Synthesis of Material Specifications for 100 Year Service Life of Corrugated High Density Polyethylene Pipe

Research Report Number: FL/DOT/SMO/04-474

Prepared for:

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July 8, 2004

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INTRODUCTION

Due to the long required time to fulfill the entire test protocols defined in the full specification, the Department is creating an interim materials specification, discussed below. The interim specification is developed based on technical information and specifications of polyethylene used in other applications, such as gas pipes and geomembranes.

For products made from high-density polyethylene (HDPE), their long-term performance is basically governed by two mechanisms: stress crack resistance (SCR) and oxidation resistance (OR). The relationship between these two mechanisms with respect to the service life of the product is illustrated in the flow chart in Figure 1. For scenario (1), the stress cracking takes place before oxidation degradation is occurring in the pipe. For scenario (2), the failure of the pipe is governed by the oxidation degradation, which would lead to multiple brittle cracking in the pipe. The SCR and OR can be evaluated separately or together. When both properties are tested simultaneously, it is believed that the synergistic effect of stress and oxidation has greater challenging to the material. The tensile stress enlarges micro-defects in the material and provides greater opportunity for oxygen to diffuse into the material and react with the material.

The first draft interim specification for 100-year design life of corrugated HDPE pipes was proposed in September 2003, as shown in Table 1. The specification consists of two parts addressing the SCR and OR of the pipe. For the SCR evaluation, the junction and longitudinal profiles of the pipe are tested under a tensile stress of 250 psi in water at 80^{o}C for duration of 1260 hours. The test duration of 1260 hr was based on the Popelar's shift factor and 100 years service life at 20^{o}C. For oxidation degradation, the initial oxidative induction time (OIT) of 25 minutes was required as well as the OIT retained value after water incubation at 85^{o}C after 90 days.

In the last 6 months, new technical information has been reviewed, evaluated and discussed. The industry put forward the ISO 9080 standard “Plastics Piping and Ducting Systems – Determination of Long-Term Hydrostatic Strength of Thermoplastics Materials in Pipe Form by Extrapolation” in which a list of shift factors is provided to extrapolate the SCR data from different elevated testing temperatures to the site temperature. The shift factors in the standard are obtained based on the Arrhenius equation and should provide greater confidence in the extrapolation than the Popelar's shift factors.

For the OR of the pipe, the variety of antioxidant packages used in the corrugated PE pipes creates uncertainty in the interpretation of the OIT test results. It has found that the OIT depletion rate varies significantly among different antioxidant packages, particularly in the water environment. Unless the correlation between OIT and mechanical degradation is established, the OIT retained value of a water incubated pipe sample may give misleading information on the OR of the pipe. Figures 2 to 4 show the response of four material properties in 200 days of water incubation at a temperature of 85^{o}C for three different pipes. The mechanical properties and melt index value remain constant throughout the incubation period. Contrary, the OIT values in all three pipes decrease rapidly in the first 90 days and then level off at approximately 10% OIT retained value (2 to 4 minutes). The test data suggest that the onset of the oxidation does not occur in a water environment at this low level of OIT due to the low concentration of oxygen. Thus, the mechanical property together with OIT test should be used to determined the OR performance.

In this document, the revised interim specification is presented together with the supporting technical information. In addition, the full specification is developed based on the uncertainly of the interim specification.

REVISED INTERIM SPECIFICATION

Table 2 shows the details of the revised interim specification, which targets both SCR and OR using a single test.

Technical Information for Interim Specification Requirements

This section of the document describes the technical information that is used to determine the required values in the revised interim specification.

In the revised interim specification, the SCR and OR are evaluated simultaneously using a constant tensile stress test on pipe junctions and longitudinal profiles. The reaction rates are accelerated using water and elevated temperature so that a 100-year lifetime can be predicted within a reasonable time period. Since the current available data are insufficient to determine shift factors for the extrapolation, the shift factors in the ISO 9080 standard are applied to decide the testing duration at an elevated testing temperature.

Background of the shift factors in ISO 9080

The ISO 9080 standard describes the extrapolation procedure to predict the long-term hydrostatic strength of thermoplastic pipe. The standard addresses only the SCR behaviors of the thermoplastic pipes but not the oxidation mechanism of the polymer.

The shift factors (k_e) in ISO 9080 are calculated using Equation (1) with activation energy (E_a) of 110 kJ/mol and test temperature (T_{max}) at 110°C. (Note that the shift factor changes with the test temperature (T_{max}); a high T_{max} yields a low shift factor with the same ΔT value.) Table 3 shows the shift factors obtained by Equation (1) as well as those listed in the ISO 9080.

$$k_e = exp\left[\frac{E_\sigma}{R} * \frac{\Delta T}{T_{max}\left(T_{max} - \Delta T\right)}\right] \qquad (1)$$

where:

where:	k_e	=	shift factor
	E_σ	=	activation energy of stress cracking
	R	=	gas constant
	ΔT	=	Temperature difference between the testing temperature and site temperature
	T_{max}	=	elevated test temperature

The standard allows the shift factor to be applied to test temperature lower than 110°C (for example shifting 60°C data to 20°C, ΔT of 40°C, using a shift factor value of 50). For ΔT greater than 50°C, the shift factor is obtained by plotting k_e versus ΔT, as shown in Figure 5. Three sets of data are presented based on Minimum, Maximum and Average ΔT within each range. The response curves are expressed by Equations (2) to (4).

Min. Temp $$k_e = 0.8842e^{0.1008\Delta T} \qquad (2)$$

Max. Temp $$k_e = 0.6998e^{0.0897\Delta T} \qquad (3)$$

Ave. Temp $$k_e = 0.7733e^{0.0953\Delta T} \qquad (4)$$

Table 4 shows the calculated k_e values at ΔT of 70°C (90°C testing temperature and 20°C site temperature). For 100 years service life, the duration at 90°C test temperature ranges from

36 to 98 days. If one changes the test temperature to 80°C (ΔT of 60°C), the test duration time would be from 98 to 240 days, as shown in Table 5.

Applying the shift factor in ISO 9080 for OR evaluation

In the ISO 9080 standard, it is assumed that pipes are following the scenario (1) path in Figure 1 with sufficient antioxidants to protect the polymer. However, the OR of corrugated HDPE pipes is unknown at this time and there is no field data to ensure t_{ch} is greater than100 years. Corrugated pipes that passed the SCR test requirement based on ISO 9080 shift factor (i.e., 155 days at 80°C or 60 days at 90°C), do not guaranty a 100-year OR property. The reason is that activation energies of the two mechanisms are not the same. The reaction mechanism with a lower activation energy requires a longer incubation time at the elevated temperature to validate the 100-year performance than that with a higher activation energy, as shown in Figure 6. Based on the published literature, Viebke et al (1994)[1] used water pressure test to assess the oxidation degradation of unstabilized HDPE pipes and found the activation energy for unstabilized pipes to be 75 kJ/mol. It is assumed that the oxidation degradation mechanism of stabilized and unstabilized PE pipe is the same, i.e., their activation energies are very similar; the only difference between them is the materials constant (A) in the Arrhenius equation, as shown in Equation (5).

$$R_t\left(\frac{1}{lifetime}\right) = A\,exp\left(\frac{-E_{OX}}{RT}\right) \tag{5}$$

where: R_t = rate of reaction
A = materials constant
E_{ox} = activation energy of oxidation
R = gas constant
T = temperature

The schematic diagram in Figure 7 illustrates the different predicted lifetime of stabilized and unstabilized PE with the same activation energy but different (A) values. The stabilized material has much longer lifetime than the unstabilized material.

Since the activation energies of stabilized corrugated PE pipes are unknown at this time, a conservative approach is to utilize the activation energy from the unstabilized material (75

[1] Viebke, J., Elble, E., Ifwarson, M. and Gedde, U.W. (1994), “Degradation of Unstabilized Medium-Density Polyethylene Pipes in Hot-Water Applications,” Polymer Engineering and Science, Vol. 34, No. 17, pp. 1354-1361

KJ/mol). The shift factor (k_{OR}) is determined using Equation (1) and testing temperature of 80^oC. The resulting shift factors (k_{OR}) are shown in Table 6. (Noted that the Viebke's aging study was performed at incubation of temperatures 90, 80, 70^oC.) The (k_{OR}) shift factors are relatively close to those obtained from ISO 9080 (k_{ISO}). At 80^oC incubation temperature, the required test durations are 155 days and 195 days for (k_{ISO}) and (k_{OR}), respectively. Although the (k_{OR}) gives a longer incubation time than the (k_{ISO}), the prediction is based on unstabilized resin. With additional of antioxidants, the required incubation duration should be shortened. The similarity between (k_{OR}) and (k_{ISO}) makes the combined test for both the stress cracking test and oxidation resistance test acceptable.

However, as indicated in Table 6, the shift factor increases exponentially with ΔT, so do the ratio ($\frac{k_{ISO}}{k_{OR}}$). At 80^oC testing temperature, the k ratio is 1.2, while at 90^oC the ratio changes to 1.6. In addition, the uncertainty of the extrapolation increases with increasing ΔT. Therefore, at a higher testing temperature, such as 90^oC, the shift factor and target length of test should be according to the (k_{OR}) instead of (k_{ISO}) to increase the confidence of the test. This results in a target test length of approximately 96 days rather than 60 days using (k_{ISO}).

Validating the Antioxidant Level after the SCR Test

As demonstrated in the earlier part of this document, the mechanical properties remain unchanged when the OIT values of 2 to 4 minutes is measured in the incubated pipe samples. To assure the initial OIT value of 25 minutes is sufficient to protect the HDPE pipe from oxidation; the OIT values of the junction specimens after the SCR test (either 155 days at 80^oC or 90 days at 90^oC) should have an average value of 3 ($\pm$1) minutes.

Evaluate the applied stress

Regarding the applied stress that should be used for the elevated temperature SCR tests, the ISO 9080 standard does not provide shift factor for the stress. The appropriate question to predict the applied stress at different test temperature is the rate process method (ASTM D 2837), as shown in Equation (6). However there are no data currently available to determine the three constants in the Equation (6). The Popelar's stress shift factor is the only equation that can offer the temperature and stress relationship, as shown in Table 7. In the full specification, we should be able to obtain a better stress target at different test temperatures.

$$\log t = A + \frac{B}{T} + \frac{C * \log \sigma}{T} \qquad (6)$$

where: t = failure time
σ = applied stress
T = test temperature
$A, B\ and\ C$ = constants

Uncertainties of the Interim Specification

The approach of the interim specification is based on the activation energy values of PE pressure pipes on both SCR and OR mechanisms. The PE pressure pipes are made from resins in the medium density range while the corrugated PE drainage pipes are used resins in the high density range. Furthermore, the SCR test of pressure pipes is performed using smooth pipe with no stress concentration along the test specimen; in contrast, the SCR test specimens of corrugated pipe consist of junction or longitudinal profile, which is a stress raiser. The long-term specification should provide data to confirm the applicability of the ISO shift factors to the corrugated pipes.

Regarding oxidation degradation, a conservative activation energy value (75 KJ/mol) based on unstabilized PE is used in the interim specification. Note that the 75 KJ/mol was obtained using water/air pressure test on smooth HDPE pipes. The oxidation process, including the depletion of antioxidant and degradation of polymer, in water environment is not well defined. It is known that the antioxidant depletion proceeds at a faster rate in water than in air due to leaching and hydrolytic reactions. On the other hand, the oxidation degradation of polymer would be slower in water than in air due to lower oxygen content in water.

The lack of information in the oxidation resistance of corrugated PE materials, some uncertainties in the interim specification are inevitable.

FULL SPECIFICATION IN STRESS CRACK RESISTANCE AND OXIDATION RESISTANCE

Table 7 shows the details of the full specification, which evaluates the SCR and OR separately.

Technical Information for Full Specification Requirements

This section of the document describes the technical information that is used to determine the required parameters for the full specification.

Stress Crack Resistance

The ISO 9080 standard and ASTM D 2837 are good technical resources in developing a test protocol for the prediction of the SCR of corrugate HDPE pipes. However, the applicability of shift factors to corrugate pipe should be validated as part of the full specification. The validation should be carried out using the SCR junction test on number of pipes with different diameters and resins. It is proposed to evaluate two pipes with different diameters for each processing setup. Three different test temperatures and three different applied stresses should be evaluated. Thus, each pipe generates 45 data points, which are shifted to a lower temperature, 20^{o}C, using the rate process method (ASTM D2837), and then used to obtain the temperature and stress shift factors. The shift factors should be independent of the material and should be similar to those listed in the ISO 9080 standard. Once the temperature shift factors are confirmed, a pipe junction test at a single stress level and test temperature can be implemented as part of the qualification testing for other pipe with different diameters.

Oxidation Resistance

In the interim specification, both SCR and OR are evaluated simultaneously using a single test. The activation energy of the OR is based on the unstabilized resin. There is no information regarding the oxidation degradation of the stabilized corrugated HDPE pipes in water. Long-term oxidation resistance should be performed on different antioxidant package to ensure the 100 years of OR of the pipe. If the predicted lifetime of oxidation degradation (t_{ch}) is longer than the predicted lifetime of stress cracking (t_{σ}), then a single combined test for SCR and OR can be adopted in the qualification testing.

The OR full specification is required to be performed on each antioxidant package. The incubation environment is water at three different temperatures. The depletion of antioxidants and tensile property are measured at different interval times to monitor changing of the pipe. The test can be stopped when tensile elongation decreases to 20% of its original value.

Table 1 – First Draft Interim Specification for 100-year Design Life of Corrugated HDPE Pipes (Dated September 2003)

Pipe Location	Test Method	Test Conditions	Requirement
Part I – Stress Crack Properties of Pipe			
Pipe Liner	*FM 5-572, Procedure A	• 10% Igepal solution at 50°C • 600 psi applied stress • 5 replicates	Will be based on the recommendation from the NCHRP 4-26 (due December 2003)
Junction	FM 5-572, Procedure B	• 80°C water, • 250 psi applied stress • 5 replicates	4 out of 5 test specimen ≥ 1260 hr
Longitudinal Profile	FM 5-572, Procedure C	• 80°C water • applied stress see Eq. (3) and (4) in test method • 5 replicates	4 out of 5 test specimen ≥ 1260 hr
Part II – Oxidation Resistance of Pipe			
Liner and/or Crown	OIT test (ASTM D 3895)	• 200°C test temperature • 2 replicates	25 min
Liner and/or Crown	Incubation test (FM 5-574) and OIT test (ASTM D 3895)	Incubation in 85°C forced air for 90 days* 2 replicates of OIT test on one incubated pipe sample	Minimum OIT percent retained ≥ 60%**

*FM = Florida Method of Test

** The test condition will be changed to water at 85°C with new percent retained value in December, 2003

Table 2- Interim Specification for 100-year Design Life of Corrugated HDPE Pipes

Pipe Component	Test Method	Test Conditions	Requirement
Stress Crack Resistance and Oxidation Resistance Properties - Option 1			
Liner and/or Crown	OIT test (ASTM D 3895)	• 2 replicates	25 min
Pipe Liner	FM 5-572, Procedure A	• 10% Igepal solution at 50°C • 600 psi applied stress	As per Final Report for NCHRP Project No. 4-26
Pipe Corrugation (molded plaque)	ASTM D 2136	• 10% Igepal solution at 50°C • 600 psi applied stress	As per Final Report for NCHRP Project No. 4-26
Junction	FM 5-572, Procedure B	• 80°C water, • 250 psi applied stress	95% statistical confidence ≥ 155 days (3720 hr)
Longitudinal Profile	FM 5-572, Procedure C	• 80°C water • Applied stress per Eqs. (3) and (4) in test method	95% statistical confidence ≥ 155 days (3720 hr)
SCR Junction specimens	OIT test (ASTM D 3895)	• After 155 days at 80°C 3 data points (each data point is average of 2 replicates from each SCR specimen)	Minimum average of the 3 data points shall be 2 minutes

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Table 1 – Cont.

Pipe Component	Test Method	Test Conditions	Requirement
Stress Crack Resistance and Oxidation Resistance Properties - Option 2			
Liner and/or Crown	OIT test (ASTM D 3895)	• 2 replicates	25 min
Pipe Liner	FM 5-572, Procedure A	• 10% Igepal solution at 50°C • 600 psi applied stress	As per Final Report for NCHRP Project No. 4-26
Pipe Corrugation (molded plaque)	ASTM D 2136	• 10% Igepal solution at 50°C • 600 psi applied stress	As per Final Report for NCHRP Project No. 4-26
Junction	FM 5-572, Procedure B	• 90°C water, • 220 psi applied stress	95% statistical confidence ≥ 90 days (2160 hr)
Longitudinal Profile	FM 5-572, Procedure C	• 90°C water • Applied stress per Eqs. (3) and (4) in test method	95% statistical confidence ≥ 90 days (2160 hr)
SCR Junction specimens	OIT test (ASTM D 3895)	• After 90 days at 90°C 3 data points (each data point is average of 2 replicates from each SCR specimen)	Minimum average of the 3 data points shall be 2 minutes

Notes: NCHRP=National Cooperative Highway Research Program
FM=Florida Method of Tests

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Table 3 – Relationship between ΔT and shift factor for polyolefins

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Calculated based on Arrhenius Equation				ISO listed Values	
Test Temperature T_{max} (^{o}C)	ΔT (^{o}C)	Extrapolate T (^{o}C)	**k_e (calc.)**	ΔT (^{o}C)	**k_e (ISO)**
110	10	100	**2.5**	≥ 10 and < 15	**2.5**
	15	95	**4.1**	≥ 15 and < 20	**4**
	20	90	**6.7**	≥ 20 and < 25	**6**
	25	85	**11.2**	≥ 25 and < 30	**12**
	30	80	**18.8**	≥ 30 and < 35	**18**
	35	75	**32.3**	≥ 35 and < 40	**30**
	40	70	**56.2**	≥ 40 and < 50	**50**
	45	65	**99.40**	≥ 50	**100**
	50	60	**178.92**		

Table 4 – Shift factor of ΔT = 70^{o}C

Equation	ΔT (^{o}C)	k_e	Time at 90^{o}C Test (day)
(1) – Min. Temp.	70	783	36
(2) – Max. Temp.	70	373	98
(3) – Ave. Temp.	70	610	60

Table 5 – Shift factor of ΔT = 60^{o}C

Equation	ΔT (^{o}C)	k_e	Time at 80^{o}C Test (day)
(1) – Min. Temp.	60	302	98
(2) – Max. Temp.	60	152	240
(3) – Ave. Temp.	60	235	155

Table 6 – shift factors of two different activation energies

$T_1\,(^oC)$	$T_1\,(K)$	$T_2\,(^oC)$	$T_2\,(K)$	$\Delta T(^oC)$	$k_{OR}\,(E_a=75)$	k_{ISO}
20	293	30	303	10	2.8	2
20	293	35	308	15	4.5	3
20	293	40	313	20	7.2	5
20	293	45	318	25	11	8
20	293	50	323	30	17	13
20	293	55	328	35	27	22
20	293	60	333	40	40	35
20	293	70	343	50	89	91
20	293	80	353	60	187	235
20	293	85	358	65	268	379
20	293	90	363	70	379	610

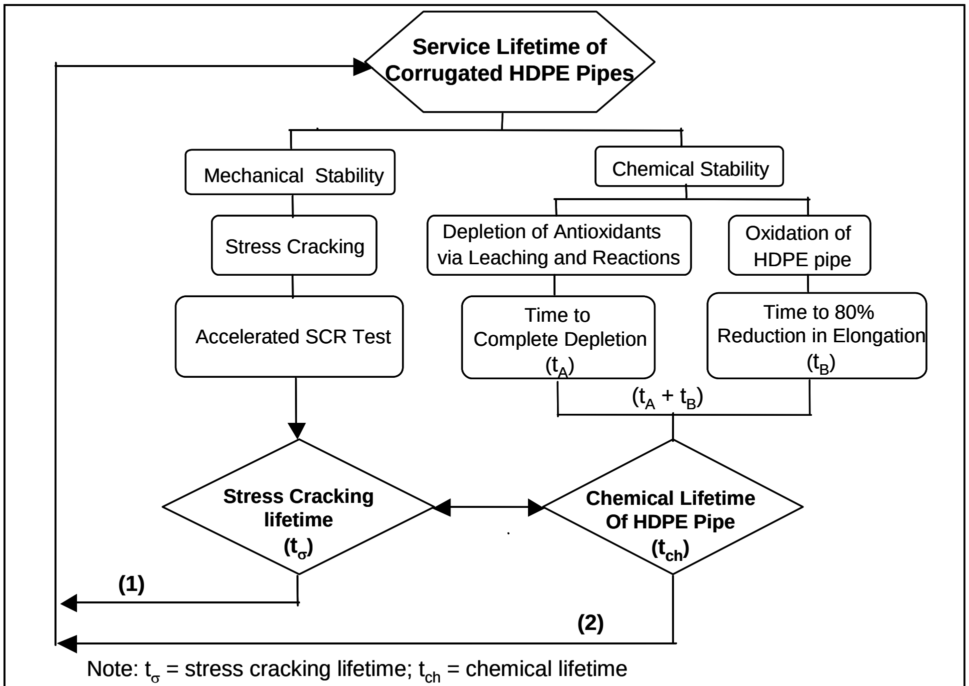
Table 7 – Shift factors for applied stress used in SCR tests

Test Temperature (^{o}C)	Reference Temperature (^{o}C)	ΔT (^{o}C)	Popelar's Shift Factor	Applied Stress (psi)
80	20	60	0.50	250
85	20	65	0.47	235
90	20	70	0.44	220

Table 8 – Full Specification for 100-year Design Life of Corrugated HDPE Pipes

Pipe Location	Test Method	Test Conditions	Requirement
Stress Crack Resistance of Pipes			
Pipe Liner	FM 5-572, Procedure A	• 10% Igepal solution at 50°C • 600 psi applied stress • 5 replicates	As per Final Report for NCHRP Project No. 4-26
Pipe Corrugation (molded plaque)	ASTM D 2136	• 10% Igepal solution at 50°C • 600 psi applied stress	Will be based on the recommendation from the NCHRP 4-26 (due August 2004)
Junction	FM 5-572, Procedure B and FM 5-573	• Test temperatures 70, 80 and 90°C • Three applied stresses,	• Calculate three constants • Failure time at 500 psi at 20°C ≥ 100 years (95% statistical confidence)
Longitudinal Profile	FM 5-572, Procedure C, and FM 5-573	• Four test temperatures, • Three applied stresses	• Calculate three constants • Failure time at 500 psi at 20°C ≥ 100 years (95% statistical confidence)
Oxidation Resistance of Pipes			
Liner and/or Crown	Incubation test (FM 5-574) and OIT test (ASTM D 3895) Tensile test (ASTM D638)	• Three incubation in water baths at 70, 80 and 90°C • Retrieve incubated sample every 3-month and perform -OIT test on the liner - tensile test on the liner	• Predict lifetime of antioxidant at 20°C • Predict lifetime of pipe liner at 20°C based on 20% break elongation retained (95% statistical confidence)

Notes: NCHRP=National Cooperative Highway Research Program
FM=Florida Method of Tests



(1) $t_{ch} \le 100$ years & $t_\sigma \ge 100$ years, lifetime is governed by chemical stability

(2) $t_\sigma \le 100$ years & $t_{ch} \ge 100$ years, lifetime is governed by stress cracking

Figure 1 – Service life in terms of stress cracking and oxidation

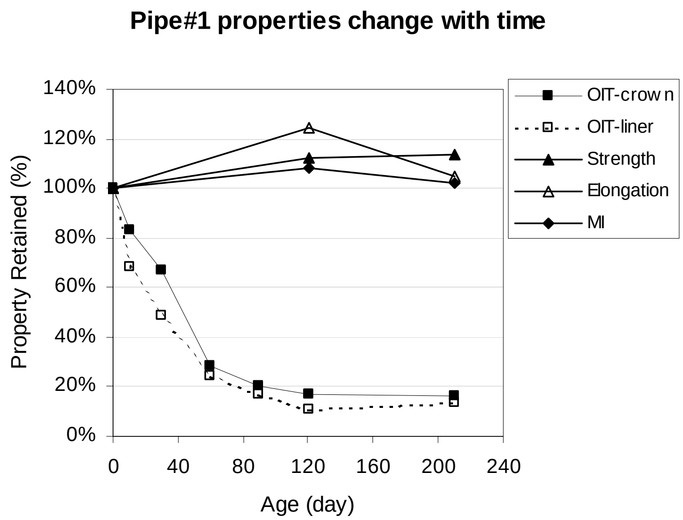


Figure 2 – Material properties change with incubation time at 85°C water for Pipe #1

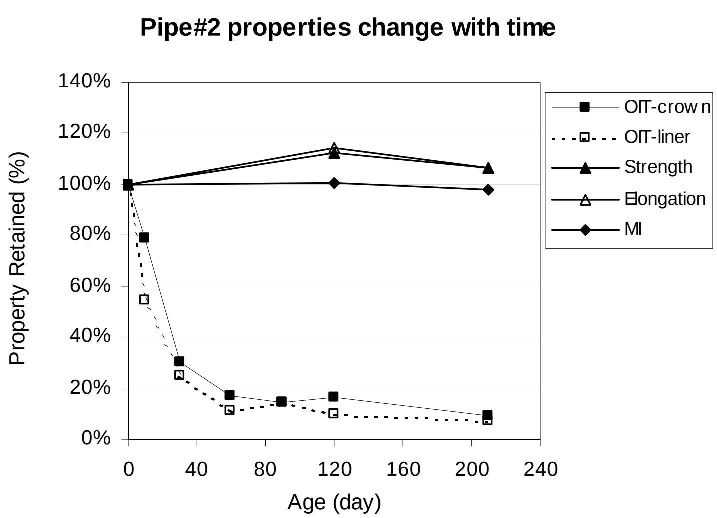


Figure 3 – Material properties change with incubation time at 85°C water for Pipe #2

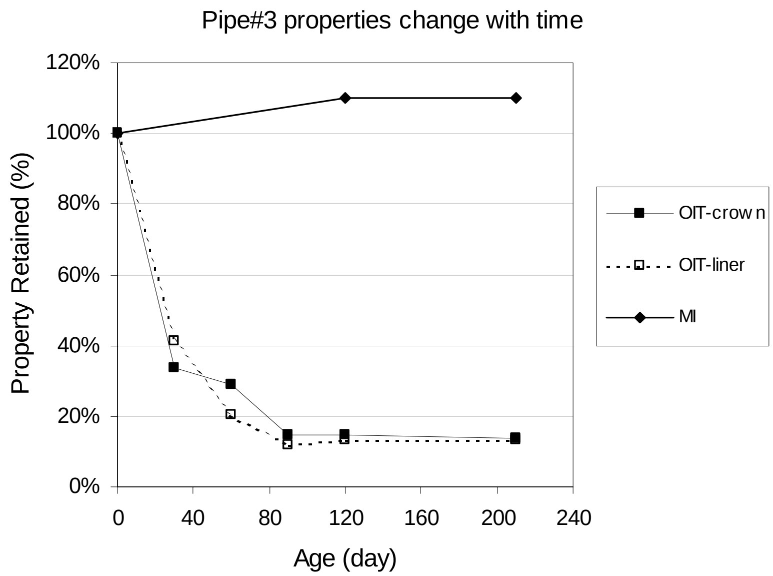


Figure 4 – Material properties change with incubation time at 85^{o}C water for Pipe #3

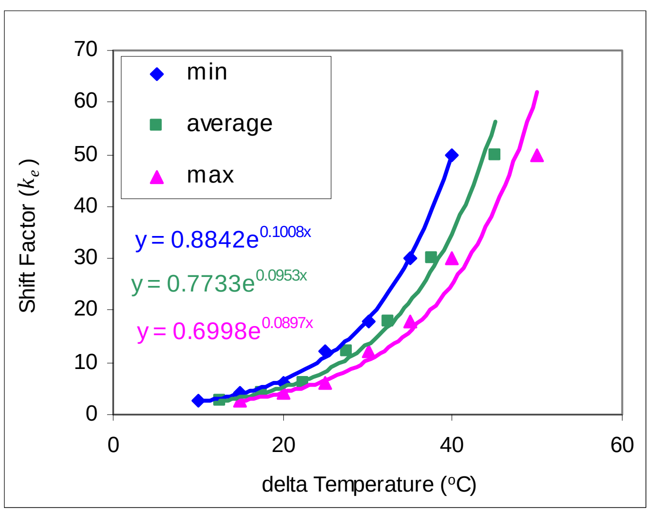


Figure 5 – k_e value versus ΔT plot

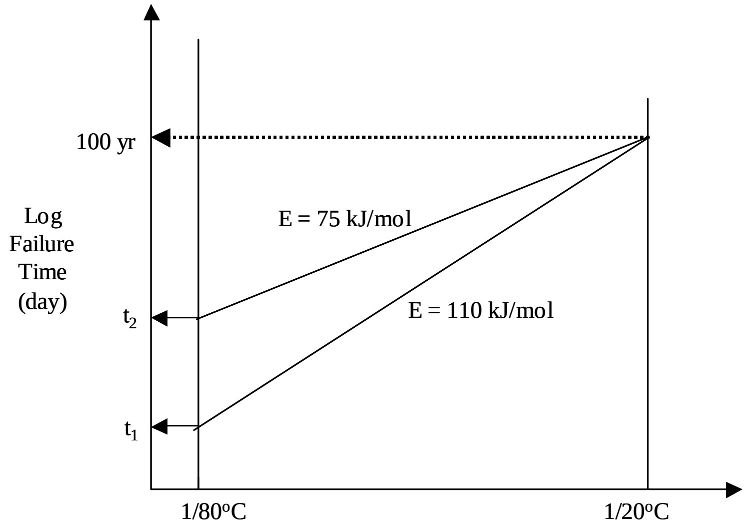


Figure 6 – Extrapolation from high temperature to low temperature with different activation energies

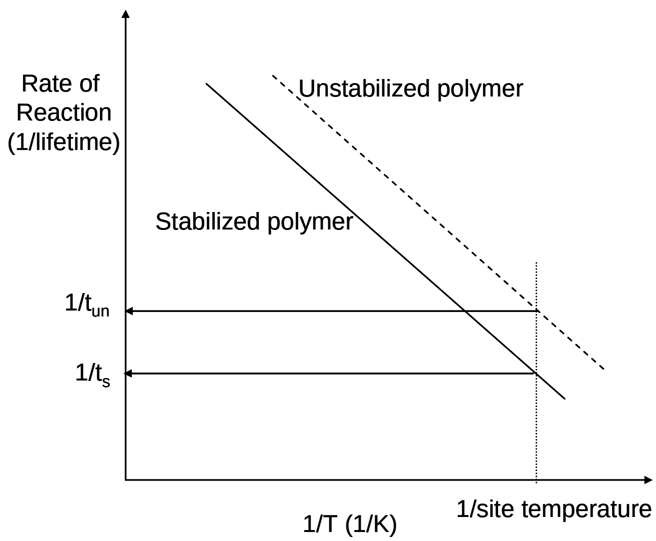


Figure 7 – Prediction lifetimes of materials with the same activation energy but different material constants