table 2-9 Change of extractable

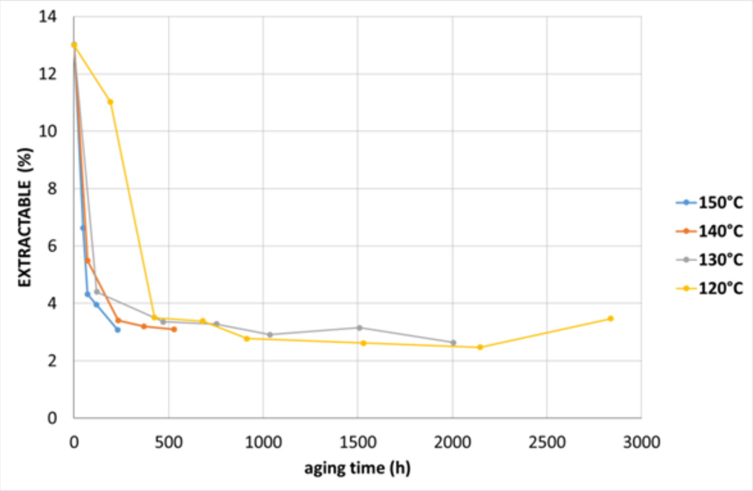


Fig. 2-5 Change of extractable

The CIV value drops with the elapse of ageing time. For all temperatures, CIV reaches a plateau (shown in Fig. 2-6. With the CIV=1.2 dl/g as end of life, the lifetime at 150°c , 140° c , 130°c and 120°c are 50h, 100h, 300h and 550h respectively.

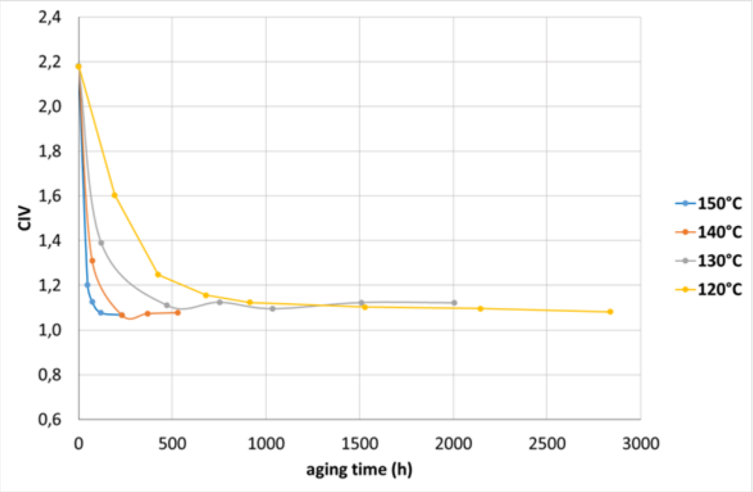


Fig. 2-6 Change of CIV

2.7 Aging lifetime

To predict the behaviour at service temperature, two end of life criteria were considered :

(1) End of life = the time when CIV is 1.2 dl/g

(2) End of life = the time when strain at break is 50%

The end of life under the two criteria can be referred as above .

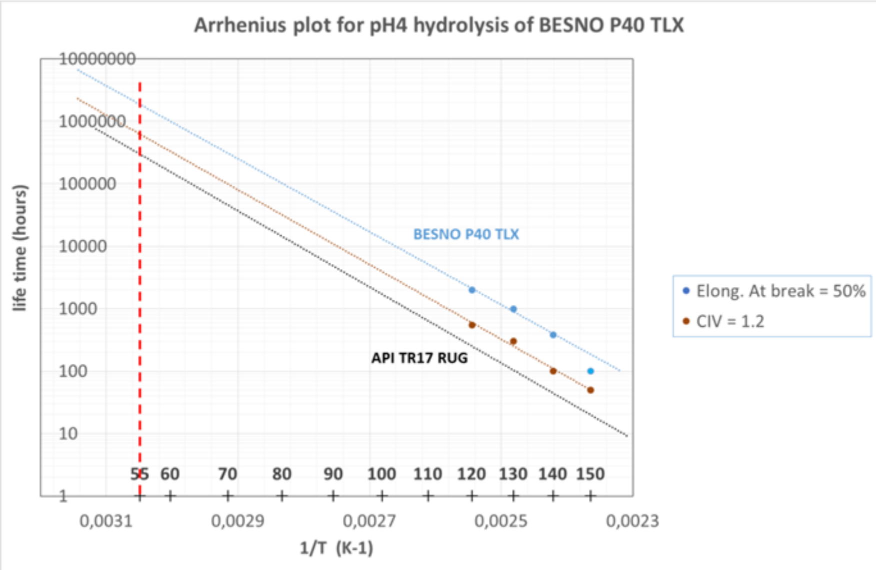


Fig. 2-6 Plot life of PA11 ageing

Plot the lifetime curve versus ageing temperature and fit their relationship, demonstrated in Fig.2-7. The Arrhenius equation under the above two criteria are as follows :

(1) CIV = 1.2 dl/g

$$t(h) = 2.838 \times 10^{-12} \times \exp\left(\frac{1.346 \times 10^{4}}{T(℃)+273}\right)$$

(2) Strain at break = 50%

$$t(h) = 3.615 \times 10^{-13} \times \exp\left(\frac{1.377 \times 10^{4}}{T(℃)+273}\right)$$

Conclusion:

CIV as ageing critical of PA11 using for design is more conservation than elongation at break for flexible pipe and we can select ageing critical between CIV and elongation at break to predict the ageing life of the material.

Acknowledgements:

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