UNIVERSITÉ DE SHERBROOKE
Faculté de génie
Département de génie civil et de génie du bâtiment

PRÉDICTION DE LA DURÉE DE VIE DES TUYAUX EN POLYÉTHYLÈNE À HAUTE DENSITÉ (PEHD) FABRIQUÉS AVEC ET SANS RÉSINES RECYCLÉES UTILISÉES DANS LES INFRASTRUCTURES DE TRANSPORT

Thèse de doctorat
Spécialité : Génie civil

Nguyen Quoc Khanh

Sherbrooke (Québec) Canada

Janvier 2022

MEMBRES DU JURY

Prof. Brahim BENMOKRANE
Directeur de thèse

Prof. Mathieu ROBERT
Co-Directeur de thèse

Prof. Saïd Elkoun
Rapporteur et évaluateur

Dr. Patrice Cousin
Évaluateur

Prof. Pierre Ovlaque
Évaluateur externe

RÉSUMÉ

Le tuyau en polyéthylène à haute densité (PEHD) est un type de tuyau en plastique flexible, fabriqué à partir de PEHD thermoplastique et possédant un haut niveau d'imperméabilité, une forte liaison moléculaire, une résistance aux agents chimiques, un faible coût de maintenance et une procédure d'installation simple. Grâce à ces caractéristiques, les tuyaux de PEHD sont largement utilisés en génie civil et dans le bâtiment et les travaux publics (BTP). Ils sont souvent utilisés pour remplacer les canalisations principales en béton ou en acier vieillissantes. Cependant, le PEHD est sensible au vieillissement oxydatif. Par contre, les tuyaux de PEHD recyclé, et non vierge, est encore très limitée. Certaines craintes subsistent, qu'après plusieurs années de service, certaines dégradations, telles que l'apparition de fissures dues à une charge élevée ou une oxydation, puissent affecter ces tuyaux recyclés. Les conditions de service des tuyaux varient considérablement en fonction de l'emplacement géographique, des techniques d'installation, de la température et du type de sol environnant ainsi que de la charge de trafic. Le manque de compréhension des mécanismes de dégradation ainsi que d'information sur la capacité des tuyaux de plastique recyclé à offrir les mêmes performances que les tuyaux de résine vierge soulèvent de nombreuses questions pour les fabricants quant à leur utilisation à grande échelle. Ces lacunes soulèvent des questions sur leur durabilité et c'est dans cette optique que ce projet de thèse fut conduit.

L'objectif de cette thèse est d'évaluer la durabilité et la durée de vie des tuyaux en PEHD avec et sans résines recyclées en étudiant les propriétés physico-chimiques et mécaniques à court et long terme des tuyaux après une série de vieillissements accélérés. L'étude des performances a permis d'évaluer l'impact du vieillissement thermo-oxydatif et de la déformation des tuyaux et de répondre à deux questions principales. En premier lieu, l'évaluation des propriétés mécaniques à long terme des tuyaux a été réalisée par la méthode SIM (méthode isotherme par paliers) tandis que la résistance à la fissuration sous contrainte (SCR) a été évaluée par la méthode SCR FM 5-572. Dans les deux cas, les données à court terme ont été extrapolées à long terme pour prédire la durée de vie des tuyaux. Le module à long terme a été calculé par la méthode de Superposition Temps-Température (TTS) qui permet fournit une indication du module à long terme à basse fréquence ainsi que des informations sur la rigidité du matériau. En second lieu, la rupture s'est avérée fragile par thermo-oxydation avec l'exposition aux sels de déglaçage, aux cycles de gel-dégel, à l'abrasion et aux UV. Les divers essais et calculs de prédiction de la durée de vie des tuyaux de PE recyclé confirment que ceux-ci peuvent remplir les mêmes fonctions que les tuyaux de résine vierge dans les infrastructures de transport.

Mots-clés: Polyéthylène à haute densité (PEHD); résine recyclée; durée de vie; vieillissement; propriétés physico-chimiques et mécaniques; thermo-oxydation; déformation à long terme.

À mon père et ma mère pour leur confiance indéfectible, leur patience, leur clairvoyance, leur rigueur, leur bonté et les grands sacrifices qu'ils ont consentis

À ma défunte grand-mère pour les grands sacrifices qu'elle a consentis

À mes sœurs pour leurs encouragements

À tous mes amis pour leur présence

REMERCIEMENTS

Est venu le jour où je m'apprête à récolter le fruit de longues années de recherches. Le chemin a été long et difficile et j'avoue que je n'y serais jamais parvenu sans l'aide et le support de plusieurs personnes avec qui j'ai eu le privilège de travailler. Je tiens à mettre de l'avant l'ensemble des personnes qui m'ont entouré pendant ces trois années. Aussi, permettez-moi d'exprimer toute ma gratitude pour tout leur support.

En tout premier lieu, je tiens à remercier mon directeur de thèse, le professeur Brahim Benmokrane qui a accepté de diriger ce travail de recherche. Qu'il trouve ici l'expression de ma plus grande gratitude pour son soutien bienveillant et ses conseils. Je le remercie de m'avoir fait confiance et d'avoir cru en moi. Merci d'avoir été toujours disponible et prêt à pousser ce travail jusqu'au bout. Je souhaite également remercier mon codirecteur, le professeur Mathieu Robert, pour sa grande disponibilité et ses conseils pertinents. Je vous remercie tous deux de m'avoir donné l'opportunité d'explorer ce domaine tout en m'apportant votre expertise scientifique.

Je remercie aussi le docteur Patrice Cousin pour son appui au laboratoire et les longues discussions enrichissantes sur la caractérisation des polymères. Son partage de connaissances m'a permis de mieux comprendre la dégradation des polymères. Merci pour ses explications et sa patience et de m'avoir transféré sa passion de la recherche et sa rigueur scientifique. Je le remercie aussi pour sa gentillesse, sa disponibilité, ses conseils et son apport pour ce travail.

J'adresse également mes remerciements à Khaled Mohamed pour m'avoir accompagné tout au long de mes travaux et pour son soutien. Merci pour son aide sur la réalisation des expérimentations et ses conseils bienveillants durant toutes les différentes phases de ma thèse.

Ce travail n'aurait pas été possible sans le soutien du ministère des Transports du Québec (MTQ) ainsi que des entreprises collaboratrices dans ce projet (Soleno, ADS, Armtec et Infra Pipe Solutions Ltd).

Ce travail n'aurait pas pu être réalisé sans le support des techniciens et particulièrement de Jérôme Lacroix et Steven Mac Eachern. Merci pour leur aide au laboratoire.

Je tiens ensuite à adresser mes remerciements à l'ensemble des membres du groupe de recherche et mes collègues. Je remercie Louis Schutz pour son aide avec les mesures de la masse moléculaire et Richard Thomas (TRI Environmental Inc.) pour les discussions sur l'essai SCR.

J'aimerais remercier tous mes amis à Sherbrooke (Kien, Chi, Hue, Thang, Tai, Guillaume, Anthony, Stefanie) pour nos sorties et autres activités. Merci à vous tous pour votre amitié et plus spécialement à ceux qui partageaient avec moi ma vie de tous les jours. Je remercie mes amis de France également comme Nha An, Uy, Vincent, mes amis du Vietnam comme Nghia, Minh Tam, mes amis des États-Unis comme Tin, Duc pour tous ces moments partagés à distance. Merci, Vincent, de m'avoir rendu visite cet automne.

Pour terminer, je remercie ma famille, mes parents et sœurs pour leur soutien, leurs sacrifices et leur amour inconditionnel. Merci de m'avoir supporté et encouragé tout au long du parcours.

TABLE DES MATIÈRES

LISTE DES FIGURES

LISTE DES TABLEAUX

LISTE DES ACRONYMES

Acronyme	Définition
PE	Polyéthylène
PEHD	Polyéthylène à haute densité
HDPE	High-density polyethylene
FHWA	Federal Highway Administration
FDOT	Florida Department of Transportation
BNQ	Bureau de normalisation du Québec
MTQ	Ministère des transports du Québec
SCR	Stress crack resistance
UV	Ultraviolet
ASTM	American Society for Testing and Materials
SIM	Stepped Isothermal Method
TTS	Time-temperature superposition
NCLS	Notched, Constant Ligament Stress
PENT	Pennsylvania notched test
OIT	Oxidation induction time
IT	Oxidation induction temperature
FTIR	Fourier-transform infrared spectroscopy
TMA	Thermomechanical analysis
DMA	Dynamic mechanical analysis
SEM	Scanning electron microscopy
DOT	Department of Transportation
ESC	Environmental stress cracking
DSL	Design Service Life
EMSL	Estimated Material Service Life
AASHTO	American Association of State Highway and Transportation Officials
CSA	Canadian Standards Association
PIR	Post-industrial recycled
PCR	Post-consumer recycled
APR	Association of Plastic Recyclers
ACC	American Chemistry Council
NCHRP	National Cooperative Highway Research Program
UCLS	Un-Notched, Constant Ligament Stress
SCG	Slow crack growth
ESCR	Environmental Stress Crack Resistance
SCC	Stress corrosion cracking
ISO	International Organization for Standardization
HDB	Hydrostatic design basis
SSM	Stepped Isostress Method
MSIM	Modified Stepped Isothermal Method
DSC	Differential scanning calorimetry
RPM	Rate Process Method

PSM	Popelar’s Shift Method
NSERC	Natural Science and Engineering Research Council of Canada
FQRNT	Fonds québécois de la recherche sur la nature et les technologies
MFI	Melt flow index
HT-GPC	High-temperature gel permeation chromatography
TGA	Thermogravimetric analysis
MTS	Material Testing Systems
MWD	Molecular-weight distribution
ANOVA	Analysis of variance
CB	Carbon black
ATR	Attenuated total reflection
EDS	Energy dispersive spectrometry
WLF	Williams–Landel–Ferry
SD	Standard deviation
COV	Coefficient of variation

1 CHAPITRE 1: INTRODUCTION

1.1 Mise en contexte et problématiques

Depuis sa découverte dans les années 1950, le PE a été utilisé pour différentes applications. En particulier, les tuyaux ondulés en polyéthylène à haute densité (PEHD) sont largement utilisés dans les infrastructures de transport pour le drainage des routes, le drainage souterrain et la collecte d'eaux pluviales [1,2] du fait de leurs bonnes propriétés physico-chimiques et mécaniques et de la facilité d'installation de nombreux types de tuyaux (Type C, S, D) [3]. Résistants à la corrosion chimique, légers et offrant un faible coût de maintenance, ces tuyaux sont souvent exposés au sol et à l'eau. Le PE n'étant pas conducteur, les tuyaux en PEHD sont immunisés contre le processus de corrosion électrochimique causée par des électrolytes tels que les acides, les bases et les sels [1,4]. De plus, comme la structure des parois intérieures lisses de ces tuyaux permet de réduire le frottement, elle joue un rôle important dans les applications pour les collecteurs d'eaux pluviales et le drainage. Pour le secteur des infrastructures souterraines, les tuyaux en PEHD sont considérés comme flexibles par rapport aux matériaux traditionnels tels que l'acier et le béton.

Un ponceau peut être décrit comme un conduit hydraulique court acheminant de l'eau à travers un remblai de chaussée ou au-delà d'un autre obstacle à l'écoulement (Figure 1.1). Les matériaux de ponceau sont choisis en fonction de la résistance structurelle requise, de la rugosité hydraulique, de la durabilité, de la résistance à la corrosion et à l'abrasion nécessaires pour une application particulière [5]. Le béton, l'aluminium ondulé et l'acier ondulé sont trois matériaux couramment utilisés pour les ponceaux. Un nouveau règlement de la Federal Highway Administration (FHWA) en 2006 indiquait qu'une « considération égale » devait être accordée à la spécification de matériaux de tuyaux alternatifs, y compris le plastique et l'aluminium ondulé, jugés de qualité satisfaisante et jugés également acceptables aux bases de l'analyse technique et économique [6]. Par conséquent, l'utilisation de tuyaux thermoplastiques, en particulier de tuyaux PEHD ondulés, a commencé à prendre de l'ampleur. Facilement installé, le tuyau ondulé en PEHD est un produit idéal pour remplacer et maintenir les autres matériaux de drainage dégradés. Utilisés sous les routes et sous les voies de chemin de fer, ces tuyaux sont souvent courts avec les deux extrémités du tuyau ouvertes pour diriger l'eau directement. De plus, les drains médians (entre les voies divisées de l'autoroute) et les drains de bord (le long du bord de la chaussée) sont couramment utilisés pour le drainage. Ces tuyaux ont souvent un grand diamètre et recueillent l'eau de l'écoulement de surface. Les tuyaux de PEHD doivent être solides pour résister aux fortes charges de la circulation.

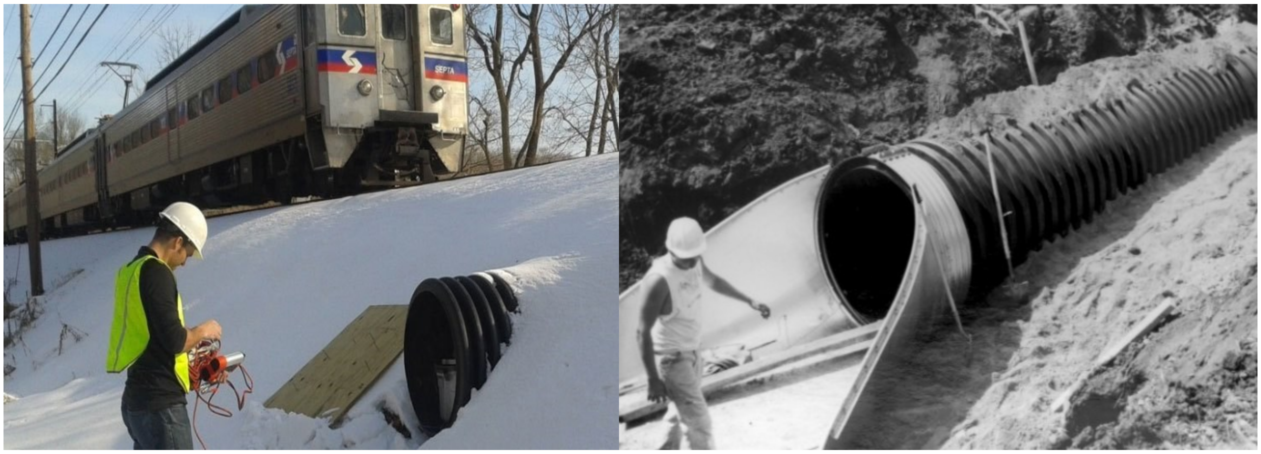


Figure 1.1 Ponceau en PEHD pour drainage des routes et sous les voies ferrées [1]

Un égout pluvial est une infrastructure conçue pour évacuer les excès de pluie et les eaux souterraines des surfaces imperméables telles que les rues pavées et les stationnements (figure 1.2). La conception des drains pluviaux varie des petits puits secs résidentiels aux grands systèmes municipaux. Les égouts pluviaux en PEHD sont légers et d'une longueur de 6,1 m ce qui permet de les connecter facilement et de réduire de moitié le temps d'installation et l'utilisation d'équipements lourds par rapport aux tuyaux en béton. Les tuyaux en PEHD résistent aux écoulements abrasifs, à la corrosion, aux attaques chimiques et même aux outils de nettoyage d'égout les plus agressifs. En général, les eaux de ruissellement peuvent contenir des hydrocarbures et d'autres produits chimiques, une large gamme de solides en suspension ainsi que des débris ramassés à la surface du sol pendant les précipitations. Le criblage, la filtration ou les systèmes hydrodynamiques peuvent être nécessaires pour améliorer la qualité du ruissellement. Le tuyau en PEHD ondulé est idéal pour les systèmes d'égout pluvial, tels que les systèmes de rétention souterrains, car il possède la résistance structurelle permettant de contrôler la quantité de ruissellement. Les égouts pluviaux sont dimensionnés pour des taux de ruissellement spécifiques, des systèmes mal dimensionnés peuvent créer une condition surchargée. Les tuyaux en PEHD sont plus flexibles que les tuyaux en béton armé dans les enterrements profonds en raison de leur capacité à interagir efficacement avec le remblai. S'ils sont enterrés correctement, ces tuyaux peuvent être enterrés à des profondeurs de 6 m ou plus. De plus, le tuyau en HDPE ondulé avec un intérieur lisse permet un écoulement efficace à travers un système de drainage pluvial fermé. Avec un système hydraulique aussi performant et un facteur de Manning (n) faible, il est souvent possible de réduire le diamètre du tuyau par rapport à son homologue avec intérieur ondulé [1].

D'autre part, les tuyaux en PEHD peuvent être utilisés pour les systèmes de gestion des eaux de rétention/détention (Figure 1.2). Un système de rétention/détention est un système de canalisation enterré qui stocke les eaux de ruissellement. Dans les zones nouvellement aménagées, ces systèmes sont généralement dimensionnés pour maintenir le taux de ruissellement avant l'aménagement, de sorte que les égouts pluviaux, les fossés et autres structures existants restent dans les limites de leur capacité. Les réseaux de conduites souterraines permettent aux terrains situés au-dessus du système d'être utilisés pour des stationnements et d'autres usages [1].

Figure 1.2 Tuyaux en PEHD pour les égouts pluviaux (gauche) et les systèmes de gestion des eaux de rétention/détention (droit) [1]

Les points résumés ci-dessous expliquent pourquoi les tuyaux en PEHD sont largement utilisés et constituent un des matériaux idéaux pour les projets d'infrastructure de transport [1]:

- *Économies de coût du cycle de vie.* Le coût du cycle de vie d'un tuyau en PE peut être considérablement inférieur à celui des tuyaux fabriqués avec d'autres matériaux. Choisir des tuyaux en PEHD est non seulement bénéfique pour la construction et la maintenance d'un drain mais constitue également un investissement positif dans la durabilité et la fiabilité de l'infrastructure de transport. La fiabilité, les performances en termes de débit et les faibles coûts à long terme en font un élément important d'une solution complexe pour faire face aux demandes futures [7].

- *Résistance à la corrosion et aux produits chimiques.* Les tuyaux en PEHD ont une excellente résistance chimique et constituent le matériau de choix pour de nombreux environnements chimiques difficiles.

- *Résistance à la fatigue et flexibilité.* Le tuyau en PEHD peut être plié avec un rayon d'environ 30 fois le diamètre nominal du tuyau et offre une résistance exceptionnelle à la fatigue. En effet, il peut supporter de multiples événements de pression et de surtension jusqu'à 100% de plus que sa pression de fonctionnement maximale sans que cela nuise à sa capacité de performance à long terme [1].

- *Construction simple et facile à installer.* Le PEHD est un matériau relativement léger, sa densité étant 1/8 fois celle de l'acier [1]. Il réduit l'utilisation d'équipement de levage lourd pour l'installation ce qui facilite le transport et l'installation ainsi que les coûts.

- *Hydrauliquement efficace.* D'une texture lisse à l'intérieur et avec un facteur de Manning (n) faible, les tuyaux en PEHD possèdent une grande capacité hydraulique.

- *Durabilité.* La valeur estimée de la durée de vie des tuyaux en PE est d’environ 50 à 100 ans si le système est correctement conçu, installé et utilisé conformément aux recommandations du fabricant. Cette valeur dépend de la composition du tuyau et des additifs (stabilisants, antioxydants, etc.) qui contribuent également à augmenter leur durée de vie [8,9].

Malgré le grand nombre d'applications mentionné ci-dessus, certaines limitations subsistent après plusieurs années de service, particulièrement en ce qui concerne le comportement à long terme tel que la fissuration des tuyaux ondulés de drainage en PEHD en raison de la contrainte et de la charge [10]. Aussi, des fissures peuvent être simultanément initiées en raison de l'oxydation thermique [11,12]. Les conditions de service varient considérablement en fonction de l'emplacement géographique, de l'installation des tuyaux, de la température et du sol ainsi que de la charge de trafic. Cela signifie que les agents physiques, chimiques ou mécaniques combinés ou pas peuvent dégrader les tuyaux en PEHD. Une des principales voies de dégradation est la thermo-oxydation, la déformation aux temps longs sous l'influence de la température et des conditions environnantes ainsi que la propagation des fissures sous contrainte. Pour ce faire, la durabilité des tuyaux en PEHD sera évaluée et les performances en service des tuyaux avec et sans résines recyclées seront comparées. Il est en effet important d’étudier en profondeur les tuyaux de PE recyclé et de confirmer que leur utilisation offrira la même résistance et durabilité que les tuyaux de résine vierge, ce qui contribuera à réduire les coûts de production et les impacts environnementaux.

Très peu de recherches traitent de la performance à long terme et de la prédiction de la durée de vie des tuyaux en PEHD fabriqués avec et sans résines recyclées pour les infrastructures de transport. Les performances à court terme des tuyaux ont été largement étudiées par des essais in situ et en laboratoire [13,14]. Cependant, la plupart de ces études portaient principalement sur les propriétés mécaniques [15-17] alors que les propriétés physico-chimiques et thermiques étaient rarement mentionnées. Pour les performances à long terme, plusieurs méthodes d’essai permettent d’évaluer la fissuration sous contrainte; cependant, peu d'essais traitent de la fissuration à long terme dans un délai raisonnable. De plus, il n’existe toujours pas de consensus clair au sujet de la durée de vie des tuyaux utilisés pour les égouts pluviaux et les ponceaux. Une étude réalisée en 2005 par Y.G. Hsuan et T.McGrath [18] pour le compte du Florida Department of Transportation (FDOT) a déterminé les spécifications requises pour garantir une durée de vie de plus de 100 ans. Ces chercheurs ont élaboré un modèle de vieillissement permettant d’estimer le temps requis avant qu’une rupture fragile ou une rupture fragile chimique surviennent en service. Pour y parvenir, différents essais de vieillissement accéléré (résistance à la fissuration lente, résistance à l’oxydation, résistance à la traction à long terme et module à long terme) ont été réalisés. Toutefois, l’effet des facteurs propres à l’environnement au Québec tels que les cycles de gel-dégel, les sels de déglaçage et l’abrasion n’a pas été abordé par ces chercheurs puisque l’étude ciblait avant tout les conditions d’exposition de la Floride. Parallèlement, dans un souci de réduction de l’empreinte environnementale des tuyaux de PEHD, l’utilisation de PEHD recyclé est maintenant permise et encadré par la norme BNQ 3624-120 [19] avec entre autres, de nouvelles classes de résine et l’ajout de nouveaux essais et critères de durabilité. Toutefois, l’aspect durabilité des tuyaux recyclé est encore peu documenté. Par conséquent, le Ministère souhaite savoir si les tuyaux en PEHD recyclé peuvent atteindre une durée de vie similaire à celle des tuyaux en PEHD vierge.

Ce projet de recherche vise donc, non seulement à pallier le manque de compréhension des mécanismes de dégradation des tuyaux en PEHD, mais aussi à fournir des informations concernant les performances des tuyaux de plastique recyclé utilisés dans les infrastructures de transport. Une étude expérimentale visant à étudier les performances à long terme des tuyaux a été entreprise et les résultats permettront de comprendre leur comportement et leur mécanisme de dégradation. La comparaison des performances des tuyaux de PE recyclé et vierge a été effectuée et la prédiction de leur durée de vie dans les conditions d'exposition du Québec a été étudiée. Cette étude est d'un grand intérêt pour les ingénieurs, les maîtres d'ouvrages et les entrepreneurs en ce qui concerne l'applicabilité des tuyaux de PE recyclé dans les infrastructures de transport. Finalement, cette étude établit une corrélation entre le temps de dégradation accéléré en laboratoire et le temps réel in situ permettant d'atteindre le même niveau de dégradation des tuyaux.

1.2 Objectifs

L'objectif principal de ce projet de recherche est d'évaluer la durée de vie des tuyaux à paroi ondulée de PEHD vierge et recyclé. Le projet fait partie d'un contrat avec le MTQ. La résistance à la traction à long terme, la résistance à la fissuration sous contrainte (SCR) et le module d'élasticité à long terme ont été évalués de même que les caractéristiques physiques et la stabilité à l'oxydation. Les essais à court terme sélectionnés incluent l'utilisation d'une concentration de contrainte (entaille) et d'une température élevée, de solutions environnementales et de contraintes élevées. La dégradation oxydative et la déformation à long terme des tuyaux dans les conditions d'exposition du Québec, y compris leurs caractéristiques dans des conditions d'exposition aux sels de déglaçage, aux cycles de gel-dégel et à l'abrasion ont été investiguées. Les objectifs spécifiques du projet de recherche sont:

1. Étudier les performances mécaniques, physiques et chimiques des tuyaux en PEHD fabriqués avec et sans résines recyclées et exposés à différents milieux environnementaux et aux cycles de gel-dégel afin de simuler les conditions d'exposition du Québec.
2. Comparer les performances à court terme des tuyaux fabriqués avec résines recyclées et vierges.
3. Développer des méthodes d'essais permettant de caractériser le comportement à long terme des tuyaux après exposition aux ultraviolets (UV), aux sels de déglaçage, au gel-dégel et à l'abrasion.
4. Évaluer et comparer les paramètres de conception à long terme (résistance à la traction, module d'élasticité et résistance à la fissuration sous contrainte) des tuyaux fabriqués avec ou sans résine recyclée, et générer des courbes d'enveloppe de durée de vie.
5. Établir une corrélation entre le temps de dégradation accéléré en laboratoire et le temps réel permettant d'atteindre le même niveau de dégradation.

1.3 Méthodologie

Pour atteindre les objectifs ci-dessus, sont présentés dans le tableau 1.1 le programme expérimental et les méthodes d'essais suggérées. Les propriétés mécaniques et physiques, telles que la résistance à la traction, le module d'élasticité, la résistance à la propagation lente des fissures, la densité, la température de ramollissement, le taux de noir de carbone, l'indice de fusion, le stabilisant de couleur et aux UV, la stabilité thermique de même que la résistance à l'oxydation ont été mesurées. Le tableau indique également la méthode d'essai ASTM correspondant à chacun de ces essais et le nombre d'échantillons. Une comparaison des propriétés des tuyaux fabriqués avec et sans résines recyclées est aussi présentée tant pour les propriétés à court terme avec la détermination de la résistance à la traction (ASTM D638 - type IV [20]) que pour les propriétés à long terme avec la mesure de la résistance à la traction et du module ainsi que la résistance à la fissuration sous contrainte.

La résistance à la traction et le module à long terme ont été évalués en utilisant la méthode SIM, TTS et la méthode conventionnelle. Les méthodes TTS et conventionnelle nécessitent de nombreuses expériences afin d'obtenir une courbe maîtresse pour un certain niveau de charge (au moins 3 essais de fluage à court terme par température pour la validation statistique), impliquant une durée d'essai longue et des coûts élevés. En revanche, la méthode SIM (ASTM D6992 [21]) ne nécessite qu'un seul échantillon testé sous une charge constante et soumis à différents palliers de température. Cette approche minimise à la fois les effets de dispersion et le temps d'essai, ce qui rend cette méthode très intéressante. La méthode SIM est donc recommandée et a donné des résultats satisfaisants par rapport aux deux autres méthodes.

La résistance à la fissuration lente (SCR) peut être mesurée avec l'essai de contrainte de ligament entaillé constant (NCLS) ou de l'essai d'entaille de Pennsylvanie (PENT) spécifié par les normes ASTM F2136 [22] et ASTM F1473 [23]. Les deux essais exigent que les échantillons soient conditionnés dans une solution à 10% d'Igepal (nonylphénol noxypoly). Cependant, les chercheurs ont constaté que cette solution pouvait considérablement accélérer le processus de fissuration lente dans les tuyaux en PEHD [24], ce qui ne pouvait pas refléter les conditions réelles d'exposition. L'essai de la Floride FM5-573 [25] précise toutefois que l'essai peut aussi être effectué avec de l'eau. Il convient de mentionner que Hsuan et McGrath (2005) [18] ont effectué des essais de résistance à la fissuration lente conformément à la norme FM5-573 en utilisant différents milieux d'exposition (air, eau et solution d'Igepal) et comparé les courbes de ductilité des tuyaux en PEHD obtenus dans chaque milieu d'essai. Les résultats ont révélé que la solution d'Igepal accélérait davantage la fissuration sous contrainte alors que l'eau et l'air fournissaient des données similaires pour les temps de rupture. En conséquence, l'essai FM5-573 a été adopté avec l'eau comme environnement même si la résistance à la fissuration des tuyaux dans l'air mérite aussi d'être étudiée.

Pour ce qui est de la résistance aux UV, des spécimens ont été prélevés après exposition et leur morphologie et chimie de surface, leur point de fusion, leur résistance à la traction ainsi que leur dureté (ASTM D2240 [26]) ont été caractérisés.

Pour une représentation plus précise des propriétés du matériau soumis aux environnements du Québec, les facteurs environnementaux tels que le sol, l'eau et l'air qui sont normalement en contact avec les tuyaux, ont été pris en compte. C'est pour cette raison que les essais ont été construits pour inclure des échantillons exposés à des environnements salin, acide et basique. Cependant, il est à noter que le PE étant très résistant à la corrosion, la présence de sel, d'acide et de base présente a priori peu de risques pour la longévité des tuyaux [1]. Par conséquent, il a été suggéré d'obtenir tout d'abord la rétention de la résistance à court terme après exposition au sel, à l'acide et à la base et d'évaluer par la suite si cette mesure à long terme cette fois devait être effectuée dans un deuxième temps. Généralement, les propriétés physiques et mécaniques des plastiques sont influencées par la température et l'humidité relative (ASTM D618 [27]). La province de Québec se distingue par de nombreux cycles de gel-dégel avec une température annuelle moyenne de 10^0C. Par conséquent, l'effet de ces cycles sur les performances à court et long terme des tuyaux est l'un des critères les plus importants pour déterminer de manière adéquate leur durée de vie. Ainsi, des cycles de gel-dégel de - 40^0C à + 40^0C ont été effectués avant d'évaluer la résistance à la traction, le module à court et long terme et la résistance à la propagation lente des fissures.

La résistance à long terme à la dégradation par thermo-oxydation peut être évaluée à l'aide de l'essai OIT (ASTM D3895 [28]) ou de l'essai IT (ASTM D3350 [29]). Les chercheurs ont constaté que l'essai OIT est plus sensible aux types d'antioxydant tandis que l'essai IT est plus approprié pour évaluer le mécanisme des stabilisants [18,30]. Par conséquent, le contenu en antioxydants et leur taux d'épuisement ont été évalués à l'aide de l'essai OIT après une incubation dans de l'eau à 60^0C pendant une durée de 12 mois afin d'induire une dégradation du matériau.

Tableau 1.1 Essais proposés pour les tuyaux en PEHD

Critère d'essai	Méthode d'essai	Nombre d'échantillons				
		Réf	Gel-dégel[a]	Sel[b]	Acide[c]	Base[d]
Tuyaux neufs						
Densité	ASTM D792	3	-	-	-	-
Point de ramollissement	TMA et DMA	3	-	-	-	-
Taux de noir de carbone	ASTM D5805	3	-	-	-	-
Stabilité thermique	ASTM E2550	3	-	-	-	-
Stabilité aux UV	ASTM D4329	3[e]	-	-	-	-
Indice de fusion	ASTM D1238	3	-	-	-	-
Résistance à la traction	ASTM D638	5	5	5	5	5
Résistance à la traction à long terme	ASTM D6992	3	-	-	-	-
Résistance à la fissuration sous contrainte (SCR)	FM 5-573	5	5	5	5	5
Module à long terme	TTS par DMA	3	3	3	3	3
Oxydation	ASTM D3895	3	3	3	3	3
Tuyaux sur le terrain						
Résistance à la traction à long terme	ASTM D638	5[f]	-	-	-	-
Résistance à la fissuration sous contrainte (SCR) à long terme	FM 5-573	5[f]	-	-	-	-
Module à long terme	TTS par DMA	3[f]	-	-	-	-
Oxydation à long terme	ASTM D3895	3[f]	-	-	-	-
Chimie de surface	FTIR	3[f]	-	-	-	-

[a] Essai réalisé dans l'air après exposition à 625 cycles de gel-dégel dans l'eau durant 12 mois.

[b] Essai réalisé dans l'air après immersion dans une solution de NaCl 1M à 60°C durant 12 mois.

[c] Essai réalisé dans l'air après immersion dans une solution de H_2SO_4 à pH 2 à 60°C durant 12 mois.

[d] Essai réalisé dans l'air après immersion dans une solution de NaOH à pH 10 à 60°C durant 12 mois.

[e] 3 échantillons pour chaque essai (morphologie de surface, chimie de surface, point de fusion (Tm) et flux de chaleur (Hm), dureté) et 5 échantillons pour l'essai de traction.

[f] Essai effectué sur les tuyaux prélevés sur le terrain après une certaine période de service.

1.4 Contribution et originalité du projet de recherche

Les résultats de ce projet de recherche représentent une étape importante vers le développement de guides et recommandations destinées aux ingénieurs pour la conception des infrastructures de transport comportant des tuyaux en PEHD. De plus, les données recueillies viendront appuyer les travaux des comités techniques nord-américains chargés d'élaborer des normes et des guides de conception pour les tuyaux en PEHD fabriqués avec des résines recyclées et destinés à un environnement nordique.

Par ailleurs, le 5 novembre 2015, le Gouvernement du Québec a déposé à l'Assemblée nationale la « Stratégie gouvernementale de développement durable 2015-2020 ». À cet effet, ce projet de recherche rejoint les principes de développement durable suivants :

- « Prévention » : Le développement de modèle de prédiction de la durée de vie des tuyaux en PEHD soumis à des facteurs propres à l'environnement québécois permettra de prévenir une éventuelle détérioration du matériau.

- « Production et consommation responsable » : Une meilleure connaissance de la durabilité de ces tuyaux permettra de mieux définir les usages appropriés pour les différents types (lisse ou ondulé, avec ou sans résine recyclée) de façon à atteindre les durées de vie ciblées des ouvrages et ainsi éviter le gaspillage de matériaux utilisés à mauvais escient.

- « Accès au savoir » : Pour le Québec, la recherche dans le domaine de la durabilité des tuyaux en PEHD est encore récente et doit être stimulée.

1.5 Organisation du document

Cette thèse est organisée comme suit :

- Le chapitre 2 (article 1) présente la revue de littérature sur laquelle se base le projet de recherche. Il dresse un bilan des connaissances actuelles sur les phénomènes de dégradation des tuyaux en PEHD. Après une description des applications des tuyaux dans le secteur d'infrastructure de transport, paramètres, les critères pour déterminer leur durée de vie sont définis. Le chapitre établit aussi des recommandations sur les méthodes expérimentales de prédiction à long terme de la durée de vie des tuyaux.

- Le chapitre 3 (article 2) présente les résultats expérimentaux des essais sur les performances à court terme des tuyaux avec et sans résines recyclées. Les propriétés physico-mécaniques et thermiques incluent la densité, la température de ramollissement, le taux de noir de carbone, la masse moléculaire, l'indice de fusion, la stabilité thermique et la résistance à la traction. Ce chapitre compare les performances à court terme des tuyaux avec et sans résines recyclées.

- Le chapitre 4 (article 3) se focalise sur les effets du rayonnement ultraviolet sur les tuyaux recyclés et vierges. Des échantillons de PEHD ont été prélevés après exposition aux UV et analysés par spectroscopie infrarouge, microscopie électronique à balayage, calorimétrie différentielle à balayage, mesures du temps d'induction oxydative et essais de traction.

- Le chapitre 5 présente une étude sur le comportement de fluage à long terme des tuyaux. La méthode de SIM (Stepped Isothermal Method) a été employée afin de déterminer la sensibilité du PEHD à la rupture par fluage.

- Le chapitre 6 (article 4) évalue la résistance à la propagation lente des fissures sous une contrainte constante (SCR) et la prédiction de la durée de vie des tuyaux de PEHD recyclé et vierge pour le Québec. Des échantillons de référence et conditionnés en solutions acide, basique et saline ou soumis à des cycles de gel-dégel duront 12 mois ont été étudiés.

- Le chapitre 7 (article 5) présente l'impact des conditions d'exposition sur la consommation des antioxydants, la résistance à la traction et le module à long terme des tuyaux en PEHD recyclé et vierge. La teneur en antioxydants est mesurée après conditionnements avec des cycles de gel-dégel ou immersion en solutions saline, acide et alcaline afin d'estimer les risques de défaillance par oxydation. De plus, les effets des différents conditionnements sur la résistance à la traction et le module à long terme ont été évalués.

- Le chapitre 8 porte sur l'étude de tuyaux en service au Québec qui ont été prélevés pour fins d'analyses, sans qu'il soit toutefois possible de retracer leur origine ni la nature de leurs composants (type de résine, additifs, etc.). Seule, leur durée en service et leur environnement étaient connus. Les différentes analyses et essais effectués sur ces échantillons ont permis de brosser un portrait précis de leurs propriétés à court et long terme ainsi que de leur maintien au cours du temps dans les conditions d'utilisation anticipées.

- Le dernier chapitre regroupe la conclusion générale, la limite du projet de recherche ainsi que des recommandations pour les travaux futurs.

2 CHAPITRE 2: REVUE DE LITTÉRATURE - MÉTHODES D'ESSAI À LONG TERME POUR DES TUYAUX EN PEHD - AVANTAGES ET INCONVÉNIENTS : UNE REVUE

Avant-propos

Auteurs et affiliation:

Khanh Q. Nguyen: Étudiant au doctorat, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l’Université, Sherbrooke (Québec) J1K 2R1, Canada.

Célestin Mwiseneza: Étudiant au doctorat, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l’Université, Sherbrooke (Québec) J1K 2R1, Canada.

Khaled Mohamed: Stagiaire postdoctoral, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l’Université, Sherbrooke (Québec) J1K 2R1, Canada.

Patrice Cousin: Chercheur associé, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l’Université, Sherbrooke (Québec) J1K 2R1, Canada.

Mathieu Robert: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l’Université, Sherbrooke (Québec) J1K 2R1, Canada.

Brahim Benmokrane: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l’Université, Sherbrooke (Québec) J1K 2R1, Canada.

Date de publication: 27 février 2021

Titre du journal: Engineering Fracture Mechanics

Titre en anglais: Long-term testing methods for HDPE pipe - advantages and disadvantages: a review.

Référence: Nguyen, K. Q., Mwiseneza, C., Mohamed, K., Cousin, P., Robert, M., & Benmokrane, B. (2021). Long-term testing methods for HDPE pipe-advantages and disadvantages: A review. Engineering Fracture Mechanics, 107629. https://doi.org/10.1016/j.engfracmech.2021.107629.

Résumé en français:

Le polyéthylène à haute densité (PEHD) est devenu un matériau d'intérêt pour les ponceaux et les canalisations de drainage en raison de ses nombreux avantages. La question de la durée de vie utile des tuyaux en PEHD fabriqués avec des résines recyclées par rapport aux résines vierges a fait l'objet d'études de recherche au cours des dernières années. Un certain nombre d'études récentes ont confirmé que le PEHD est durable avec une durée de vie prolongée de plus de 100 ans. Néanmoins, les critères d'exposition, tels que l'environnement et les conditions de service, pourraient affecter la durée de vie des tuyaux. Par conséquent, donner des chiffres précis sur la durabilité des tuyaux en PEHD doit être clairement abordé. De telles préoccupations ont été soulevées par le ministère des Transports du Québec (MTQ) pour évaluer la durabilité des tuyaux en PEHD fabriqués avec des résines recyclées ou vierges en tenant compte des conditions d'exposition dans les climats nordiques. Cet article donne un aperçu de la recherche sur les principaux facteurs et mécanismes qui affectent la durée de vie des tuyaux en PEHD. Des méthodes pour prédire la durabilité à vie des tuyaux en PEHD, y compris la résistance à la rupture par fluage, la résistance à la fissuration sous contrainte et l'oxydation, sont présentées, soulignant les avantages et les inconvénients de chaque technique. Les études menées sur les performances en fatigue des tuyaux sont mises en évidence. De plus, un aperçu des études sur l'utilisation de tuyaux en PEHD fabriqués avec des résines recyclées pour des applications d'infrastructure de transport est également présenté.

Mots-clés: Tuyau en polyéthylène à haute densité (PEHD); les applications d'infrastructure de transport; résines recyclées et vierges; fluage, fatigue, température, contrainte, fissuration, rupture ductile/fragile, méthodes de prédiction de la durée de vie; performances et durabilité à long terme; spécifications et codes de conception, durée de vie de 100 ans; les essais accélérés.

Abstract

High-density polyethylene (HDPE) has become a material of interest for culverts and drainage pipelines as a result of its numerous advantages. The issue of service-life expectancy of HDPE pipes made with recycled versus virgin resins has been the focus of research studies over the past several years. A number of recent studies have confirmed that HDPE is durable with an extended service-life performance of over 100 years. Nevertheless, exposure criteria, such as environment and service conditions, could affect pipe service life. Consequently, giving specific figures on the durability of HDPE pipes needs to be clearly addressed. Such concerns were raised by Quebec's Ministry of Transportation (MTQ) to assess the durability of HDPE pipes made with recycled or virgin resins taking into consideration exposure conditions in northern climates. This paper provides an overview of the research on the main factors and mechanisms that affect HDPE pipe lifetime. Methods for predicting the lifetime durability of HDPE pipes—including the creep rupture strength, stress crack resistance, and oxidation—are introduced, emphasizing the advantages and disadvantages of each technique. Studies conducted on the fatigue performance of the pipes are highlighted. Moreover, an overview of studies on the use of HDPE pipes manufactured with recycled resins for transportation infrastructure applications is also presented.

Keywords: High-density polyethylene (HDPE) pipe; transportation infrastructure applications; recycled and virgin resins; creep, fatigue, temperature, stress, cracking, ductile/brittle failure, lifetime prediction methods; long-term performance and durability; specifications and design codes, 100-year service life; accelerated tests.

2.1 Introduction

Since its discovery in 1898 by Hans von Pechman [31], polyethylene (PE) has become one of the most widely used thermoplastic materials in the world. The original application for PE was insulation for radar cables during World War II [32]. In 1953, high-density polyethylene (HDPE) was first produced at virtually the same time by two separate research groups: that of John P. Hogan and Robert L. Banks (US) and of Karl Ziegler (Germany). In North America, HDPE pipes were used for industrial, oil, and gas production applications [4]. Given the performance benefits in these early applications, HDPE pipes have been widely used for culverts as well as drainage and potable-water distribution systems. HDPE pipes have excellent corrosion resistance, flexibility, and hydraulic efficiency. Moreover, they are light and have fused joints unlike other types of pipes [33-36]. In addition, HDPE pipes are easy to install and entail low maintenance costs. These properties coincide with applications that require a flexible material for long-term performance. HDPE pipes were first used for road-drainage systems in the 1970s by the Iowa Department of Transportation (DOT) and by the Georgia DOT; corrugated HDPE pipes were used by the Ohio DOT in 1981 [37].

Nowadays, HDPE pipes for street and highway drainage applications are commercially available in sizes up to 60 inches in diameter [3]. The desire to use large diameter pipes as well as recycled resins are the current subject of concern. The uncertainty about the long-term performance and durability of pipes under repeated loading, problems related to chemical attack by soil and water, stability under UV light, environmental conditions, installation quality and practice, and resin types calls for investigation by researchers. Many studies were conducted early on. Corrugated HDPE pipes with different diameters were tested to determine the relationship between buried HDPE-pipe deflection and soil-cover height at various backfill densities [38,39]. Moser (1994) [40] tested three 48-inch diameter HDPE profile wall pipes to investigate the structural performance characteristics as a function of depth cover. Goddard (1992) [41] and Nazar (1988) [42] indicated the possibility of HDPE-pipe failure modes, such as ring deflection; localized wall bucking; compressive wall stress; pipe wall strain [41]; tensile, compressive, and flexural failure; creep rupture failure; and environmental stress cracking (ESC) [42]. Many researchers [43-47] have investigated the performance of virgin and recycled corrugated HDPE pipes. Their results indicate that HDPE pipes—whether made with recycled or virgin resins—were durable and provided long-term performance. There is, however, a diversity of production and installation standards for HDPE pipes. The durability of HDPE pipes depends on the conditions at each installation area, such as service conditions, soil modulus, and the regional and local geological environment. Therefore, the integrity of the factors that affect the durability of HDPE pipes over long-term use should be taken into account. This helps managers devise strategies for economic development, operation, and pipeline repair.

Recently, a research project was initiated at the University of Sherbrooke in collaboration with Quebec's Ministry of Transportation (MTQ) to assess the durability of HDPE pipes made using recycled and virgin resins produced by local manufacturers (Figure 2.1). The objective of the research study is to evaluate the performance of pipes under exposure conditions in the province by implementing state-of-the-art durability testing of HDPE pipes. Unlike the existing studies mentioned above, which are assessing structural-performance limits (e.g., design and product, the effect of the installation process, wall buckling, and deflection), or other system performance limits (e.g., joints and connections). The present study is an overview of the existing long-term testing methods for HDPE pipe. This review looks into the origin of the degradation mechanism of HDPE pipes to propose testing methods and predict their lifetime. The main objective of this article is to identify the advantages and disadvantages of all existing long-term testing methods and then recommend suitable testing methods for the specific purpose of researchers and engineers.

The present study is laid out as follows. Design Service Life (DSL) and Estimated Material Service Life (EMSL) overview requirements are presented in section 2.2. The standard specifications for recycled pipes and the current status of their use are clarified in sections 2.3 and 2.4. This is followed by the review of long-term performance in section 2.5, long-term fatigue in section 2.6. Long-term testing methods and temperature factors for these testing methods are presented in sections 2.7 and 2.8. Finally, the discussion and main conclusions are drawn in sections 2.9 and 2.10 of the paper.

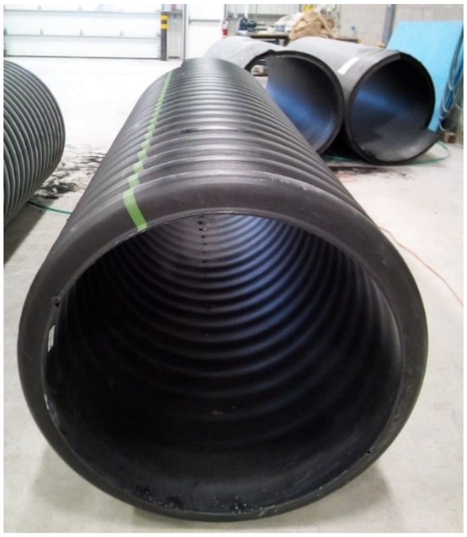


Figure 2.1 HDPE pipes under investigation

2.2 Design Service Life (DSL) vs. Estimated Material Service Life (EMSL)

HDPE pipes for infrastructure projects should be designed with a targeted design service life (DSL). The DSL for road/highway projects depends on the type of roadway, traffic volume, and the function of the gravity pipe. Pipe DSL is defined as the minimum number of years that a pipe must remain in service without major distortions or significant degradation preventing it from performing its intended functions [48].

The EMSL is defined as the number of years for which a material can function in its intended role. In other words, the EMSL is the length of time that a material performs satisfactorily before rehabilitation or replacement [49]. In infrastructure applications, this would be the length of time that a pipe (such as sewer, culvert, or drainage pipe) can ensure both hydraulic capacity and provide roadway structural support. The difference between the DSL and EMSL should be clearly understood. In general, the EMSL must meet or exceed the DSL [48]. The literature has only limited data on predicting EMSL values for HDPE, while there are many methods with specific equations for predicting the EMSL of concrete pipe such as the Ohio DOT, Hurd, Hadipriono, and Florida models [50-52] and of steel pipe such as the California method [48]. There is, however, no reliable model with specific equations to predict the EMSL of HDPE pipes. Some research results on HDPE components used in landfills [8,53] support predicting EMSL values for HDPE pipes. While such results yield a few quantitative EMSL values, the component materials are not ideal for representing HDPE pipes. Moreover, the service and environmental conditions were completely different. Recent research by Hsuan and McGrath has gradually shed light on EMSL prediction methods [18,54,55]. Most managers often use a constant value for the EMSL for all types of HDPE pipes for different applications without reliable EMSL prediction models. Florida DOT recommends that an EMSL of 100 years should be used for HDPE pipe. The Ministry of Transportation of Ontario (MTO) accepted an EMSL value of at least 75 years for HDPE pipe. The Ministry of Transportation of Québec (MTQ) accepts an EMSL of 50 years for HDPE pipe for transportation infrastructure applications. In addition, this EMSL value does not refer to HDPE pipes made from recycled resins.

2.3 HDPE-pipe Standard Specifications

There are a number of American standards for HDPE pipe, including Standard Specification for Corrugated Polyethylene Pipe, 300- to 1500-mm (12- to 60-in.) Diameter [3] and Standard Specification for Polyethylene (PE) Large Diameter Profile Wall Sewer and Drain Pipe [56]. In January 2018, the American Association of State Highway and Transportation Officials (AASHTO) specification was revised to allow the manufacture of HDPE pipes with either virgin or recycled materials based on (Pluimer et al.)'s (2018) research [47]. There are also two Canadian standards for the HDPE pipe: Profile Polyethylene (PE) Sewer Pipe and Fittings for Leak-Proof Sewer Applications (CSA B182.6) [57] and Profile Polyethylene (PE) Storm Sewer and Drainage Pipe and Fittings (CSA B182.8) [58]. CSA B182.8 allows HDPE pipe products containing recycled material to be used as drainage or sewer lines under highways. Currently, only a few studies have addressed the durability of HDPE pipe containing recycled material [47].

2.4 Recycled HDPE pipes in the literature

HDPE is an ideal material for recycling, as many packaging products are made of it. Blended recycled materials make the HDPE pipes more sustainable and cost-effective compared to pipes made of virgin materials. The literature makes a few rare references to the use of recycled materials for HDPE pipes. In general, there are two types of recycled materials used in HDPE pipes: post-industrial recycled (PIR) (pre-consumer) and post-consumer recycled (PCR). PIR materials are waste from the original manufacturing process, whereas PCR materials are waste materials (detergent bottles and milk jugs) that have been used by consumers. Blom et al. (1998) [59] showed that PIR materials are relatively easy to deal with because contamination from other resins is unlikely in comparison to PCR materials. In contrast, PCR materials are often a mixture of several polymers. Two main reasons account for this. First, consumers tend not to sort plastic when disposing of it. Second, there are blends in the recycled stream when products are made up of two or more different homopolymers. It can therefore be concluded that current PIR materials are easily recyclable, while PCR materials are not. Dormer et al. (2013) [60] pointed out that using recycled plastics can reduce the carbon footprint of a product. Specifically, a 24% decrease in carbon footprint can be obtained when the product contains 100% recycled content. The Association of Plastic Recyclers (APR) [61] conducted a study on the life cycle impact of post-consumer recycled resins. The results indicated significant reductions in energy requirements; water consumption; solid wastes; and greenhouse-gas emissions for the collection, transport, separation, and reclaiming processes with PCR materials. This analysis showed that recycled plastic reduced total energy consumption by 88% for HDPE. Korhonen and Dahlbo (2007) [62] also considered that recycling plastic can reduce the use of raw materials and energy in the production process and generate fewer greenhouse-gas emissions. Although PCR materials contain higher concentrations of impurities than virgin resins, the recycling process is suitable for PCR HDPE [63] based on the super-clean recycling process for highly volatile compounds. Therefore, a PCR certification program is currently being conducted by the Association of Plastic Recyclers (APR). The American Chemistry Council (ACC) is also establishing principles for certification standards to monitor recycling content through chemical recycling processes.

Very little has been found in the literature about predicting the long-term behavior and service life of corrugated HDPE pipes made with recycled materials for road and infrastructure applications. Some recent works [44-47,64] have investigated this issue. Recently, according to NCHRP Research Report 870 [47], corrugated HDPE pipes with recycled resin meets the same service-life requirements for road and infrastructure applications as pipes made with virgin resin. In addition, these recycled HDPE pipes meet the UCLS requirements. Therefore, AASHTO M924 standard was updated in 2018 and ASTM F2306/F2306M [65] was in 2019 to bring corrugated HDPE pipes manufactured with recycled (PCR or PIR) materials in line with specified requirements.

2.5 Long-term Performance and Durability of HDPE pipes

In the case of infrastructure projects, durability is one factor that determines pipe selection and use. The most basic definition of durability is the ability to last for a long time during which the original properties of the material are retained [66]. This includes the ability to withstand impacts due to environmental or other service conditions as well as the ability to resist erosion, material degradation (e.g., corrosion resistance and mechanical resistance), and loss of function. In general, there are three failure modes to consider for the long-term performance and durability of corrugated HDPE pipe: (1) structural-performance limits (e.g., design and product, the effect of the installation process, wall buckling, and deflection); (2) material degradation (effect of environmental conditions); and (3) other system performance limits (e.g., joints and connections) [67]. This article reviews the main factors according to the basic methods predicting the lifetime of HDPE pipes.

The characteristics of pipe constituent materials are the main factors affecting the durability performance of pipe used for culverts and storm drains [68]. Each characteristic affecting the long-term performance and durability of HDPE pipes is investigated with a different method.

This article highlights the advantages and disadvantages of each method, as presented in past studies.

Applied stress leads to one of three types of pipe failure as a function of time and based on the stress level [69]. As shown in Figure 2.2, these are ductile failure (stage I), brittle failure due to slow crack growth (SCG) (stage II), and failure due to chemical/molecular degradation (stage III). The transition point from stage I to stage II is called the "mechanical knee" and from stage II to stage III, the "chemical knee."

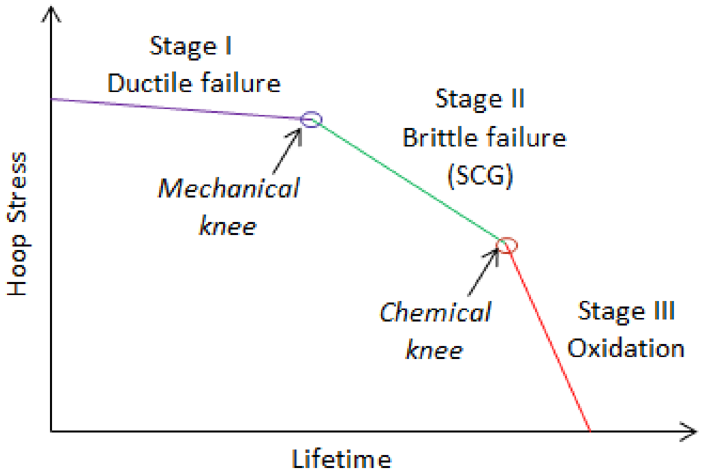


Figure 2.2 Three failure modes for HDPE pipe

A high stress level indeed leads to ductile failure (stage I) associated with large irreversible deformation. The ductile failure mode occurs when the HDPE pipe is subjected to high stresses close to its elastic capacity. The ductile behavior of polyethylene in tension is influenced by the material's semicrystalline nature. When referring to the stress–strain curve for ductile failure at the microscale, before the yield point, no visible deformation occurs, and the load is carried by rigid crystalline lamellae. As strain increases, stress increases, and yield occurs. The maximum point corresponds to the formation of a drag section with a significantly smaller surface. The load on the sample remains at a relatively constant level. After a period of time, a large increase in the stress value with an increase in deformation indicates the occurrence of work hardening. During work hardening, the amorphous phase reaches its full extension, and the material undergoes transformation with the rupture and triggering of the lamellae [70,71]. Certification tests for characterizing this type of failure [72,73] are expensive and time-consuming. Several methods consider high-temperature factors as an accelerated method for predicting long-term ductile failure of HDPE pipe. These methods shorten test time and extrapolate the test results at low temperatures when testing would otherwise be lengthy. Such methods are time-temperature superposition (TTS) and the stepped isothermal method (SIM).

At the intermediate stress level (stage II), failure occurs in a brittle manner. This result involves brittle cracking through slow crack growth. The process of slow crack formation is largely influenced by the location of the initiation site. The time to crack initiation varies from 20% to 80% of the total lifetime [74]. At a relatively low load level, the failure of a pipe is mainly due to either mechanical stress or a combination of stress and chemically aggressive environment. Stress corrosion cracking (SCC) and environmental stress cracking (ESC) result from long-term exposure of the pipe to a chemical environment [75,76]. Fully linear HDPE resins (without comonomer) are very fragile and prone to cracking under environmental stress. To resolve this problem, small amounts of comonomer are incorporated into HDPE during manufacture, which decreases the density and crystallinity while increasing the resistance to cracking under environmental stress (ESCR). At the initial stages of brittle failure, the amorphous materials begin to stretch under stress. Due to the longer duration, interlamellar bonds under tension begin to loosen and dissociate from each other until the number of remaining bonds becomes very low. When the few remaining interlamellar bonds are stretched to the extreme, they are unable to maintain the lamellae, resulting in brittle rupture of the polymer [71,77]. This article introduces the standards for evaluating the resistance to brittle failure (ASTM F1473-18 [23]; ASTM D1693-15e1 [78]; ASTM F2136-18 [22]; FM 5-573 [25]).

The final step (stage III) occurs once there is no apparent yield deformation, and the rupture does not depend on stress level. In other words, the material at this stage is no longer able to bear any load [11,12]. In stage III, the failure occurs mainly because many cracks start at the same time. The main factor is that the pipe is subjected to an environment generating oxygen radicals (e.g., thermal oxidation, photochemical oxidation by UV, X-rays, chemical oxidation by peroxide, or chlorine). An adequate content of carbon black or additives such as antioxidants or UV stabilizers can help extend pipe lifetime [79] [80]. Therefore, it is important to determine the amount of antioxidants needed to prevent stage III before the expected lifetime. Oxidation induction time (OITtime) and oxidation induction temperature (OITtemp) tests to examine antioxidant consumption in HDPE pipes are presented herein.

There are two main mechanisms for controlling HDPE pipe failure: ductile (stage I) and brittle (stage II SCG and stage III SCC) mechanisms, respectively, characterizing short- and long-term failures. Figures 2.3 and 2.4 [81] illustrate typical ductile and brittle (stage II) failures in HDPE pipes. Stress corrosion cracking (SCC) in HDPE pipes can be observed from the formation of the main crack as a combination of small cracks, as shown in Figure 2.5 [82]. For brittle fracture, the damage zone is dominated by a single craze and structural voids. For the ductile fracture, the craze is longer as well and the material significantly transformed comparatively to the brittle fracture, as observed under polarized-light microscopy [81].

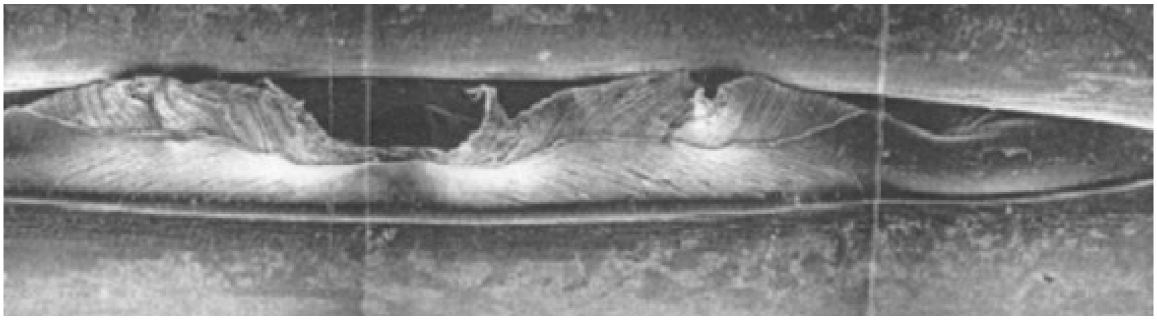


Figure 2.3 Ductile failure of HDPE pipe (stage I) [81]

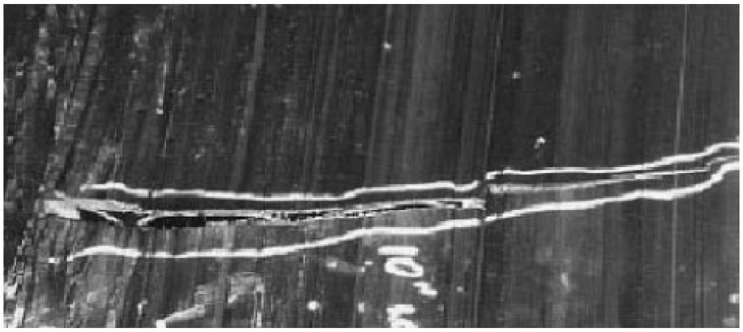


Figure 2.4 Brittle failure of HDPE pipe (stage II) [81]

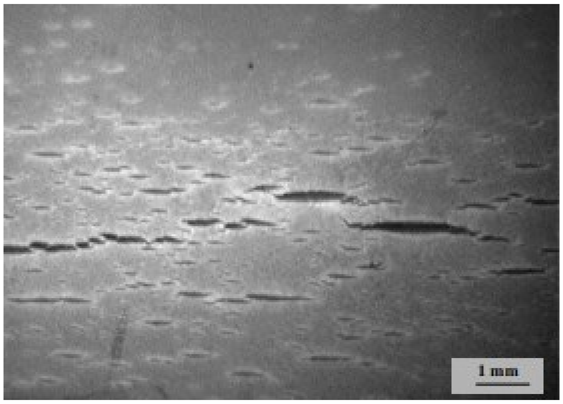


Figure 2.5 Brittle failure of HDPE pipe (stage III) [82]

Temperature and stress levels are used as acceleration factors to shorten the testing time in many laboratory tests. This is based on the relationship between the acceleration of failure time depending on the stress level and the temperature factor for semicrystalline polymers [69]. According to the Arrhenius law, brittle failure occurs after a long time at lower temperatures and shorter time at high temperatures. The use of the extrapolation method in ISO 9080-12 [72] can easily predict the time of brittle failure at room temperature by investigating ductile and brittle failures data at 40^0C, 60^0C and 80^0C. The following section discusses acceleration methods for predicting the lifetime of HDPE pipes.

2.6 Long-Term Fatigue Crack Growth

The expected service life failures of HDPE pipes in stages I and II occur under static loading conditions, while fatigue could also induce cracking or accelerate crack propagation [83]. Zhou and Brown (1992) [84] indicated that crack growth due to fatigue loading results from two damage processes, known as pure fatigue or combined fatigue and creep. Pure fatigue damage occurs when the number of cycles to failure was independent of frequency, while, with combined fatigue and creep, the total time to failure decreased with increasing frequency. Accordingly, researchers have suggested that increasing the frequency of fatigue loading can significantly accelerate the creep cracking rate without the need for accelerated agents or stress risers, such as high temperatures, notching, or incubation media [85,86]. Additionally, researchers observed significant correlation between fatigue and SCR failures, including the similar appearance of fracture surface and failure progression [24]. Zhang (2005) [24] reported that fatigue testing at ambient temperature could be performed to predict the SCR of HDPE. Such testing, however, could be hindered by the scarcity of acceptable testing protocols for the fatigue performance of HDPE pipes for evaluating SCR.

Research addressing the potential fatigue damage of HDPE pipes reported that HDPE material made with virgin resins had high fatigue resistance [83,87]. Pluimer et al. (2015) [44], however, indicated that PCR HDPE pipes could be vulnerable to fatigue-related cracking due to the presence of contaminants that act as stress risers. Therefore, a research team from Villanova University and Southeastern Pennsylvania Transit Authority conducted comprehensive field and laboratory tests to assess the performance of HDPE pipes manufactured with recycled materials. The project involved installing the first large-diameter corrugated HDPE pipe manufactured using recycled resin underneath commuter railroads in conjunction with similar pipes made with virgin resins. Pluimer et al. (2018) [47] observed that both pipes exhibited similar strains and deflections that were well below industry recommendations. Additionally, the field and laboratory studies under fatigue loading indicated that long-term fatigue crack growth is not a concern for HDPE pipes made with virgin or recycled material.

2.7 Long-term Testing Methods for HDPE pipe

There are investigation methods corresponding to each stage of pipe failure. Each method, however, has certain advantages and disadvantages, as described in Table 2.1. The choice of method for determining the long-term durability of a pipe depends on the specific purpose and conditions.

2.7.1 Creep Rupture Strength (Stage I)

2.7.1.1 Hydrostatic Design Basis (HDB)

The hydrostatic design basis (HDB) test is used to determine the long-term hydrostatic strength of thermoplastic materials for piping applications. The procedures for estimating long-term hydrostatic strength or pressure-strength are the two basic procedures in this method. They are based on similar principles of evaluating the stress rupture (induced only by internal pressure) under various stress levels and temperatures. The test time for this method is at least 10,000 hours at the different temperatures (40^0C, 50^0C, 60^0C, 80^0C, and 100^0C) [73]. The data can be extrapolated with a logarithmic scale to calculate the resistance at the desired lifetime of a pipe. The same basic test will reveal failures in stages I and II. Nevertheless, this test method does not reflect the actual working conditions of corrugated HDPE pipes for infrastructure projects, where the pipe is not subjected to internal stresses. Therefore, other methods for assessing stage-I failure of HDPE pipes are introduced in the next section.

Table 2.1 Long-term testing methods for HDPE pipe

Long-Term Test Method	**Test Standard**	**Type of Degradation**	**Advantage**	**Disadvantage**	**Reference**
Creep rupture strength (stage I)					
HDB	ASTM D2837	Mechanical degradation	▪ Test on full-scale pipes.	▪ Test duration: at least 10,000 hours. ▪ Does not reflect the actual working condition of pipe in infrastructure projects.	
TTS	-	Mechanical / Thermal degradation	▪ Simple test procedure. ▪ Easy to analyze the test results.	▪ Requires many replicated specimens. ▪ Relatively long test that can be run for thousands of hours.	[88]
DMA TTS	-	Mechanical / Thermal degradation	▪ Relatively short test (within hours). ▪ Easy to analyze the test results.	The formation is limited to 0.04%.	[18]
SIM	ASTM D6992	Thermal degradation	▪ Relatively short test (24 hours). ▪ Only one specimen undergoes a series of temperatures.	▪ Requires an iterative process to analyze the results. ▪ Very sensitive to the thermal expansion of the material	[46,89]
Conventional test	ASTM D5262	Mechanical degradation	▪ Test is performed only at 23^0C.	▪ Relatively long test that can run for years before collecting results.	[88]
SSM	-	Mechanical degradation	▪ Relatively short test (24 hours to 1 week). ▪ The test uses stress at the activation energy ▪ Only one specimen at 23^0C.	▪ Method relies on adjusting the fitting parameters to give a smooth master curve. ▪ Rupture could occur at a stress level near the ultimate strength.	[64,90]
Long-term resistance to stress cracking (stage II)					
ESCR	ASTM D1693	Chemical degradation	▪ The test is performed in different environments. ▪ Easy to perform and detect ruptured specimens.	▪ The stress relaxation and the stress relaxation rate are difficult to quantify. ▪ Large standard deviation.	[24]
PENT	ASTM F1473	Mechanical degradation	▪ Flexible test that can be implemented with different temperatures and/or stress levels.	▪ Relatively long test that can run for thousands of hours. ▪ Test method is ambiguous regarding the transition of brittle to ductile failure mode.	[91]
NCLS	ASTM F2136	Mechanical degradation	▪ Relatively short test (done within 1000 hours). ▪ Simple procedure and analysis of the results.	▪ The incubation environment is very aggressive.	[46,79,92]
Florida Test Method	FM 5-573	Mechanical degradation	▪ The test is performed in a water incubation environment.		[18]
UCLS	ASTM F3181	Mechanical degradation	▪ Can be implemented with recycled HDPE resins. ▪ Validated using experimental field results.		[47]
Long-term Oxidation (stage III)					
OIT$_{time}$	ASTM D3895	Thermal / Oxidative degradation	▪ More sensitive.	▪ Not related to the long-term performance of pipes	[8,18]
OIT$_{temp}$	ASTM D3350	Thermal / Oxidative degradation	▪ Low standard deviations	▪ Not related to the long-term performance of pipes. ▪ The distinction between different polymer samples is poor.	[93]

2.7.1.2 Time-Temperature Superposition (TTS)

Time-temperature superposition (TTS) is an accelerated method to evaluate the viscoelastic behavior of a polymer. The tensile strength and long-term modulus of HDPE pipe (stage I) can be determined with this method. Data are obtained at several temperatures corresponding to a range of time or frequencies. By using shift factors, creep or stress relaxation curves obtained at elevated temperature can be shifted to create a master curve and the behavior of the polymer can be determined at a specific temperature (reference temperature). The temperature-dependent shift factors for stress levels (vertical shift factor) and time/frequency (horizontal shift factor) are used in this method [94].

Once a specimen has been to a constant load at a certain temperature, the creep curves vs. log (time) are plotted (Figure 2.6a). Similar experiments are repeated at different temperatures. Using the shift factor calculated from the Williams-Landel-Ferry equation (Eq. 2.1) at the reference temperature (T_R), a single curve called a master curve may be draw, as illustrated in Figure 2.6b [95].

$$\mathrm{Log}(a_T) = \frac{C_1(T-T_R)}{C_2 + T - T_R} \tag{2.1}$$

where a_T is the shift factor, T the temperature, T_R the reference temperature, and C_1 and C_2 are empirical constants. When the glass transition temperature of the polymer T_g is selected as the reference temperature, C_1 and C_2 become universal constants equal to 17.44 K and 51.6 K, respectively.

Popelar et al. (1991) [96] found that horizontal shifting (temperature) alone cannot yield a coherent master curve, due to the effect of temperature on crystallinity. Vertical shifting, however, can help produce a master curve. Accordingly, Popelar et al. (1991) [96] developed Popelar's Shift Method (PSM) using two shift factors to create a master creep curve with a_T as the horizontal shift factor and b_T the vertical shift factor (Eqns. 2.2 and 2.3).

$$a_T = \exp[0.109(T-T_R)] \tag{2.2}$$

$$b_T = \exp[0.0116(T-T_R)] \tag{2.3}$$

PSM is used in the Florida test method for predicting the long-term modulus of corrugated HDPE pipes [97]. ASTM D2837-13e1 [73] and ISO 9080-12 [72], however, endorse another shifting method (known as the Rate Process Method or RPM) to analyze the stress cracking of pressure pipes. ISO 9080-12 uses a four-coefficient model, while the ASTM procedure recommends a three-coefficient model, as shown in Eq. 2.4.

$$\log(t) = A + \frac{B}{T} + \frac{C \log \sigma}{T} \tag{2.4}$$

where *t* is the time to failure in hours, *σ* is the applied stress in MPa, and *T* is the test temperature in *K*. The three constants—*A*, *B*, and *C*—are determined using least-squares multivariable regression analysis on data obtained from accelerated tests. The equation with known constants can then be applied to predict the master curve at any temperature and stress under the same test conditions. Hsuan et Zhang (2005) [92] and Hsuan and McGrath (2005) [18] found that RPM yielded a good prediction for HDPE pipes and considered it a more reliable method for predicting the long-term strength of HDPE pipes. In contrast, the field tests conducted by Pluimer et al. (2018) [47] suggest that PSM would be more accurate in predicting failure times than RPM.

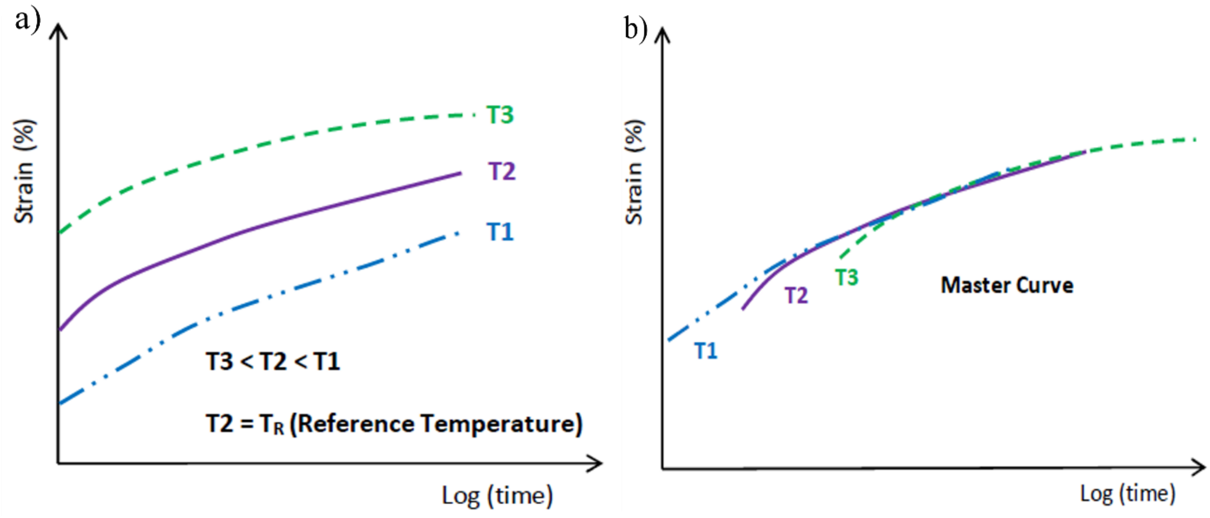


Figure 2.6 Time-temperature superposition results: a) creep curves at different temperatures, and b) typical master curve

2.7.1.3 DMA (Dynamic Mechanical Analysis) TTS Method

Hsuan and McGrath (2005) [18] conducted stress relaxation tests with a DMA analyzer according to the TTS method. The rectangular specimen was clamped between two mechanical arms and the deformation limited to 0.04%. According to ASTM D2412-11-18 [98], the parallel-plate test should be performed at a deflection of 5% under a stress relaxation mode that reflects the actual working conditions of the pipe. The temperature steps were 27.5^{0}C, 35^{0}C, 42.5^{0}C, 50^{0}C, 57.5^{0}C, and 65^{0}C with a master curve obtained at 27.5^{0}C. Moreover, a master curve at 27.5^{0}C using PSM also gave results similar to shifted data with the DMA TTS. Using a DMA makes it possible to use TTS software, which can create a master curve with only a few operations and shorter testing time compared to conventional and long-term TTS methods. The specimen size for the DMA TTS method is relatively small (approx. 4x8 mm^2). Variability, as well as small variations in specimen size, might affect the results.

2.7.1.4 Stepped Isothermal Method (SIM) - Long-Term SIM Tests

SIM is an accelerated test method for creep in tension and creep rupture of materials (stage I) based on the TTS method. The method was first used to assess the creep behavior of geosynthetics [88,99-101], and successfully implemented to evaluate the long-term tensile strength of HDPE pipes [46,89]. Based on ASTM D6992-16 [21], SIM is performed by loading a single specimen to a certain strain level maintained at ambient temperature. Then the surrounding temperature of the HDPE specimen is increased in 7^{0}C steps for the 10,000 second dwell time until the sample yields. The creep rupture stress evolution throughout the test duration should be recorded to plot stress versus log (accelerated) time on the rupture curve for each tested specimen.

Figure 2.7a shows typical testing data generated by a single SIM test. Due to differences in the experimental approach between TTS and SIM, the analysis of SIM data implies some additional steps prior to applying the shifting procedure to generate a master curve. This is mainly attributed to the change in temperature during testing. Therefore, the measured deformation does not exclusively account for the creep behavior. Thus, a vertical shift is required to remove the deformation caused by thermal expansion while heating the sample. Consequently, the starting time of single dwell time is less than the actual starting time, t, and identified as the virtual start time, t', as in Figure 2.7b. According to the Boltzmann superposition principle [102], the creep curves at different temperature steps can be treated as individual tests starting at t'.

The value of t' is calculated in an iterative procedure starting with an arbitrary value until the slope at the beginning of a temperature step matches the slope at the end of the preceding step on the logarithmic time scale (Figure 2.7c). This procedure is repeated iteratively for all temperature steps until the SIM data rescaled in the virtual time scale is equivalent to a set of independent isothermal creep curves. A master curve employing horizontal shifting can therefore be generated (Figure 2.7d).

Yeo (2007) [89] found, however, that the analytical procedure using t' is not applicable for HDPE geogrid due to its high thermal expansion and low thermal conductivity properties. HDPE geogrid requires a much longer time to reach equilibrium than polyester geogrid at each elevated temperature step. Thus, Hsuan (2012) [103] proposed a modified procedure (MSIM) to analyze the data from SIM. MSIM accounts for t' as the same value of t. The initial nonequilibrium portion of the creep curve is removed, and the remaining creep curves are shifted horizontally and vertically to create the master curve. Thus, the resulting master curve is much shorter than that obtained with the SIM procedure using the same number of temperature steps. Hsuan (2012) [103] showed that SIM and MSIM values are basically the same, and both methods can be used to create the master creep curve for HDPE pipes.

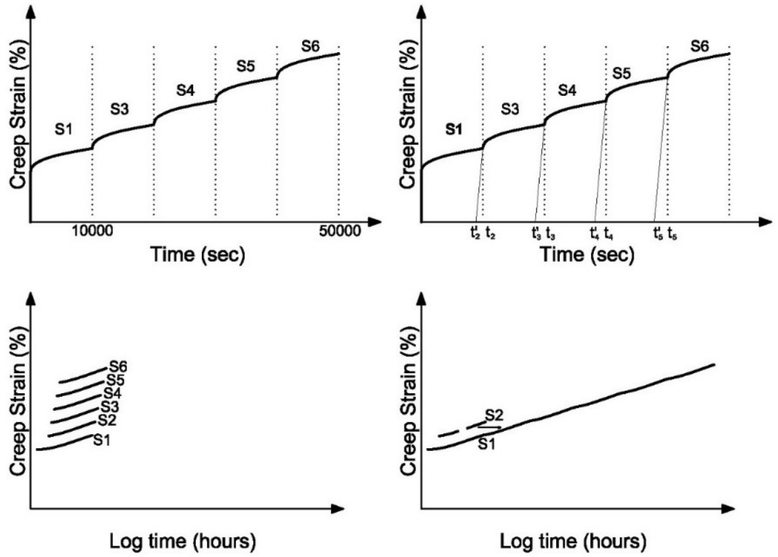


Figure 2.7 Principle of the SIM procedure: (a) raw SIM data, (b) determining virtual start time t', (c) rescaling, and (d) development of master creep rupture curve (adopted from Achereiner et al., 2013 [104])

2.7.1.5 Conventional Tests

The conventional tests are performed according to ASTM D5262-07-16 [105]. This method is quite similar to the TTS method, but the testing time is up to 1.1 years. Nevertheless, the method is also deemed representative to predict the lifetime of HDPE pipes. Yeo (2007) [89] compared the master curve between the three TTS, SIM, and conventional methods. Although there are differences in the implementation process and testing time, the data on the master curve of these tests was relatively similar.

2.7.1.6 Stepped Isostress Method (SSM)

Giannopoulos and Burgoyne (2011) [90] developed the stepped isostress method (SSM) on Kevlar 49 yarns. This method is based on the SIM, although it uses stress at the activation energy instead of temperature for TTS and SIM. High temperature is one of the factors affecting the chemical properties of materials, therefore, the SSM method avoids using this factor. The specimen undergoes a number of tensile loading steps. The average time of each test is usually from 24 hours to 1 week. The result is then analyzed to produce a master curve for predicting the long-term tensile strength of the material.

The results of SSM showed good correction between SSM, SIM, and conventional tests. Recently, Shaheen (2018) [64] used this method to predict the creep behavior of corrugated HDPE pipes manufactured with post-consumer recycled (PCR) material. This test requires only one specimen, and the test temperature is room temperature. Giannopoulos and Burgoyne (2011) [90], however, reported that the method could be subjective due to its reliance on adjusting the fitting parameters to give a smooth master curve. Furthermore, since there is no control of stress level at failure, the rupture of the material can occur at a load level near or at ultimate strength. Accordingly, the authors suggested further research to overcome these shortcomings.

2.7.1.7 Other Methods

Crystallization significantly affects the mechanical, thermal, and chemical properties of polymers. It is considered as an effective parameter of polymer mechanical properties. Another method that could be considered uses differential scanning calorimetry (DSC) measurements on the samples subjected to tensile tests as a function of elongation. As the crystallization increases upon stretching, the curve showing the relationship between the crystallinity rate or enthalpy of fusion versus elongation is produced. This test is also used to investigate the change in crystallinity rate during tensile tests.

2.7.2 Long-Term Resistance to Stress Cracking (Stage II)

2.7.2.1 Environmental Stress Crack Resistance (ESCR)

The environmental stress crack resistance (ESCR) tests are performed according to ASTM D1693-15e1 [78]. This test aims to determine the sensitivity of PE plastics to cracking under environmental stress (stage II), when subjected to different environments such as soaps, wetting agents, oils, or detergents. Under certain stress conditions and in these environments, PE plastics can exhibit mechanical failures due to cracking. Ten rectangular specimens notched longitudinally on the surface are cut, bent at 180°, and confined within the flanges of small metal. Specimens are immersed in a surface-active agent, such as nonylphenoxy poly (ethyleneoxy) ethanol (e.g., Igepal CO-630) or in other surface-active agents (soaps or any liquid organic substance) that is not significantly absorbed by the sample. Different test conditions are available depending on the specimen size, notch depth, and test temperature. After a certain time, the specimens are examined, and the proportion of broken specimens recorded. Test duration varies from 24 to 1,000 hours, depending on the specifications defined by the different HDPE manufacturers. This test is easy to perform, and broken specimens are easily observed. Significant stress relaxation occurs, however, during the test and the stress relaxation rate is difficult to quantify. For current HDPE types, this test was unable to distinguish resistance to cracking under stress. Moreover, the test results exhibited a large standard deviation [24].

2.7.2.2 Pennsylvania Notched Test (PENT)

Pennsylvania notched test (PENT) is widely used to measure the slow crack growth resistance of PE pipes according to ASTM F1473-18 [23]. Two types of notches are used: the main notch and two side notches. The notch depth depends on the thickness of the pipe wall. The specimens are tested at 80^0C in air under a single stress of 2.4 MPa. Various temperatures and stress levels can also be used. The breaking time is recorded for each sample, and a master curve can be established to extrapolate the long-term resistance. With the standard conditions (80^0C, 2.4 MPa), however, failure can take thousands of hours. As mentioned above, temperature changes and stress levels can be applied to reduce the testing time. Domínguez et al. (2012) [91] reduced the failure time by a factor of 6 when the test was conducted at 90^0C, and the applied stress was 2.8 MPa.

2.7.2.3 Notched, Constant Ligament Stress (NCLS)

The slow crack-growth resistance (SCR) of corrugated HDPE pipe can be determined with the notched, constant ligament-stress (NCLS) method based on ASTM F2136-18 [22]. This test uses dumbbell-shaped samples notched on one side. The notch depth is 20% of the specimen thickness. The test is carried out in a 10 vol.% solution of nonylphenoxy poly (ethyleneoxy) ethanol in deionized water at 50°C under the ligament stress based on the cross-sectional area of the test specimen. This test method measures the failure time associated with a given test specimen at a single temperature with a specific stress value, which makes it difficult to offset the data obtained with the PSM model.

2.7.2.4 Florida Test Method

The Florida Method of Test for Predicting the Crack Free Service Life of HDPE Corrugated Pipes [25] was first presented by Hsuan and McGrath (2005) [18] subsequent to Florida Department of Transportation (FDOT) requirements concerning the theoretical service life of 100 years for HDPE pipe. The test uses data obtained from the FM 5-572 [106] test method to assess the stress crack resistance (SCR) of HDPE pipes (stage II). FM 5-572 describes the stress crack resistance (SCR) test methods for joint and longitudinal pipe sections (Procedures B and C, respectively). Then, FM 5-573 specifies applying stresses of 650 psi (4.48 MPa) at 70^0C, and 650 and 450 psi (4.48 and 3.1 MPa) at 80^0C with five repetitions at each level of constraint. Then, the test data is shifted to form a master curve based on the RPM equation, as shown in Eq. 4.

The Florida Test Method requires that specimens be incubated in a water environment instead of the surfactant solution (10 vol.% Igepal) as in the NCLS method [22]. Hsuan and McGrath (2005) [18] examined the SCR of HDPE pipes in different exposure media (air, water, and 10% Igepal solution) and reported that the surfactant solution provided the most significant acceleration effect on stress cracking, which could not be realistic. Hsuan and Zhang (2005) [92] also confirmed that the slope of the brittle curve in the surfactant solution was much steeper and the failure time was seven times faster compared to water and air. On the other hand, tests in water and air showed similar responses [18,92]. Nevertheless, Hsuan and McGrath (2005) [18] recommended incubating the specimens in a water environment to provide homogenous temperatures during testing.

2.7.2.5 Un-Notched, Constant Ligament Stress (UCLS)

The UCLS method was first developed by Pluimer (2016) [45] to assess the stress crack resistance of HDPE materials containing recycled content. It was then publ ished as an ASTM test method ASTM F3181-16 [107] and adopted by AASHTO M 294-18 [3]. The test is similar to the NCLS test, but it is performed with large specimens to increase the number of contaminant particles in the specimen that might grow cracks. Additionally, the method follows the recommendation of Hsuan and Zhang (2005) [92] of using deionized water instead of a surfactant solution as incubation media. Furthermore, the test is performed on various un-notched specimens instead of notched specimens as in NCLS test. This was crucial for HDPE materials containing recycled content, as the crack initiation and propagation rates in polymers are dependent on the presence of contaminants or voids in the material, which are minimized in virgin material [47]. In PCR materials, the presence of randomly oriented irregular contaminants acts as stress raisers, therefore, adding artificial notching to emanant cracking would not reveal the effect of the contaminants on crack initiation.

Pluimer et al. (2018) [47] implemented full-scale creep rupture tests on HDPE pipes to validate the UCLS test results using field tests. The testing included loading four different types of HDPE pipes installed in an extremely severe conditioning environment to stresses up to 1200 psi (8.27 MPa). The whole system was installed in precast reinforced concrete chambers and loaded via dead weights attached to a lever arm assembly that applied a sustained stress to the pipes. Pipe deflections and soil pressured were monitored throughout the duration of the test. Based on the test results, Pluimer et al. (2018) [47] found that the UCLS test can accurately predict the SCR of HDPE pipes manufactured with recycled resins.

The UCLS procedure based on ASTM F3181-16 [107] is specified for corrugated HDPE pipes containing PCR materials. It can also be implemented, however, in other applications. Applying the test method to HDPE pipes produced with virgin resins might result in the material having a high UCLS failure time. As a result, the NCLS test could be more practical for such materials. Pluimer et al. (2018) [47] reported that the NCLS and UCLS tests are only related in that the materials' performance would tend to be the same with both methods.

2.7.3 Long-Term Oxidation (Stage III)

2.7.3.1 Oxidation Induction Time (OIT_{time})

The oxidation induction time (OITtime) test is a standardized test performed with differential scanning calorimetry (DSC), which measures the thermal stability according to ASTM D3895-19 [28]. This test does not specify the additives, although the antioxidant content in HDPE pipes can be measured. A small specimen placed in a DSC under inert atmosphere and heated to a determined temperature, generally 200^0C. Once the temperature stabilizes, the inert atmosphere is replaced with oxygen. After a certain time, the sample starts decomposing, which is visualized as heat-flow changes. The time between the introduction of the oxidizing atmosphere and the start of decomposition is the OITtime. In general, a reduction in OITtime over the lifetime of the HDPE pipe indicates the consumption or extraction of antioxidants. When the antioxidant is consumed, the HDPE pipe becomes susceptible to oxidative attack. This test is well recognized as one of the analytical tools used to assess the amount of antioxidants in polymers. Hsuan and McGrath (2005) [18] and Hsuan and Koerner (1998) [8] used the Arrhenius model to extrapolate the rate of antioxidant depletion from high test temperatures to lower temperatures. Then, the time required to exhaust all the antioxidants from the material was calculated.

2.7.3.2 Oxidation Induction Temperature (OIT_{temp})

The oxidation induction temperature (OIT_{temp}) test has traditionally been used on scrapings from the inner walls of thick-walled pipes after manufacturing. An oxidation induction temperature below 220^0C means that the HDPE sample has degraded during processing. The OIT_{temp} test is performed according to ASTM D3350-14 [29] by placing a small sample in a DSC and heating the test specimen in air at a rate of 20^0C/min until the polymer oxidizes. The temperature is then recorded. It should be noted that both OIT_{time} and OIT_{temp} do not reflect the long-term performance of HDPE pipes. These tests evaluate the oxidation behavior at 200^0C and 220^0C, which is not the actual aging condition of HDPE pipes [46]. Schmid et al. (2006) [93] studied the correlation between OIT_{time} and OIT_{temp} results for six different grades of PE. The results indicated that the OIT_{temp} test had a significantly low standard deviation in terms of repeatability and reproducibility than the OIT_{time} test. The ability to distinguish between different polymer samples with OIT_{temp} decreased significantly with increasing temperature. Therefore, the OIT_{time} test is recommended for recycled HDPE pipe.

2.8 Temperature Factor for Accelerated Tests

As mentioned above, high temperature is considered one of the acceleration factors in predicting the long-term properties of materials. The rate of chemical degradation increases exponentially, however, with temperature. Robert et al. (2010) [108] found that the limit for acceleration tests for vinyl-ester-based glass fiber-reinforced polymer (GFRP) bars was 60^0C. They also pointed out that these acceleration tests were valid only when the temperature alone affected the rate of degradation and complied with the Arrhenius law. It should be pointed out that there is a strong risk of excessive degradation and underestimation of the long-term performance and durability of materials when tested at high temperatures. Moreover, the structural rearrangement in semicrystalline (PE) materials creates the same effect as residual crystallization once the materials are exposed at high temperatures. To prevent this phenomenon, the upper temperature limit for testing PE materials is recommended not to exceed the softening point at 60^0C. At higher temperatures, the structure and behavior of HDPE could be modified. Consequently, there exists a significant risk of drawing erroneous conclusions from durability tests performed at these temperatures.

2.9 Discussion

As shown in earlier studies, applied stress leads to one of three types of pipe failure as a function of time and based on the stress level. These are ductile failure (stage I), brittle failure due to slow crack growth (SCG) (stage II), and failure due to chemical/molecular degradation (stage III). There are investigation methods corresponding to each stage of pipe failure. On the basis of the work presented here, the advantages and disadvantages of each testing methods are outlined:

Creep rupture strength (Stage I)

- HDB test is most popular for estimating the long-term hydrostatic strength of pipes. This method evaluates on full-scale pipes. However, the HDB test does not reflect the actual working condition of corrugated HDPE pipe for transportation infrastructure applications, where the pipe is not subjected to internal stress.
- TTS method has been in use for decades and it is known as the basis for the validation procedure for PE pipe materials in ASTM D2837 [73]. SIM method is considered a relatively new method. It has been developed since the 1990s to evaluate polymeric reinforcing materials [100]. The TTS test is conducted at various elevated temperatures on different specimens, whereas the SIM test is performed at multiple temperatures on a single specimen. The advantage of the SIM method is the short testing time (24 hours), as well as obtaining reliable results due to specimen invariability compared to the TTS method. However, the SIM method is very sensitive to the thermal expansion of the material. The TTS and SIM methods are both suitable for testing recycled pipes. In addition, the conventional test is easy to conduct, test temperature at 23^0C. However, this method is a relatively long test that can be run for thousands of hours.
- DMA TTS test is further shortening the testing time (10 hours), and easy to obtain a master curve compared to conventional and long-term TTS and SIM methods. However, the specimen size is relatively small (approx. 4×8 mm^2). The maximum strain is limited to 0.04% in flexural mode. Variability, as well as small variations in specimen size, may affect the results.

- SSM test is similar to SIM test, but it uses stress at the activation energy instead of temperature for TTS and SIM. High temperature is one of the factors affecting the chemical properties of materials. It also causes many problems in slow and non-uniform heating of thick materials. Therefore, the SSM test can be used for thermoplastic specimens with large thicknesses [109]. The advantage of the SSM method is to use only a single specimen. A reasonable time (from 24 hours to 1 week) at low-stress levels allows the obtained data to have more confidence in analyzing creep-rupture behavior compared with conventional, TTS, and SIM methods [90]. This method can be used to predict the creep behavior of corrugated HDPE pipes manufactured with post-consumer recycled (PCR) material [97]. However, the method could be subjective due to its reliance on adjusting the fitting parameters to give a smooth master curve. Furthermore, since there is no control of stress level at failure, the rupture of the material can occur at a load level near or at ultimate strength. In general terms, this method has many advantages over the other methods. Therefore, it is considered a promising method for future work.

Long-term resistance to stress cracking (Stage II)

- ESCR is performed in different exposure conditions. The advantage of this method is easy to perform and detect ruptured specimens. However, for current HDPE types, this test is unable to distinguish resistance to cracking under stress. It is known to have a large standard deviation. The stress relaxation and the stress relaxation rate are difficult to quantify.
- PENT test is widely used to measure the slow crack growth resistance of PE pipes. Flexible test that can be implemented with different temperatures and/or stress levels. The test temperature at 90^0C is considered the superior limit temperature for this test. However, in earlier studies, high temperature can change the chemical properties of materials. The test method is ambiguous regarding the transition of brittle to ductile failure mode. Furthermore, the testing time is relatively long up to thousands of hours.
- The advantage of the Florida test is performed in a water incubation environment. However, the test temperature is also relatively high at 80^0C.
- NCLS test is conducted on notched specimens with relatively short testing time. The orientation of the notch matched the majority of cracking that has occurred in the field. In contrast, the UCLS test is performed with unnotched specimens. Therefore, both the crack initiation and crack propagation phase are evaluated. The incubation environment of the NCLS test is very aggressive (solution of water and Igepal® or other surfactants), whereas deionized water is used for the UCLS test. This allows the UCLS test to have more accurately predicted the service life of pipes at the field conditions than the NCLS test. On the other hand, both these methods can be implemented with recycled HDPE resins.

Long-term oxidation (Stage III)

- Both the OIT_{time} and OIT_{temp} methods are commonly used to analyze the thermal properties of polymers. OIT_{time} is a widely used method for water pipes and outdoor applications. For pipes with low OIT values (low stabilization), the OIT_{temp} is a valuable alternative. Whereas, for OIT value longer than 20 minutes, the OIT_{time} is the appropriate choice. OIT_{temp} test had a significantly low standard deviation in terms of repeatability and reproducibility than the OIT_{time} test. The ability to distinguish between different polymer samples with OIT_{temp} decreased significantly with increasing temperature. Therefore, the OIT_{time} test is recommended for recycled HDPE pipe. However, it should be noted that these tests are not related to the long-term performance of pipes. They evaluate the oxidation behavior at 200^0C and 220^0C, which is not the actual aging condition of HDPE pipes.

2.10 Conclusions and Recommendations

This paper provided an overview of various methods for predicting the long-term service life of corrugated HDPE pipes manufactured from recycled or virgin resins in road and infrastructure applications. In such applications, the performance of pipe material is the main factor affecting pipe durability. While lifetime prediction methods were given to describe the three stages of failure mechanisms, no method describes these three failure mechanisms. This paper is intended as a reference resource for HDPE pipe engineers and researchers. They may consider which method of testing the long-term performance and durability of HDPE pipes is the most suitable. Depending on the specific requirements, such as achieving a short testing time, or obtaining accurate data with high reliability, or easily conducting tests that engineers and researchers consider these testing methods.

HDPE pipes manufactured with recycled resins possess various advantages and are expected to be widely used for road and infrastructure applications. This would not only result in saving oil and energy but would also help solve the problem of plastic-waste management and its environmental impacts. In the case of HDPE pipes, some recycled materials might even offer better properties than virgin materials, depending on the grade used. The presence of contaminants (other polymers, labels, additives, fillers, impurities, etc.) and the possible decrease in HDPE molecular weight following the reprocessing of polymer waste are factors that influence the long-term performance of recycled HDPE and must be addressed. Therefore, significant efforts must be put into improving the quality and availability of PCR HDPE by developing innovative recycling processes that yield HDPE pipes that rival the reliable performance of pipes made with virgin materials. Consequently, research is needed to investigate the structural performance and durability of HDPE pipes made with PCR materials. An integral part of future research should be the combination and development of existing and novel testing methods that can be implemented in recycled HDPE resins.

Data Availability Statement

All data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

Acknowledgements

This research received financial support from the Natural Science and Engineering Research Council of Canada (NSERC), the NSERC Research Chair in Innovative FRP Reinforcement for Sustainable Concrete Infrastructures, the Tier-1 Canada Research Chair in Composite Materials for Civil structures, the Fonds Québécois de la recherche sur la nature et les technologies (FQRNT), and the Ministry of Transportation of Quebec (MTQ).

3 CHAPITRE 3: LA COMPARAISON DES PERFORMANCES À COURT TERME DES TUYAUX ONDULÉS EN PEHD FABRIQUÉS AVEC OU SANS RÉSINES RECYCLÉES POUR LES APPLICATIONS D'INFRASTRUCTURE DE TRANSPORT

Avant-propos

Auteurs et affiliation:

Khanh Q. Nguyen: Étudiant au doctorat, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Patrice Cousin: Chercheur associé, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Khaled Mohamed: Stagiaire postdoctoral, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Mathieu Robert: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Brahim Benmokrane: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Date de publication: 22 Novembre 2021

Titre du journal: ASCE Journal of Materials in Civil Engineering

Titre en anglais: Comparing short-term performance of corrugated HDPE pipe made with or without recycled resins for transportation infrastructure applications.

Référence: K. Q. Nguyen, P. Cousin, K. Mohamed, M. Robert, and B. Benmokrane, "Comparing Short-Term Performance of Corrugated HDPE Pipe Made with or without Recycled Resins for Transportation Infrastructure Applications," *J. Mater. Civ. Eng.*, vol. 34, no. 2, p. 04021427, Feb. 2022, https://doi.org/ 10.1061/(ASCE)MT.1943-5533.0004067.

Résumé en français:

Ces dernières années, les tuyaux ondulés en polyéthylène à haute densité (PEHD) fabriqués à partir de résines recyclées se sont multipliés pour les secteurs des infrastructures en raison de leurs nombreux avantages. Par rapport aux tuyaux en PEHD fabriqués avec des résines vierges, ces tuyaux recyclés aident à résoudre le problème de la gestion des déchets plastiques et les impacts environnementaux des déchets. De plus, l'utilisation de matériaux recyclés rend les tuyaux en PEHD plus durables et plus rentables. Une question se pose : les tuyaux PEHD fabriqués avec des résines recyclées auront-ils les mêmes performances et durabilités que les tuyaux vierges sous l'impact des contraintes thermiques lors de l'enfouissement, des variations environnementales, de la charge de trafic ? Cette question doit être clarifiée, car la demande de tuyaux recyclés augmente. L'objectif de cet article est d'améliorer les connaissances pour comparer les performances à court terme de ces deux types de canalisations. Les spécimens provenaient de quatre fabricants nord-américains différents avec leurs propres processus de production. Cette étude fournit des données plus détaillées sur les propriétés physico-chimiques, mécaniques et thermiques des tuyaux en PEHD. Ces propriétés ont été testées sur des équipements de laboratoire selon les normes ASTM (American Society for Testing and Materials). Les résultats des tests peuvent être utilisés pour estimer certains aspects des caractéristiques à long terme des tuyaux en PEHD.

Mots-clés: Tuyaux ondulés en polyéthylène à haute densité (PEHD); les applications d'infrastructure de transport; les performances à court terme; les résines recyclées et vierges; les propriétés physico-chimiques, mécaniques et thermiques.

Abstract

In recent years, corrugated high-density polyethylene (HDPE) pipes manufactured from recycled resins have been on the rise for infrastructure sectors as a result of their numerous advantages. Compared to HDPE pipes made with virgin resins, these recycled pipes help solve the problem of plastic-waste management and the environmental impacts of waste. In addition, using recycled materials makes HDPE pipe more sustainable and cost-effective. One question stands out: will HDPE pipes made with recycled resins have the same performance and durability as virgin pipes under the impact of thermal stress during the burial process, environmental variations, traffic load? This issue needs to be clarified as the demand for recycled pipes is increasing. The aim of this paper is to improve the knowledge to compare the short-term performance of these two types of pipes. The specimens came from four different North American manufacturers with their own production processes. This study provides more detailed data on physico-chemical, mechanical, and thermal properties of HDPE pipes. These properties were tested on laboratory equipment according to ASTM (American Society for Testing and Materials) standards. The test results can be used to estimate some aspects of the long-term characteristics of HDPE pipes.

Keywords: Corrugated high-density polyethylene (HDPE) pipes; transportation infrastructure applications; short-term performance; recycled and virgin resins; physico-chemical, mechanical, and thermal properties.

3.1 Introduction

Pipelines play an important role in transporting gas and liquids. The pipeline systems used in transportation infrastructure sectors are often buried underground. Such underground systems—especially those for drainage and culvert structures—are constantly subjected to traffic loads as well as being exposed to groundwater or wastewater containing chemical impurities. In recent years, nonmetallic materials have been used increasingly in these sectors [6]. Unlike metals, nonmetallic materials do not undergo the same chemical corrosion. Therefore, plastics have been turned to as replacements for steel and concrete as culverts and drainage pipes. In addition, plastic (HDPE) materials are also found a better choice for floating solar systems [110,111]. HDPE materials are one of the widely used thermoplastics for these projects because of their superior physico-mechanical properties. They are highly impermeable, chemically inert, and poor conductors of electricity, while resisting biological attack [1,4]. Moreover, HDPE is light, with a density that is only 1/8 that of steel [112]. Therefore, HDPE pipe is easier to install and repair than metal pipes. HDPE pipe typically comes with a smooth interior wall that provides excellent flow capacity and a corrugated exterior surface for enhanced strength.

HDPE is widely used in packaging, construction materials, automotive applications, and everyday items such as milk and detergent containers [43,113]. This has generated a significant amount of industrial waste that can be recycled for pipe manufacturing. Pipes used for infrastructure (e.g., highway, and storm sewers) must often meet more stringent material standards than those used for agricultural drainage applications. The service-life demand for infrastructure projects is greater than for agricultural applications because pipes are often subjected to traffic loads and exposed to water containing chemical impurities. Pipes must be designed with materials that can meet the above requirements [114]. Current manufacturing standards in the pipe industry do not have a clear set of provisions governing recycled materials. In general, the amount of recycled resins is decreased during the resin recycling process. Compared to virgin resins, recycled resins experience a loss in mechanical properties leading to a reduction in their quality [115]. It is important to determine the performance of pipes made with or without recycled resins for transportation infrastructure. Many works have studied short-term performance [13,14] but most have focused on mechanical properties [15-17]. Many recent reports have investigated corrugated pipes manufactured from recycled resins for highway and rail applications [43,44,46,47,64,80,116]. So far, however, there have been no specific comparisons of the performance differences between pipes manufactured from recycled or virgin resins.

Recently, the University of Sherbrooke and Quebec's Ministry of Transportation (MTQ) decided to jointly investigate the durability of HDPE pipe made with or without recycled resins under North American climate conditions. The study's objective was to define clearer, more detailed parameters for analyzing the short-term performance of pipes made from recycled or virgin resins. It adopted an approach to define the physico-chemical, mechanical, and thermal properties of HDPE pipes so as to assess their short-term performance and determine several aspects about their long-term durability. The study also compared the short-term performance characteristics of pipes made with recycled or virgin HDPE.

3.2 Materials

The materials were corrugated HDPE pipes from four different North American manufacturers used for transportation infrastructure applications (e.g., storm drainage and storm sewers), which are applications that do not involve pressure. They were assessed in accordance with AASHTO M294 [3] *Standard Specification for Corrugated Polyethylene Pipe, 300- to 1500- mm (12- to 60-in) Diameter*. A total of six pipes were studied: two manufactured with post-consumer recycled resins (A-R, D-R), one with post-industrial recycled resins (B-R), and three with virgin resins (A-V, B-V, C-V) (Fig. 3.1). A, B, C, and D designate the manufacturers, R is for recycled resin, and V for virgin resin. Post-consumer recycled (PCR) materials are waste materials previously used by consumers and reused in new pipes. The recycling process involves shredding, washing, homogenizing, and pelletizing. Post-industrial recycled (PIR) materials are the pipe's scrap generated during the manufacturing process. The regrind pipe is produced by shredding, mixing with the raw material, and drawing by extrusion without changing the process parameters. This type of regrind can be considered as a pseudo-source of recycled resins. The external diameter, length of each pipe, and its wall thickness were 900 mm, 3000 mm, and 8 mm, respectively.

To investigate the short-term properties, small specimens were cut from the inner wall of the pipes. Specimen shapes and dimensions for each investigated test were determined based on ASTM standards and are presented in detail in the experimental process section. The investigation of the short-term properties involved three main components (a) physico-chemical properties: *density, degree of crystallinity, melt flow index (MFI), molecular weight, and carbon-black content*; (b) mechanical properties: *hardness and tensile strength at yield*; and (c) thermal properties: *softening temperature and thermal stability*.

Figure 3.1 HDPE pipes under investigation

3.3 Experimental process

3.3.1 Density

HDPE types with different degrees of branching have different densities. The degree of branching plays an important role in polyolefins. Linear polyethylene has a high degree of crystallinity and density, which makes it rigid and resistant but brittle. The gaps in certain mechanical properties can be reduced, however, by adding chains with branches.

Density affects a number of physical and mechanical properties such as tensile strength, softening temperature, and stiffness [1]. The density of HDPE is classified as type III (0.941 - 0.959 g/cm^3) and type IV (0.960 g/cm^3 and above) in accordance with ASTM D1248 [117]. There are many methods for measuring density such as ASTM D1505 [118] *Test Method for Density of Plastics by the Density Gradient Technique*, ASTM D792 [119] *Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement*, and ASTM D4883 [120] standard *Test Method for Density of Polyethylene by the Ultrasound Technique.* The density of the HDPE investigated in this study was measured according to ASTM D792 due to the availability of laboratory equipment. The procedure is as follows. The mass of a small specimen of a pipe was determined in air, and the specimen was immersed in methanol. Its apparent mass upon immersion was determined and its specific gravity calculated. This was carried out at room temperature. The test specimen was a single piece of material in any shape suitable for the testing apparatus.

3.3.2 Degree of Crystallinity

A differential scanning calorimetry (DSC 6000 from Perkin-Elmer) was used to measure the degree of crystallinity of HDPE pipes. A sample of 5-10 mg was heated from room temperature to 205^{0}C under nitrogen at a rate of 20^{0}C/min. The melting point (T_m) and heat flux (H_m) of HDPE pipes were measured. By drawing a baseline below the melting peak and determining the enthalpy of fusion from the area of this endotherm, the degree of crystallinity can be calculated using Eq. 3.1.

$$X_c = \Delta H_m^a / \Delta H_m^{100} \tag{3.1}$$

where X_c is the degree of crystallinity (%), ΔH_m^a the melting enthalpy of the sample (J/g), and ΔH_m^{100} the melting enthalpy of 100% crystalline polyethylene (287 J/g) (Hitachi High-Tech 1986).

3.3.3 Melt Flow Index (MFI)

The flowability of HDPE pipes in the molten state is determined by the melt flow index (MFI). The melt flow index of the HDPE pipes tested was determined according to ASTM D1238 [121] *Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer*. A specimen was heated inside a standard-sized cylinder for a specified period of time. Then, a certain weight is applied to the top of the piston, and the specimen is forced to melt through a standard die. The mass of the specimen extruded through the die for 10 minutes was recorded. The test was carried out at 190°C with a 2.16 kg weight on top of the piston, as in procedure A in ASTM D1238. Specimens were cut into pieces suitable for the test device.

3.3.4 Molecular Weight

An Agilent Infinity II multi-detector HT-GPC (high-temperature gel permeation chromatography) equipped with a different refractive index detector operating at 160°C was used to measure the molecular weight and its distribution. A 20 mg sample from each HDPE pipe was dissolved in 10 mL of 1, 2, 4-trichlorobenzene with a flow rate of 1 mL/min. The calibration was carried out using polystyrene standards; universal calibration was applied with the following Mark-Houwink equation (Eq. 3.2).

$$[\eta] = K.M^{\alpha} \tag{3.2}$$

where, $[\eta]$ is intrinsic viscosity, M is molecular weight, and $K = 14.1 \times 10^{5}$ (dL/g), $\alpha = 0.7$ are the viscometric parameters.

3.3.5 Carbon Black

Most HDPE pipes are manufactured with carbon black to protect them from ultraviolet (UV) degradation. Moreover, carbon black is what makes HDPE pipes black. ASTM D5805-00 [122] is used to determine the carbon-black content of HDPE pipes. According to the standard, a 5-10 mg specimen was heated from room temperature to 575°C at 35°C/min in nitrogen in a TGA analyzer (thermogravimetric analysis). Then the specimen was heated from 575°C to 900°C in oxygen. The percentage of mass loss measured after switching the gas of oxygen corresponds to the mass of carbon black.

3.3.6 Hardness

Hardness measures a material's ability to resist the penetration of a hard body. In other words, hardness is defined as the resistance to elastic deformation of the surface for some polymers. There are several common methods for measuring the hardness of materials, such as Brinell (HB), Vickers (HV), Rockwell C (HRC), and Rockwell B (HRB) [123]. A very simple and effective technique commonly used with elastomers and thermoplastics is, however, Shore hardness. The two most common scales of Shore durometer are the ASTM D2240 [26] type A (HA) and type D (HD) scales. Each scale has a value from 0 to 100: the higher the value, the harder the material. This study used a Shore D durometer to measure the hardness of the HDPE pipes. This digital durometer has an impact rod with a 30° conical point. During the analysis process, pressure was applied manually for 15 seconds. The hardness value, stabilized after 15 seconds of loading, was recorded. This procedure was repeated three times on each specimen at three different locations. For this test, a rectangular specimen with a size of 15 mm x 15 mm was used. It should be noted that the specimen thickness should not be less than 6 mm.

3.3.7 Tensile Strength

The tensile strength of HDPE pipes is evaluated according to ASTM D638 [20] *Standard Test Method for Tensile Properties of Plastics*. The specimens were punched directly onto the inner wall of the pipes using a stainless-steel die. Specimen dimensions are described in detail in the ASTM D638-14. A type IV dogbone sample was clamped on each end and a load was applied with a rate of 50 mm/min (Fig. 3.2a). This test was conducted at 23°C on an MTS universal testing machine (Fig. 3.2b); five replicates were tested. An extensometer was used to measure sample extension. Once the force and extension values had been determined, the stress–strain curve of the HDPE pipes was reported.

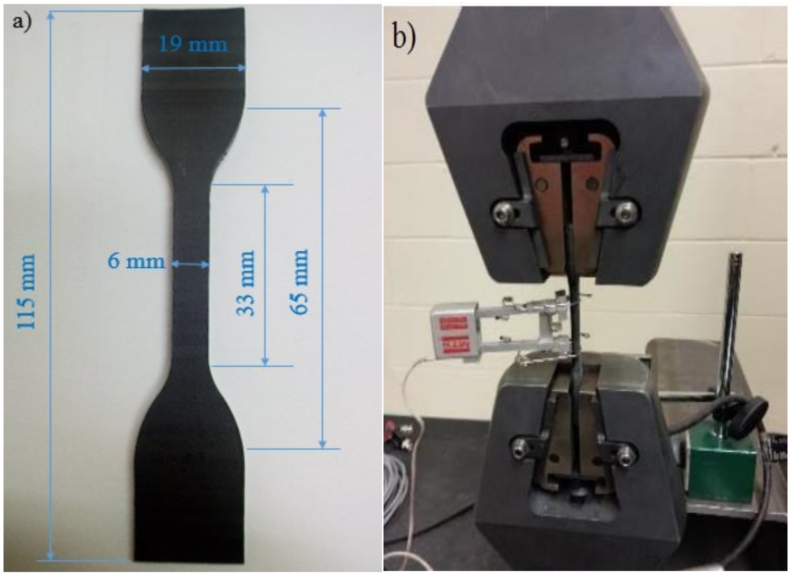


Figure 3.2 a) Dogbone sample b) MTS universal testing machine

3.3.8 Softening Temperature

The softening temperature is the temperature at which certain thermoplastics such as HDPE reach a certain degree of softening without melting. The softening temperature was measured with TMA (thermomechanical analysis) and DMA (dynamic mechanical analysis). For both methods, samples were heated from 0 to 100^0C at a rate of 5^0C/min. Samples used for TMA were cubes of 5 x 5 x 5 mm^3, while rectangular samples with a width of 3-8 mm, a length of 30-40 mm, and a thickness of 2-4 mm were used for DMA. For TMA (TA Instruments Q400), a flat-ended dilatation probe was placed on the sample in the oven. Once heated, the polymer chains in the sample become more mobile, increasing the material's coefficient of thermal expansion. The temperature at which this change takes place is the softening temperature. For DMA (Perkin-Elmer DMA 8000), the conservation and loss moduli (E' and E") are presented. The softening temperature is the onset of the E' curve or is the maximum point of the E" curve. These two values can be different, hence the need to clearly identify how they were measured.

3.3.9 Thermal Stability

The thermal stability of HDPE pipes is closely linked to carbon-black content. The carbon black helps improve the material's thermal stability [124,125]. The thermal stability of HDPE pipes is measured by TGA (thermogravimetric analysis) in accordance with ASTM E2550 [126]. The mass of a sample was measured as a function of time and temperature, which makes it possible to assess the material's thermal stability. For this test, a specimen of 5-10 mg was heated from room temperature to 650°C at a rate of 5°C/min under nitrogen.

3.4 Results and Discussions

3.4.1 Physico-chemical Properties

Table 3.1 shows the density of the HDPE pipes, measured according to ASTM D792 [119] and as the average value of three experiments. In this specific case, water could not be used since it has a higher density than PE. Methanol was used instead.

Table 3.1 HDPE pipe density

Specimen	Density (g/cm^3)	Corrected Density (g/cm^3)
A-R	0.988	0.975
A-V	0.977	0.964
B-R	0.976	0.963
B-V	0.987	0.976
C-V	0.976	0.965
D-R	0.964	0.950

The addition of carbon black, other colorants, or stabilizers protect HDPE pipes from ultraviolet (UV) degradation and increase the material's density. The addition of 1% carbon black increases the density of HDPE by 0.0044 g/cm^3. If the carbon black contained in HDPE pipes is known, an equation (ASTM D3350 [29]) can be used to determine the base resin density (Eq. 3.3):

$$D_r = D_p - 0.0044C \tag{3.3}$$

where D_r is the base resin density (g/cm^3); D_p is the compound density, including carbon black (g/cm^3); and C is the weight percent of carbon black.

Table 1 shows the density values before and after correction for carbon-black content. The results indicate that there is no significant difference in density between these HDPE pipes. It is important to know that the specification of density measurement is to be made with or without the carbon black.

The degree of crystallinity greatly not only affects the physical properties but also the mechanical properties of semicrystalline polymers [127]. Increasing the degree of crystallinity increases the hardness, density, thermal stability, and strength of materials, but lowers the impact resistance [128]. Table 3.2 displays the melting point, enthalpy of fusion, and degree of crystallinity of

HDPE pipes. For the same manufacturer, the melting point of virgin and recycled pipes is similar. However, the enthalpy of fusion, e.g., the degree of crystallinity, is different. In particular, this value is relatively low for pipes from manufacturer B (B-R, B-V) compared to pipes from other manufacturers. Moreover, the crystallinity of virgin pipes is higher than recycled pipes. It should be pointed out that the manufacturing process and properties of the resin play a key role in this difference. Mixing resins with different characteristics (molecular weight, chain branching, additives, contaminants) from several sources makes the recycled material less homogenous and therefore, less susceptible to crystallization. However, a polymer from a single source will be more uniform, which will facilitate the crystallization process [129].

Table 3.2 Degree of crystallinity of the HDPE pipes

Specimen	Melting Point (^{0}C)	Enthalpy of Fusion (J/g)	Degree of Crystallinity (%)
A-R	153	135	47
A-V	153	139	49
B-R	143	102	36
B-V	144	132	46
C-V	151	151	53
D-R	148	149	52

The melt flow index (MFI) is one of the most common parameters identifying two important properties for HDPE pipes: processability and molecular weight of PE resin [1]. A higher melt flow index indicates a lower melt viscosity and a lower molecular weight of materials. The MFI value affects some physical and mechanical properties such as tensile strength and creep resistance [130]. It also affects the thermal degradation of the materials. A lower melt flow index and higher activation energy are required for the thermal degradation of materials [131]. Table 3.3 shows the melt flow index (MFI) values of the HDPE pipes studied. The results reveal no significant differences in MFI value between the HDPE pipes made from recycled or virgin resin. This is similar to the findings in past studies. Alzerreca et al. (2015) [43] also found no significant difference between the recycled and virgin HDPE pipes used in gravity sewer systems, although the pipes were produced with recycled resins from different sources.

Table 3.3 Melt flow index of the HDPE pipes

Specimen	MFI (g/10 min)
A-R	0.103
A-V	0.077
B-R	0.066
B-V	0.061
C-V	0.115
D-R	0.058

Molecular weight is one of the factors affecting the mechanical performance of the material. Long-term strength and stress-crack resistance improve as molecular weight increases. The number average (M_n) and weight average (M_w) are usually expressed as average molecular weight. Fig. 3.3 provides molecular-weight distribution (MWD) curves for the HDPE samples. Table 3.4 gives the corresponding average molecular weight and polydispersity (M_w/M_n). A significant difference in MWD between the pipes is reported. This difference in MWD is highly dependent on the manufacturing process of the polyethylene resin. In addition, the change in molecular-weight distribution can explain the difference in the melt flow index (MFI). Materials with a low molecular weight have a high melt flow index, which might explain why sample C-V had the highest melt flow index of the pipes tested.

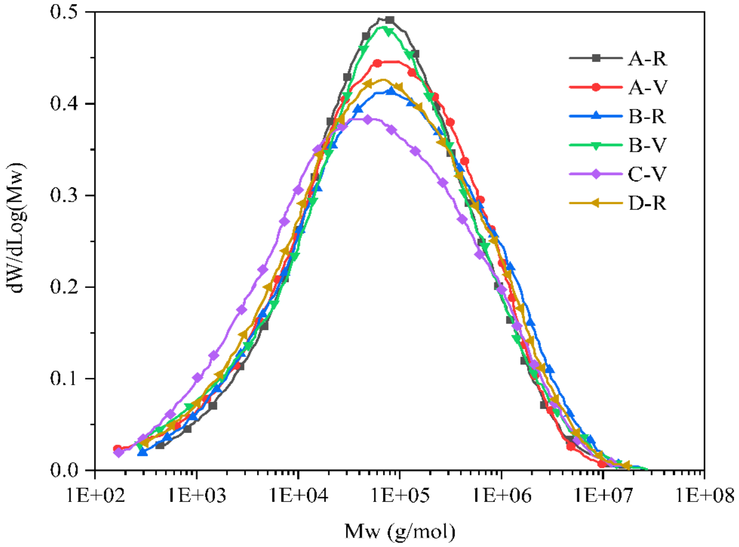


Figure 3.3 Molecular-weight distribution curves of HDPE pipes

Table 3.4 Average molecular weight and polydispersity of the HDPE pipes

Specimen	$10^{-3}M_n$ ($gmol^{-1}$)	$10^{-3}M_w$ ($gmol^{-1}$)	M_w/M_n
A-R	11.9	405	34
A-V	7.3	323	44
B-R	9.5	542	57
B-V	8.3	413	50
C-V	5.4	449	83
D-R	9.0	392	43

Carbon black is a major component that makes HDPE pipes black. Carbon black plays an important role in improving thermal stability and UV resistance. In the case of the thermal decomposition of carbon-black composites with polyethylene, carbon black of low volatile content increases thermal stability [132]. Carbon-black distribution also affects the degradation of mechanical properties. HDPE pipes with similar carbon-black concentrations but different carbon-black distributions affect pipe performance. Deveci et al. (2018) [133] studied the effect of carbon-black distribution on the degradation of post-yield mechanical properties and fracture-surface analyses of polyethylene pipe with different carbon-black contents down to zero. The tensile-test results showed that the pipes had the same yield properties, but significantly different post-yield properties. Those with carbon black had both ductile and brittle fracture, but those without carbon black experienced only ductile fracture. Other researchers have shown that incorporating 2% to 4% carbon black plays a positive role in HDPE-pipe durability [19,134]. Table 3.5 provides the carbon-black contents of the HDPE pipes tested, which ranged from 2.5% to 3.2% and are consistent with standards. The results show that the carbon black content in the recycled pipe is higher than that in the virgin pipe. It appears that carbon black is mostly added to recycled resins for aesthetic reasons to homogenize the color of the mixtures. As the recycled resins, mixing lots of colors, the final color is often brown or grey. Therefore, the addition of carbon black makes the whole materials black [135,136]. Carbon black has a small effect on the mechanical properties of HDPE. However, carbon black could affect the elastic limit by changing the mode of deformation. Two modes are typical, either by chain shearing, requiring less energy or by crystal fractionation. The addition of carbon black makes the sliding of the chains more difficult. The deformation is favored by the breaking of the small crystals. As this mode requires more energy than shearing, the elastic limit can be increased [135-138].

Table 3.5 Carbon black content, hardness, tensile strength, elastic modulus, and thermal stability of HDPE pipes

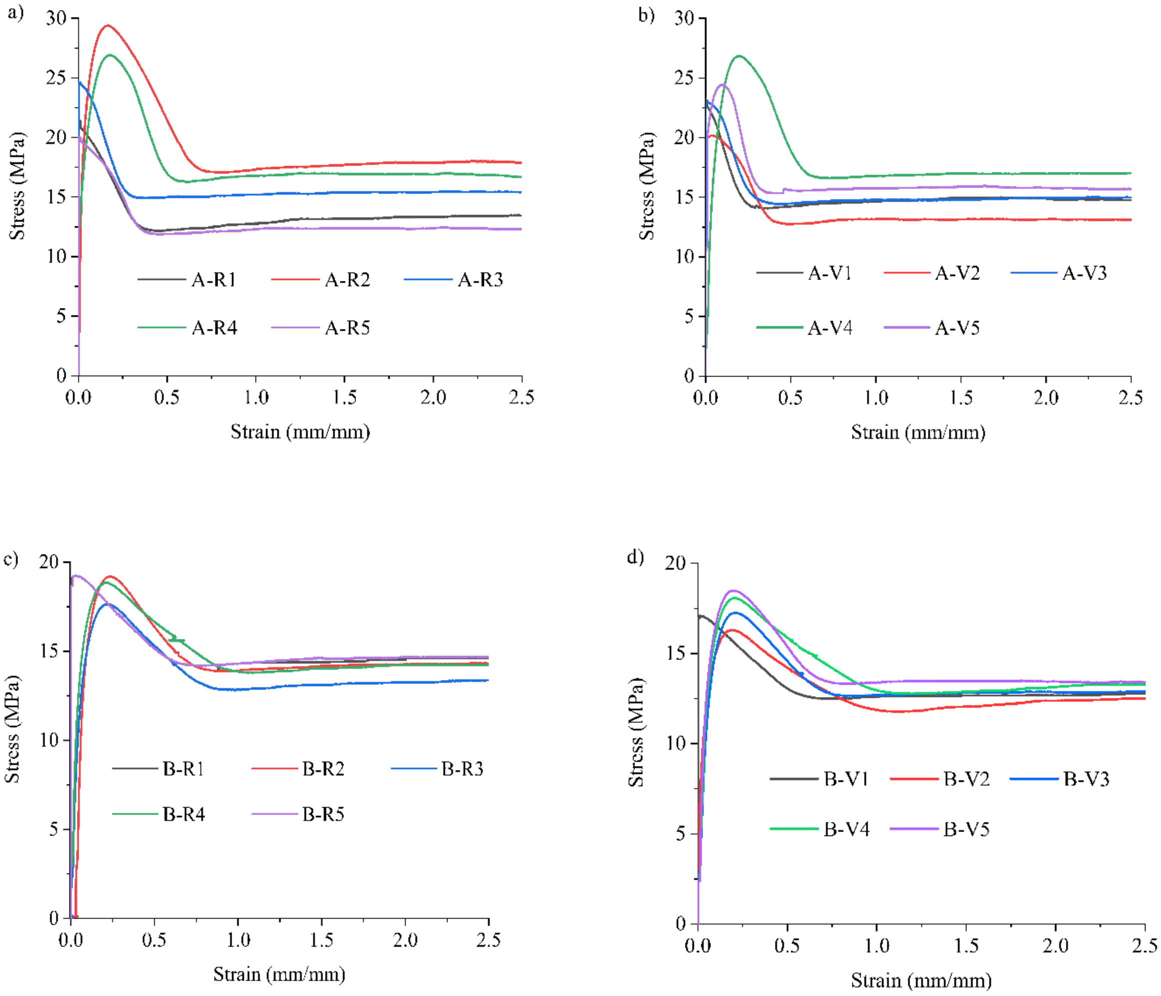
Specimen	Carbon-Black Content (%)	Hardness (HD)	Tensile Strength (MPa)	Elastic Modulus (MPa)	Mass Loss (^{0}C)
A-R	3.0	56	25.12	525	390
A-V	2.8	55	23.58	393	375
B-R	3.0	55	18.83	236	415
B-V	2.5	60	17.44	219	420
C-V	2.5	65	19.81	424	413
D-R	3.2	61	22.56	390	413

3.4.2 Mechanical Properties

Table 3.5 gives the hardness of the HDPE pipes, which was measured with a Shore D durometer. The range of 55 to 65 HD is not a significant difference between the virgin and recycled HDPE pipes. HDPE is a semicrystalline polymer, so the factors affect its hardness of act mainly in the crystalline region. In fact, polymer chains in the crystalline region are denser than in the amorphous region [139]. That accounts for the slight differences in hardness values between the HDPE pipes tested. In other words, the differences were caused by variations in the contents of the crystalline phase.

Figs. 3.4 (a, b, c, d, e, and f) provide the stress–strain curves for HDPE pipes; five replicates were tested for each pipe. The figures show significant differences in the tensile strength. As illustrated, the stress–strain behavior of the pipes was governed by 3 regions: (1) elastic (linear and nonlinear), (2) neck propagation, and (3) plastic regions. The initial portion of the stress–strain curve is defined as the elastic deformation region where the elastic modulus is defined as the slope of the stress-strain curve. The elastic modulus values are determined as shown in Table

3.5. The maximum point on the stress–strain curve corresponds to localized narrowing of the sample. This point is called the yield point. The area corresponding to the reduced stress value is often called a neck. The area with relatively stable stress values is the development phase of the neck. Fig. 3.5 shows the average values of tensile strength at yield of the HDPE pipes with values ranging from 17.44 to 25.12 MPa, and the vertical bars represented the standard deviation. This value was relatively low for samples B-R and B-V compared to the other samples tested. Moreover, the values were relatively similar with pipes made with recycled or virgin resins by the same manufacturer. This demonstrates that the pipes made with recycled resins had slightly higher tensile strength than those made with virgin resin. It can be accounted for by the carbon-black content, which improved the initial mechanical properties. In addition, the tensile-strength values of materials are closely related to their chemical bonds and microstructure. In other words, the yield strength depends on the movement of dislocations and how they interact with the second phase and with each other [140]. The composition, grain size, and extent of work hardening are factors that influence dislocation movement. It should be pointed out that the significant difference in tensile strength between pipes is closely related to the manufacturing process and properties of the resin [141,142].



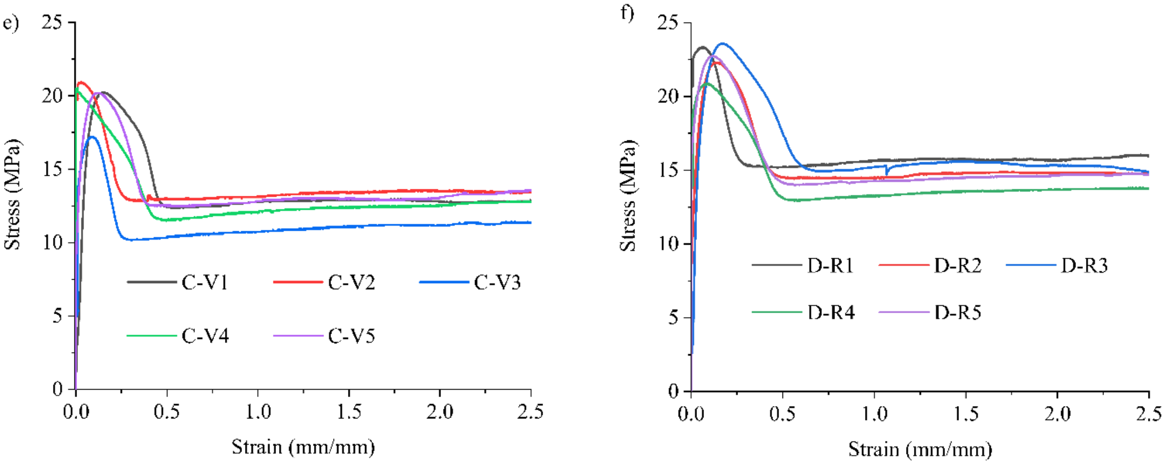


Figure 3.4 Tensile test of HDPE pipes a) A-R b) A-V c) B-R d) B-V e) C-V f) D-R

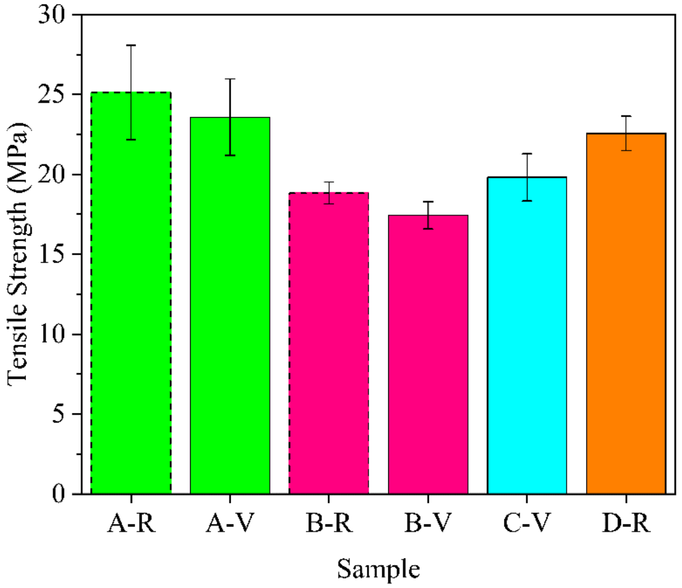


Figure 3.5 Average values of tensile strength at yield of the HDPE pipes

3.4.3 Thermal Properties

In general, the softening temperature does not provide any information about a polymer's performance. A low softening temperature, however, can indicate poor performance at high temperatures. In other words, a higher softening temperature equates to a harder polymer. This confirms that a higher softening point indicates lower temperature sensitivity. Both TMA and DMA can measure the softening temperature. Different techniques provide different values. A constant static force is applied in TMA; it monitors changes in the material such as temperature and deformation. DMA applies an oscillatory force to the sample at a defined frequency and reports changes in stiffness and damping. This method also can give the elasticity modulus. In this study, these values were reported at 0^0C and 100^0C. Table 3.6 shows the softening temperature values of the HDPE pipes, revealing no significant differences in softening-temperature values determined with the two methods. These values are similar for virgin and recycled resins.

Table 3.6 The softening temperature of HDPE pipes measured by DMA 8000, TMA Q400

Specimen	Elasticity Modulus		Softening Temperature by DMA (°C) (for E'')	Softening Temperature by TMA (°C)
	0°C (MPa)	100°C (MPa)		
A-R	1070	196	54	51
A-V	846	147	52	50
B-R	927	129	45	45
B-V	953	132	45	45
C-V	1226	211	52	45
D-R	910	145	58	54

As mentioned above, carbon black improves pipe thermal stability. The pipes tested contained the same amount of carbon black, so their thermal stability is similar (Figs. 3.6a and 3.6b). Table 3.5 shows no significant differences in the rate of oxidation of the tested samples. In addition, the thermal stability of the HDPE pipes is only slightly affected by resin type.

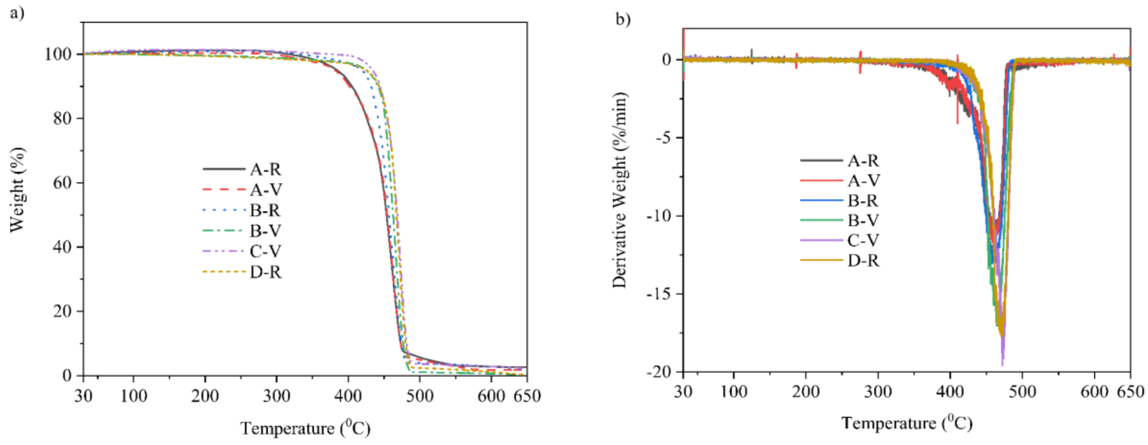


Figure 3.6 a) TGA curves of the HDPE pipes b) d(TGA)/dT curves of the HDPE pipes

3.5 Conclusions

This paper reports on a joint study into the short-term performance of six corrugated HDPE pipes (A-R, A-V, B-R, B-V, C-V, and D-R) manufactured from recycled or virgin resins that investigated physico-chemical, mechanical, and thermal properties. The pipes had a density greater than 0.941 g/cm^3 (corrected for carbon black). The relative carbon-black content is the same in the HDPE pipes made with recycled or virgin resin. The results show that these pipes had the correct amount of carbon black (2% to 4%). Sample C-V exhibited a significant difference in molecular-weight distribution, which also explains why this pipe had the highest melt flow index of the pipes tested. The pipes had the same hardness. In terms of thermal stability, the onset temperature is around 400^{0}C, and the softening temperature is around 50^{0}C. For the tensile test, the stress–strain behavior of the HDPE pipes is governed by 3 regions (1) elastic deformation (linear and nonlinear), (2) neck propagation, and (3) plastic deformation. The results show that the pipes made with recycled resins have slightly higher tensile strength than those made with virgin resin. It can be accounted for by the carbon-black content, which improved the initial mechanical properties. Samples B-R and B-V offer a relatively low tensile strength compared to other pipes tested. However, there are no significant differences between pipes made of recycled or virgin resin from the same manufacturers. Many types of HDPE are available and the number of recyclates with different properties is infinite. Assume that a recycled material will offer lower properties than a material made of virgin material without having tested the materials, is therefore wrong. In the present study, it may be concluded that the short-term performance of pipes made with recycled HDPE is similar to that made with virgin HDPE. It should be noted that many parameters of pipe manufacturing, which could possibly explain certain characteristics or differences in the properties of these materials, are production secret and therefore not available for the authors. A difficult problem still exists is how to ensure the production of recycled pipes meets standards. However, manufacturers can improve product performance based on the super-clean recycling process for highly volatile compounds. In addition, it is necessary to establish the principles for certification standards to monitor recycling content through chemical recycling processes. Based on the results obtained in the present study, the recycled HDPE promises to be one of the widely used materials in the future. Recycled HDPE should be considered for floating solar applications. The recycled HDPE composite materials should be interested in future research such as recycled HDPE clay nanocomposite for dielectric applications, concrete reinforced with recycled HDPE plastic fibers for building application.

Data Availability Statement

All data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

Acknowledgments

This research received financial support from the Natural Science and Engineering Research Council of Canada (NSERC), the NSERC Research Chair in Innovative FRP Reinforcement for Sustainable Concrete Infrastructures, the Tier-1 Canada Research Chair in Composite Materials for Civil structures, the Fonds Québécois de la recherche sur la nature et les technologies (FQRNT), the Ministry of Transportation of Quebec (MTQ), and the University of Sherbrooke Research Centre on Composite Materials (CRUSMaC). The authors are also grateful to the technical staff of the structural laboratory at the University of Sherbrooke, especially Jérôme Lacroix and Steven MacEachern, for their technical assistance.

4 CHAPITRE 4: EFFETS DU RAYONNEMENT ULTRAVIOLET SUR LES TUYAUX ONDULÉS EN PEHD RECYCLÉS ET VIERGES UTILISÉS DANS LES SYSTÈMES DE DRAINAGE-ROUTIER.

Avant-propos

Auteurs et affiliation:

Khanh Q. Nguyen: Étudiant au doctorat, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Patrice Cousin: Chercheur associé, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Khaled Mohamed: Stagiaire postdoctoral, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Mathieu Robert: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Adel El-Safty: Professeur, Département de génie civil, Université de North Florida, Jacksonville, Florida, USA.

Brahim Benmokrane: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Date de soumission: 12 octobre 2021

Titre du journal: Journal of Polymers and the Environment

Titre en anglais: Effects of Ultraviolet Radiation on Recycled and Virgin HDPE Corrugated Pipes Used in Road Drainage Systems.

Résumé en français:

Le tuyau en polyéthylène à haute densité (PEHD) est l'un des matériaux d'intérêt pour utiliser dans les systèmes de drainage routier. La combinaison du rayonnement ultraviolet (UV), de la température et de l'humidité peut produire des points faibles et entraîner une dégradation des tuyaux pendant le processus de stockage, d'installation et de réparation. L'objectif de cette étude était d'évaluer les changements dans la structure chimique, morphologique et thermomécanique des tuyaux recyclés et vierges soumis à une exposition aux UV. Des essais de vieillissement accéléré en laboratoire ont été menés en exposant les tuyaux aux UV pendant 3600 heures avec une irradiance de 0,89 W/(m2 nm) à une longueur d'onde de 340 nm. Un cycle de 12 heures, composé de 8 heures de rayonnement UV à 60°C et de 4 heures d'absence de rayonnement UV à 50°C correspondant à l'absence de condensation d'eau, a été réalisé pour conditionner les éprouvettes. Des échantillons de PEHD ont été prélevés après 3600 heures et analysés par FTIR (spectroscopie infrarouge à transformée de Fourier), SEM (microscopie électronique à balayage), DSC (calorimétrie différentielle à balayage), mesures du temps d'induction oxydative (OIT) et tests de traction. Les résultats montrent que les tuyaux recyclés conservent de bonnes propriétés et ne sont pas significativement affectés par le rayonnement UV, de la même manière que les tuyaux vierges. Une analyse statistique utilisant une analyse de variance à un facteur (ANOVA) montre qu'il n'y avait pas de différence significative entre les mesures de résistance à la traction, de module d'élasticité et de dureté avant et après l'exposition aux UV. Il n'y avait que quelques petits changements dans la surface des tuyaux. L'ajout de noir de carbone, d'antioxydants et de stabilisants UV a empêché un vieillissement supplémentaire des tuyaux pendant l'exposition aux UV.

Mots-clés: Tuyau en polyéthylène à haute densité (PEHD); tuyaux recyclés et vierges; vieillissement UV; systèmes de drainage-routier; propriétés chimiques, morphologiques et thermomécaniques.

Abstract

High-density polyethylene (HDPE) pipe is one of the materials of interest for use in road drainage systems. The combination of ultraviolet (UV) light, temperature, and moisture can produce weak spots and lead to pipe degradation during the storage, installation, and repair process. The objective of this study was to evaluate changes in the chemical, morphological structure, and thermomechanical properties of recycled and virgin pipes under UV exposure. Laboratory accelerated aging tests were conducted by exposing pipes to UV for 3600 hours with an irradiance of 0.89 W/(m^2 nm) at a wavelength of 340 nm. A cycle of 12 hours—comprised of 8 hours of UV radiation at 60°C and 4 hours of no UV radiation at 50°C corresponding to no water condensation—was performed to condition the specimens. HDPE specimens were taken out after 3600 hours and analyzed with FTIR (Fourier-transform infrared spectroscopy), SEM (scanning electron microscopy), DSC (differential scanning calorimetry), oxidative-induction time (OIT) measurements, and tensile tests. The results show that the recycled pipes maintained good properties and were not significantly affected by UV radiation, similarly to the virgin pipes. Statistical analysis using one-way analysis of variance (ANOVA) shows that there was no significant difference between tensile strength, elastic modulus, and hardness measurements before and after UV exposure. There were only a few small changes in the surface of the pipes. The addition of carbon black, antioxidants, and UV stabilizers prevented further aging of the pipes during UV exposure.

Keywords: High-density polyethylene (HDPE) pipe; recycled and virgin pipes; UV aging; road drainage systems; chemical, morphological structure, and thermomechanical properties.

4.1 Introduction

In recent years, thermoplastic pipes have become one of the most widely used materials for culverts and other highway applications [143]. High-density polyethylene (HDPE) is one of the materials of interest for these applications due to its numerous advantages. HDPE pipes are generally lighter, more flexible, and easier to install than other types of pipes as well as involving entail low maintenance costs and greater resistance to chemical attack [33-36,116]. Once the manufacturing process has been completed, however, pipes are often stored outdoors for a period of time before being installed. During the storage, installation, and repair process, the combination of ultraviolet (UV) light, temperature, and moisture can produce weak spots and lead to pipe degradation. The presence of impurities during the manufacturing process causes the pipes to absorb UV radiation. Since PE materials only contain C–C and C–H bonds, they are considered to be easily susceptible to degradation when exposed to high temperatures and UV radiation [144]. Culverts used in road drainage systems incur damage to their end openings due to UV radiation [37,67,144]. UV degradation can alter the physicomechanical, chemical, and macromolecular structure of the polymer [1,145]. This degradation can alter color, tensile strength, elastic modulus, and impact strength of the materials [67,146]. The presence of free radicals leads to chain scission, which usually takes place in the amorphous phase and at the amorphous-lamellar interface [147], whereas the crystalline phase remains inert [148]. In fact, UV stabilizers such as carbon black are integrated into pipes to prevent UV-induced reactions. In general, BNQ 3624-120 (2016) [19] and ASTM D4218 (2015) [134] require a minimum of 2% to 4% carbon black in pipes. Carbon black helps protect pipes against UV-radiation damage by limiting penetration to the external surface of the pipe wall (corrugated parts).

Attwood et al. (2006) studied the effects of UV degradation on recycled polyolefin blends. Tests were performed using QUV accelerated weathering testers with an irradiance of 0.68 w/m^2 at 340 nm. The process included 8 hours of UV radiation at 60^0C and 4 hours of condensation at 50°C. Test exposure time was 1000 to 5000 hours. The results showed that UV radiation had little effect on tensile, impact, and chemical properties; melt flow tests; gloss; and color analyses. Some surface changes were, however, observed [149].

Maria et al. (2011) studied the impact of UV radiation on PE pipes under IR-microscopy. The samples tested contained a phenolic stabilizer (Irganox 1010) as a primary antioxidant, a processing stabilizer (Irgafos 168), carbon black, and UV stabilizers. The specimens were exposed to UV radiation for 144 to 2208 hours. IR-microscopy and OIT measurements revealed loss of the phenolic antioxidant Irganox 1010 in the outer pipe wall. No changes in the degree of crystallinity were noted [150].

Jassim et al. (2017) evaluated the tensile strength at break of medium-density polyethylene (MDPE) water pipes made with and without carbon black after exposure to UV radiation for 200 hours. The results showed that the tensile strength at break for MDPE pipe made with carbon black was higher that of than virgin MDPE pipe. The tensile strength at break after exposure to UV radiation was unchanged, however, due to the carbon black playing a key role as a UV stabilizer [151].

Jiang et al. (2019) investigated the influence of UV absorbers on the UV resistance of HDPE. They concluded that the HDPE samples maintained their thermal and mechanical properties after exposure to UV irradiation for 600 hours under the aging condition of 0.51 w/m^2 and a wavelength of 350 nm. In addition, slight changes in crystallinity were noted before and after exposure to UV radiation [152].

The incorporation of UV stabilizers, carbon black, and antioxidants in the polymer matrix helps limit the impact of UV radiation on the properties of materials. Past studies, however, have focused on HDPE films or HDPE water pipes. In contrast, very little has been developed about the effect of UV radiation on corrugated HDPE pipes used in road drainage systems. In addition, the current use of recycled HDPE pipes is of interest due to their sustainability and cost-effectiveness as compared to virgin pipes [45-47,64,116]. Recycling products can reduce the carbon footprint, the use of raw materials, energy requirements, water consumption, and greenhouse-gas emissions in the production process [60,62]. The main objective of our study was to assess the effect of UV radiation on the properties of recycled and virgin corrugated HDPE pipes used in road drainage systems. The techniques FTIR (Fourier-transform infrared spectroscopy) and SEM (scanning electron microscopy) were used to investigate the formation of degradation and any changes in pipe-wall morphological parameters. The thermal properties and crystallinity of the HDPE pipes were assessed with DSC (differential scanning calorimetry). The OIT measurements were conducted to profile antioxidant concentrations. In addition, the mechanical properties of the recycled and virgin HDPE corrugated pipes—such as tensile strength and hardness—were investigated before and after accelerated UV exposure.

This study is a part of ongoing research between the University of Sherbrooke and Quebec's Ministry of Transportation (MTQ) to jointly investigate the short- and long-term performance of new corrugated HDPE pipes made with or without recycled resins for use in road drainage systems under North American climate conditions. The study included six new corrugated HDPE pipes from three different North American manufacturers, including three HDPE pipes made with virgin resins and three HDPE pipes made with recycled resins.

4.2 Experimental methods

4.2.1 Material

Six new corrugated HDPE pipes used in non-pressure road drainage systems (e.g., storm drainage and storm sewers) were provided by North American manufacturers. The pipes contained 2% to 4% carbon black: two were manufactured with post-consumer recycled resins (A-R and D-R); one with post-industrial recycled resins (B-R); and three with virgin resins (A-V, B-V, C-V). The letters A, B, C, and D designate the manufacturers; R stands for recycled resin and V for virgin resin. Post-consumer recycled (PCR) materials are waste materials discarded by consumers and reused in new pipes. The recycling process involves shredding, washing, homogenizing, and pelletizing. Post-industrial recycled (PIR) materials are pipe scrap generated during the manufacturing process. Regrind pipe is produced by shredding, mixing with the raw material, and drawing by extrusion without changing the process parameters. This type of regrind can be considered a pseudo-source of recycled resins. Each pipe measured 900 mm in diameter and 3000 mm in length. Table 4.1 provides the pipe properties.

Table 4.1 Properties of the investigated HDPE pipes

Property	Method (ASTM)	Specimen					
		A-R	A-V	B-R	B-V	C-V	D-R
Density (g/cm^3)	D792-13 [119]	0.988	0.977	0.976	0.987	0.976	0.964
MFI (g/10 min)	D1238-13 [121]	0.103	0.077	0.066	0.061	0.115	0.058
10^{-3} M_n molecular weight (gmol^{-1})	-	11.9	7.3	9.5	8.3	5.4	9.0
10^{-3} M_w molecular weight (gmol^{-1})	-	405	323	542	413	449	392
CB (%) content	D5805-00-19 [122]	3.0	2.8	3.0	2.5	2.5	3.2
Hardness (HD)	-	56	55	55	60	65	61
Softening temperature by DMA	-	54	52	45	45	52	58
Softening temperature by TMA	-	51	50	45	45	45	54
Mass loss (°C)	E2550-17 [126]	390	375	415	420	413	413
Tensile strength (MPa)	D638-14 [20]	25.12	23.58	18.83	17.44	19.81	22.56

Note: MFI = Melt flow index; CB = carbon black; DMA = dynamic mechanical analysis; TMA = thermomechanical analysis, ASTM = American Society for Testing and Materials.

4.2.2 Specimen preparation

Small specimens (15 mm x 15 mm) were cut from the corrugated part of the pipes with the exception of tensile specimens. To avoid bending due to the shape of the corrugated part and facilitate cutting, the tensile specimens were taken from the pipe liner (Fig. 4.1). The specimens cut from the corrugated parts of pipes A-R, A-V, B-R, B-V, C-V, and D-R were 7.30, 7.30, 7.80, 7.80, 3.25, and 4.50 mm thick, respectively. The tensile specimens cut from pipes A-R, A-V, B-R, B-V, C-V, D-R were 4.80, 4.40, 7.50, 7.60, 2.70, and 3.80 mm thick, respectively. The sections below provide details about specimen shape and dimensions.

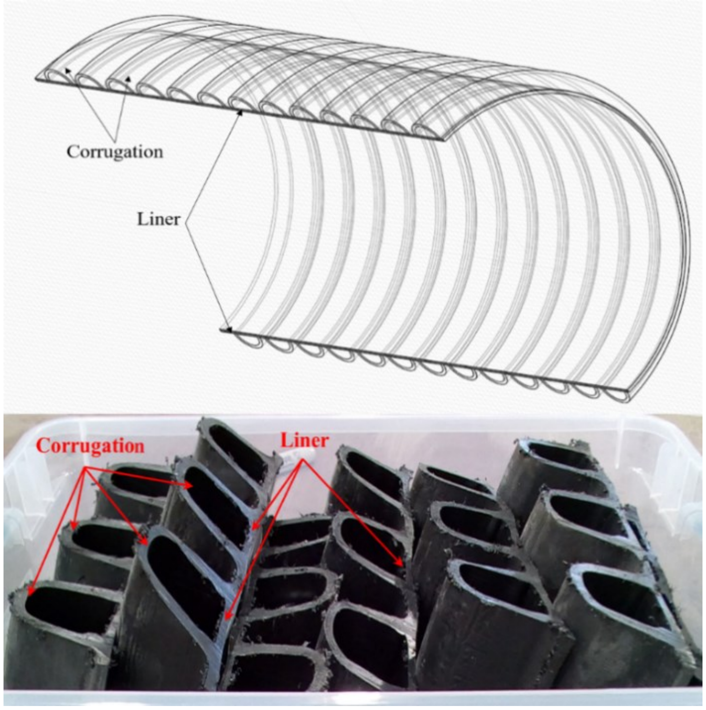


Figure 4.1 A sectional view of an HDPE pipe showing the location of the corrugation and liner.

4.2.3 UV aging

Specimens were placed under fluorescent UVA in a test chamber that simulates the spectral irradiance of daylight, as shown in Fig. 4.2. To reduce the effect of humidity on the UV aging of the pipes, the cycles for irradiance of 0.89 W/(m^2 nm) at a wavelength of 340 nm in accordance with ASTM D4329, procedure A [153], were modified in our study. Specimen conditioning consisted of a 12-hour cycle, comprised of 8 hours of UV radiation at 60^{0}C and 4 hours with no UV radiation at 50°C, corresponding to no water condensation. The text exposure time was 3600 hours.

Figure 4.2 Specimens placed for aging in the UV chamber.

4.2.4 Spectroscopy analysis

Fourier-transform infrared spectroscopy (FTIR-4600 spectrometer) was used to assess the changes in chemical structure in the HDPE pipes before and after exposure to UV radiation. With the device in attenuated total reflection (ATR) mode, a single spectrum of 32 scans was recorded at a resolution of 4 cm^{-1}. A rectangular specimen (15 mm x 15 mm) was analyzed over the 4000 to 1000 cm^{-1} wavenumber range. The test specimen thickness corresponding to the pipe wall thickness (corrugated part) was 7.30, 7.30, 7.80, 7.80, 3.25, and 4.50 mm for A-R, A-V, B-R, B-V, C-V, and D-R, respectively. The FTIR spectra of the specimens before UV exposure are considered the reference spectra. The surface spectra in direct (top) and nondirect (bottom) UV exposure through the specimen thickness were analyzed and compared to the reference spectra.

4.2.5 Microscopy observation

Scanning electron microscope (SEM) observations were carried out on a Hitachi S-4700 at a voltage of 5 kV. Our study investigated the surface morphologies of unexposed and UV-exposed HDPE specimens (15 mm x 15 mm). The specimens received a palladium–gold coating prior to analysis to prevent charging. Moreover, energy dispersive spectrometry (EDS) was then conducted to study the chemical composition of certain pipes.

4.2.6 Thermal analysis

The melting point and melting enthalpy of the HDPE pipes were determined with a differential scanning calorimetry device (DSC 6000 from Perkin-Elmer). Specimens of 5 to 10 mg were cut from both unexposed and UV-exposed HDPE pipe specimens. The measurement was performed under nitrogen at a scanning rate of 20°C/min from 30°C to 205°C. The melting point and melting enthalpy were determined by drawing a baseline below the melting peak. The degree of crystallinity was calculated from the ratio of the melting enthalpy of the specimens to the melting enthalpy of 100% crystalline polyethylene (287 J/g) [154].

The OIT of the unexposed and UV-exposed HDPE pipe specimens was measured with differential scanning calorimetry (DSC 6000 by Perkin-Elmer) in accordance with ASTM D3895 (2019) [28]. A 5 to 10 mg specimen was heated from room temperature to 200°C at a rate of 30°C/min under nitrogen. After an isotherm of 1 minute, the gas was switched to oxygen. After some time, an exothermic peak appeared and the time corresponding to the onset was taken as the OIT value. Figure 4.3 provides a typical OIT assessment from a recorded time-based thermal curve.

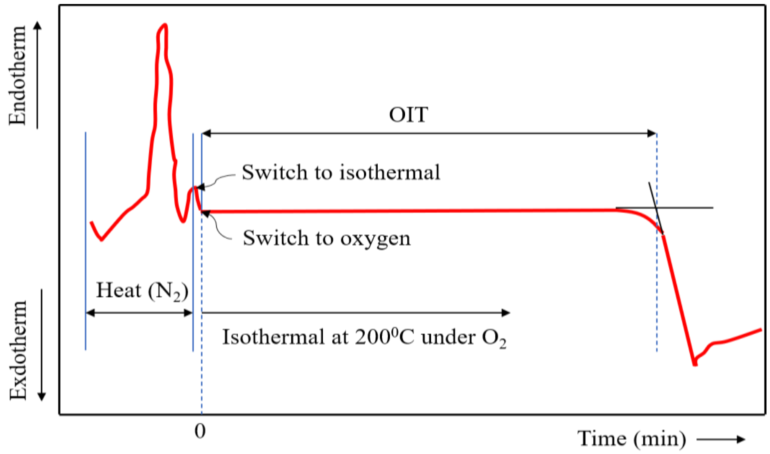


Figure 4.3 Evaluation of OIT from recorded time-based thermal curve.

4.2.7 Mechanical testing

The tensile properties of HDPE pipes were evaluated according to ASTM D638 (2014) [20] with dog-bone specimens. Tensile specimens were punched directly from the pipe liner with a stainless-steel die. The specific geometry of the specimens is described in detail in ASTM D638 (2014) and illustrated in Fig. 4.4.

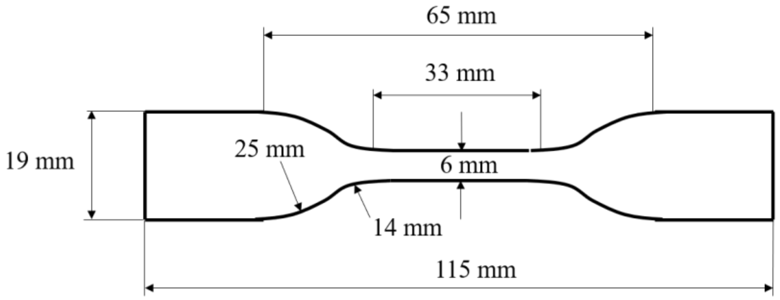


Figure 4.4 Typical dog-bone specimens for evaluating tensile strength.

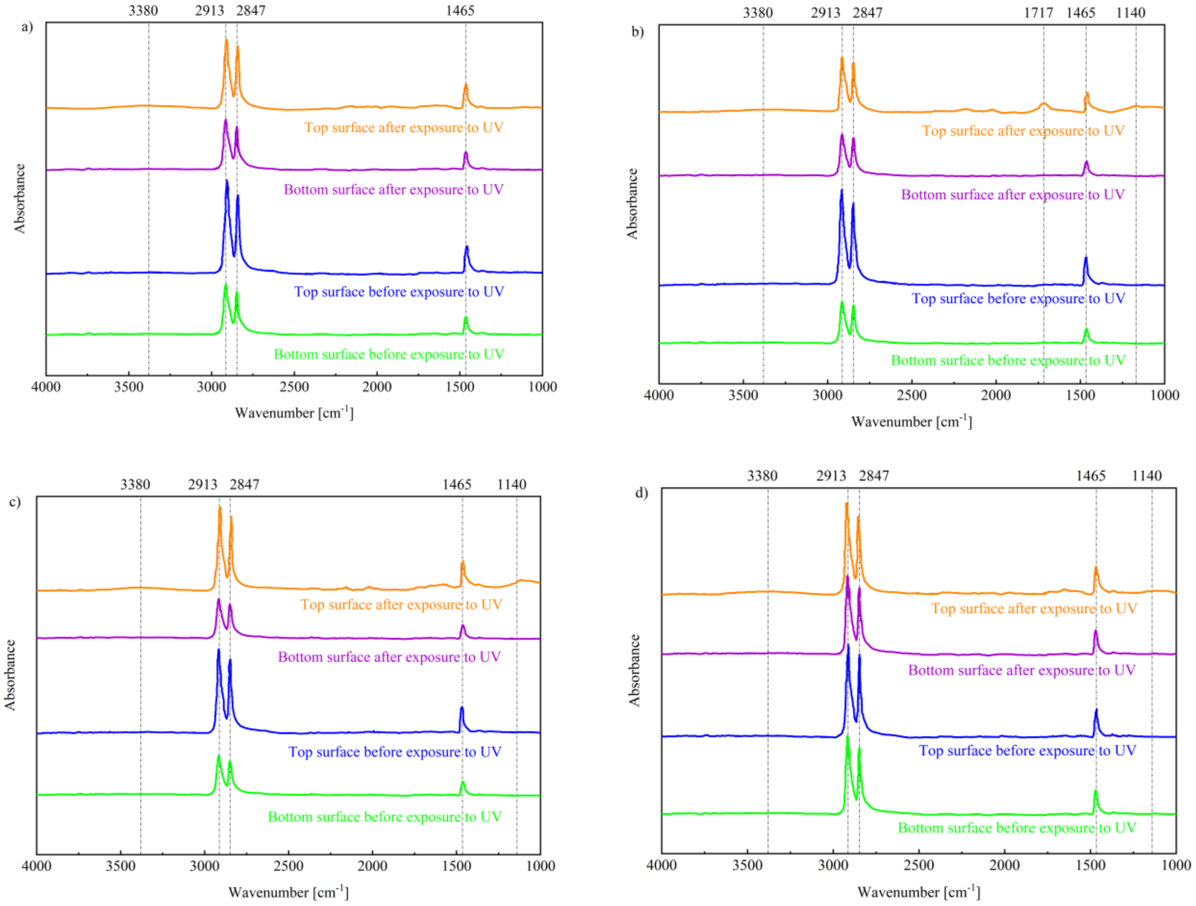
This test was conducted at 23^0C with an MTS universal testing machine equipped with a 5 kN load cell. A constant crosshead speed was maintained at 50 mm/min. A 634.12F-24 extensometer was used to measure sample extension. The stress–strain curve of the unexposed and UV-exposed HDPE specimens was reported after five replicates. The average tensile, elastic modulus values, and standard deviation were determined for each pipe.

The hardness of specimens before and after exposure to UV radiation was measured by a Shore hardness durometer using the D (HD) scale according to ASTM D2240 (2015) [26]. This digital durometer has an impact rod with a 30^0 conical point. The force was applied manually for 15 seconds, and the hardness value recorded. A rectangular specimen (15 mm x 15 mm) with a thickness not less than 6 mm was used. Three specimens for each pipe and three readings at three different locations were recorded for each specimen. The average value of each specimen is presented herein.

4.3 Results

4.3.1 FTIR analysis

FTIR analysis was performed to assess the degree of degradation. Figures 4.5 a, b, c, d, e, and f show the FTIR spectra of unexposed and UV-exposed HDPE specimens after 3600 hours of aging. For all specimens before and after exposure to UV radiation, the spectra show typical bands of C–H groups at 2913 cm^{-1} and 2847 cm^{-1}, attributed to asymmetric and symmetric stretching vibrations, respectively. The presence of bending vibrations from the CH_2 is visible at 1465 cm^{-1} [155]. Based on the results, the top and bottom surfaces of specimens before UV exposure presented the same chemical structure. There were no differences between pipes made with recycled or virgin resin. This observation is quite similar to specimens after UV exposure. In contrast, peaks of very low intensity at 1717 cm^{-1} and 1140 cm^{-1}, corresponding to the presence of C–O or C=O groups, were detected upon oxidation at the top surface compared to specimens before UV exposure [156]. In addition, the presence of a very small peak corresponding to O–H groups was observed at 3380 cm^{-1} after UV exposure. It can be concluded that UV exposure produced very small structural modifications on the top surface caused by oxidation. These light changes can in no way induce modifications that would affect material integrity.



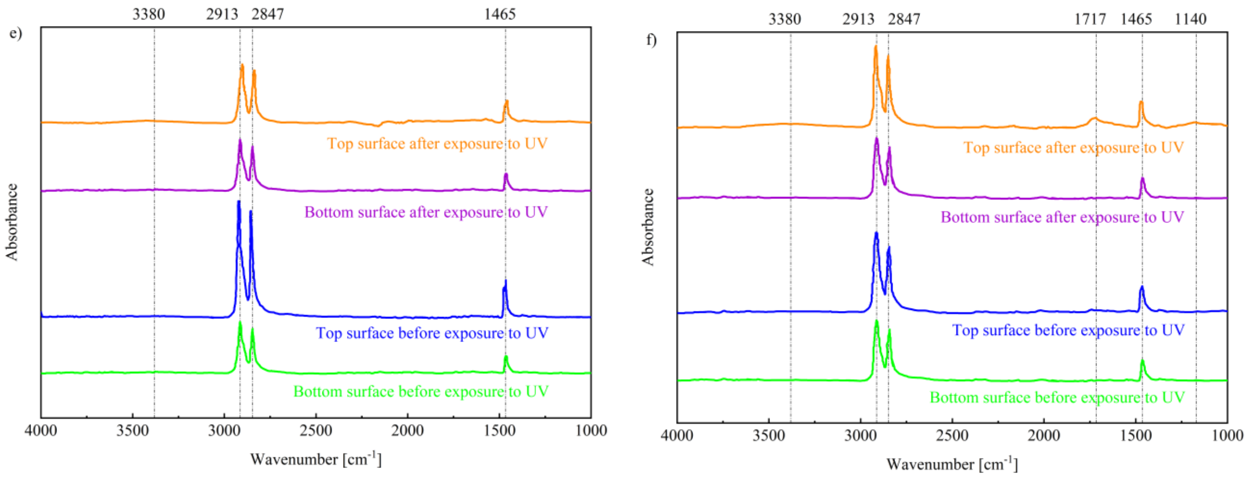


Figure 4.5 FTIR spectra of HDPE pipes before and after UV exposure: a) A-R, b) A-V, c) B-R, d) B-V, e) C-V, and f) D-R.

4.3.2 SEM/EDS analysis

SEM analysis of the surface of the HDPE pipes before and after exposure was carried out to observe any changes caused by UV radiation (Fig. 4.6). In general, as the SEM micrographs show, the specimen surfaces were relatively smooth at scan widths of 100 µm and 10 µm before UV exposure. After 3600 hours of irradiation, the specimen surface at the scan width of 100 µm evidenced no morphological changes. At the scan width of 10 µm, some cracks were observed on the surface of pipes A-V and D-R, possibly due to UV radiation. These microcracks only occurred on one light area (contaminant) of the sample surfaces and not on the darkest areas (resin). EDS (energy dispersive spectrometry) analysis revealed that these two areas had different chemical compositions (Fig. 4.7). The darkest areas were constituted of elements in the resin compound (mainly carbon); the lightest areas contained nitrogen, sulfur, and oxygen. Consequently, it may be assumed that the cracked areas did not result from the PE but rather from a contaminant probably deposited during the manufacturing process. The results are then quite consistent with the findings from FTIR analysis.

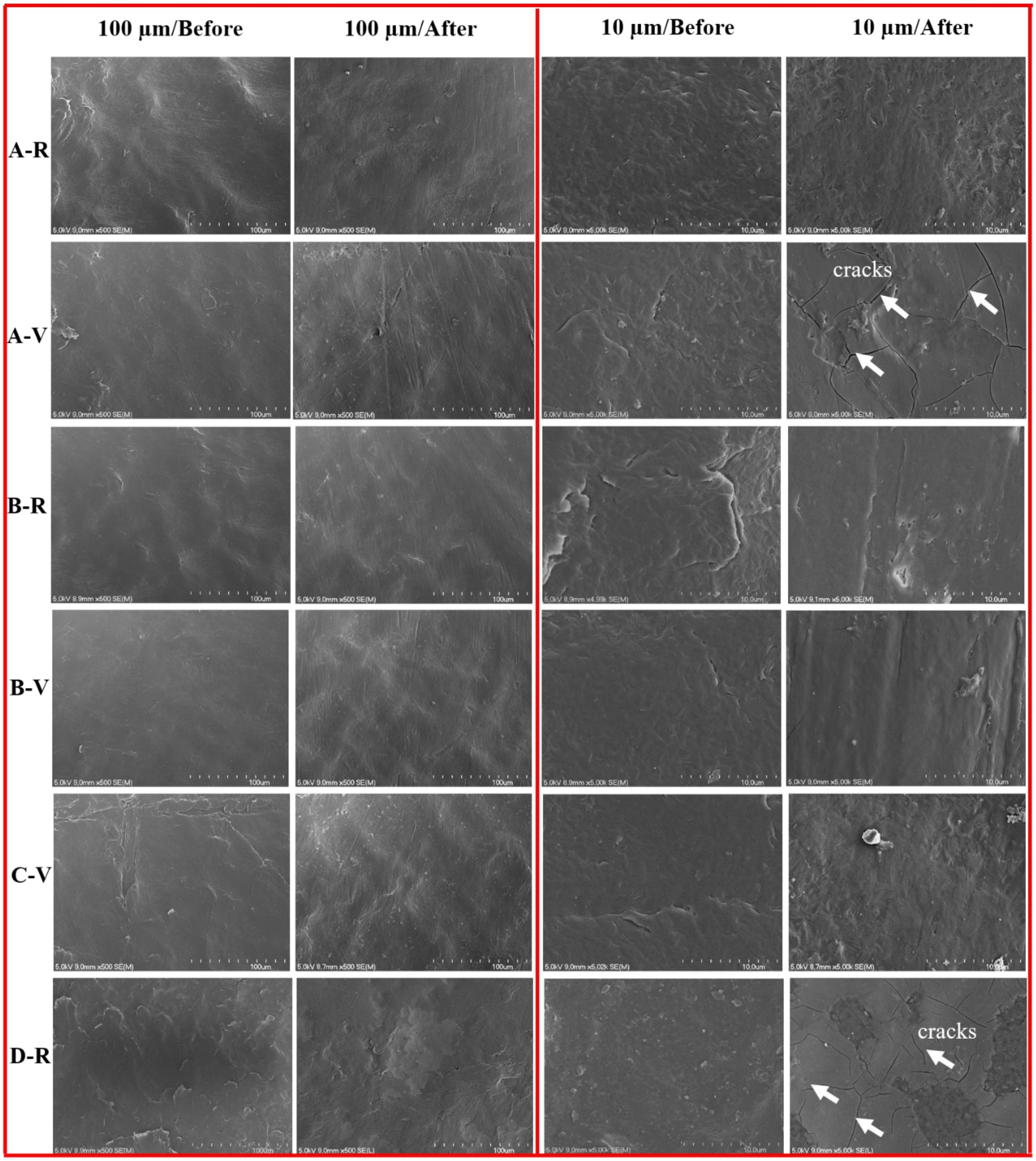


Figure 4.6 Morphological structure of HDPE pipes before and after UV exposure at scan widths of 100 µm and 10 µm.

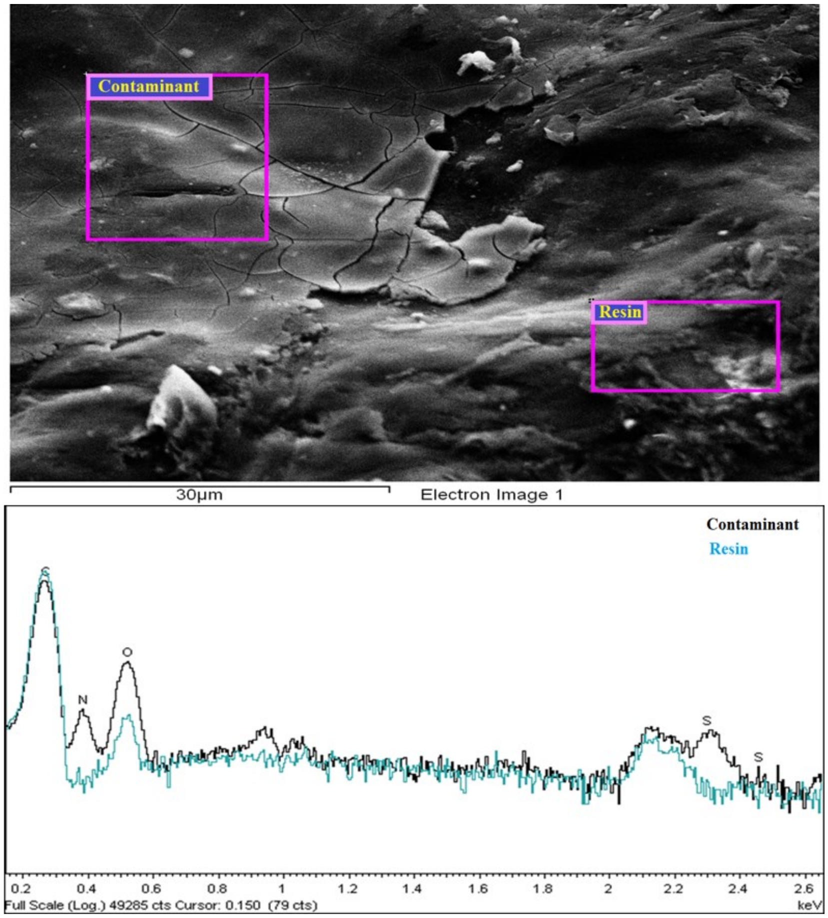


Figure 4.7 EDS analysis and chemical composition of lightest (contaminant) and darkest areas of A-V and D-R pipes.

4.3.3 Thermal behavior

Specimens before and after UV exposure were studied in order to investigate thermal properties and primarily the degree of crystallinity. The melting point and melting enthalpy of the HDPE pipes were measured with DSC. The degree of crystallinity was calculated from the ratio of the melting enthalpy of the specimens to the melting enthalpy of 100% crystalline polyethylene (287 J/g), as shown in Table 4.2. The results show that the melting point decreased (3^0C to 9^0C) after 3600 hours of UV exposure. As a result, a slight decrease of 0% to 4% of the melting enthalpy value resulted in a slight decrease of 0% to 4% crystallinity. It should be noted that photo-oxidative degradation usually takes place in the amorphous phase and in the amorphous-lamellar interface of the semicrystalline polymer [147,157]. In fact, the incorporation of antioxidants into pipes prevents the formation of free radicals during the manufacturing process. Hence, they prevent the degradation of pipes exposed to UV radiation.

Table 4.2 Melting temperature and crystallinity of the HDPE pipes before and after UV exposure

Specimen	Before UV Exposure			After UV Exposure		
	Melting point (°C)	Melting enthalpy (J/g)	Degree of crystallinity (%)	Melting point (°C)	Melting enthalpy (J/g)	Degree of crystallinity (%)
A-R	153	135	47	145	132	46
A-V	153	139	49	144	137	48
B-R	143	102	36	138	105	36
B-V	144	132	46	138	131	45
C-V	151	151	53	146	147	51
D-R	148	149	52	145	143	50

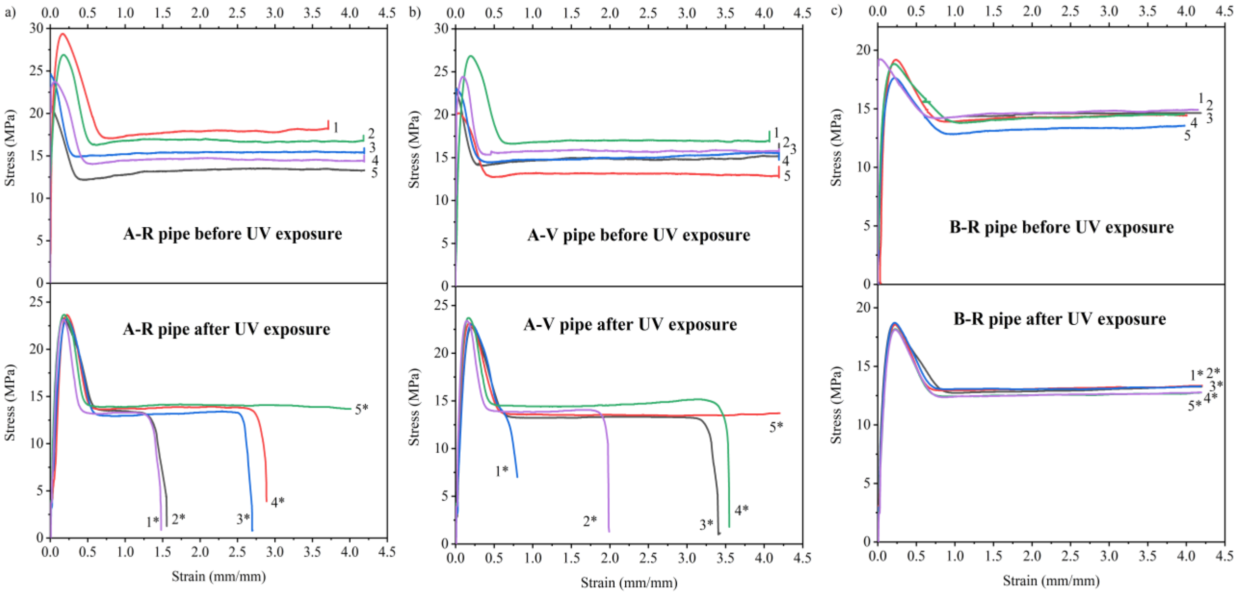
OIT is commonly used to evaluate the thermal-oxidative resistance of polymers and the depletion rate of antioxidants in their products [158-160]. Our study took OIT measurements to investigate the depletion rate of antioxidants of unexposed and UV-exposed HDPE specimens. Table 4.3 shows a decrease in OIT values after 3600 hours of UV exposure. The OIT decrease indicates the consumption of antioxidants in pipes. The reduction also depends on the thickness of the HDPE specimens. The difference between the OIT values of the exposed direct (top) and indirect (bottom) surface depends on specimen thickness, as shown in Table 3. This difference is clear with the specimens 7.30 and 7.80 mm in thickness. In contrast, there were no differences in OIT between the two sides in the thinner 3.25and 4.50 mm samples. It should be pointed out that the initial OIT values between pipes were different, assessed based on the initial antioxidant content and type in each pipe. This information is a professional secret and was unavailable to the authors. In addition, it can be concluded that all the investigated pipes met the OIT minimum of 20 minutes in ASTM D3895 (2019) [28], in which the antioxidant content was sufficient to withstand oxidation by UV radiation. The results of OIT measurements are in good agreement with the FTIR, SEM, and DSC analyses, in which antioxidants are consumed and prevent pipe degradation during UV exposure.

Table 4.3 OIT values of HDPE pipes before and after exposure to UV

Specimen	Thickness (mm)	OIT (min) - Before UV Exposure	OIT (min) - After UV Exposure	
			Top surface	Bottom surface
A-R	7.30	90	52	71
A-V	7.30	87	51	74
B-R	7.80	103	62	79
B-V	7.80	106	58	70
C-V	3.25	33	23	24
D-R	4.50	86	67	68

4.3.4 Mechanical behavior

The tensile test of specimens was carried out to determine the tensile strength and elastic modulus of the HDPE pipes. Figures 4.8 a, b, c, d, e, and f present the typical stress–strain curves for unexposed and UV-exposed HDPE specimens with five replicates for each pipe. The figures show that the stress–strain behavior of the pipes was governed by three regions: (1) elastic (linear and nonlinear), (2) neck propagation, and (3) plastic regions. The initial portion of the stress–strain curve is defined as the elastic deformation region where the elastic modulus is defined as the slope of the stress–strain curve. The maximum point on the stress–strain curve is called the yield point. The area corresponding to the reduced stress value is often called a neck. The area with relatively stable stress values is the development phase of the neck. The initial tensile strength of pipes A-R, A-V, B-R, B-V, C-V, and D-R pipes was 25.12, 23.58, 18.83, 17.44, 19.81, and 22.56 MPa, respectively. After 3600 hours of UV radiation, the tensile strength of the popes remained almost unchanged at 23.39, 23.19, 18.45, 17.40, 19.79, and 22.36 MPa for pipes A-R, A-V, B-R, B-V, C-V, and D-R pipes, respectively (Fig. 4.9a). Figures 4.8 a, b, c, d, e, and f show that the elongation of all specimens was more than 4.0 mm/mm before UV exposure. After exposure to UV radiation for 3600 hours, the elongation at break of specimens B-R and B-V remained at 4.0 mm/mm, but this value varied for the other specimens. The elongation at break of specimens A-R, A-V, C-V, D-R was 1.5 to 2.9 mm/mm, 0.8 to 3.5 mm/mm, 0.5 to 1.65 mm/mm, and 0.9 to 3.2 mm/mm, respectively. The UV radiation did not significantly affect the tensile strength of the pipes. Figures 4.8 a, b, c, d, e, and f show a variation in the elongation values at break. The behavior also varied from one manufacturer to the next and from one specimen to next from the same manufacturer. The pipes from manufacturers A, C, and D did not have uniform behavior in that they more fragile than those from manufacturer B. Figure 4.9b provides the elastic modulus of the specimens, showing that the elastic modulus of all the specimens decreased after UV exposure for 3600 hours. The standard deviation of the calculations was, however, relatively large for specimens A-R, C-V, and D-R. In fact, the addition of antioxidants and carbon black further inhibited the effect of UV radiation.



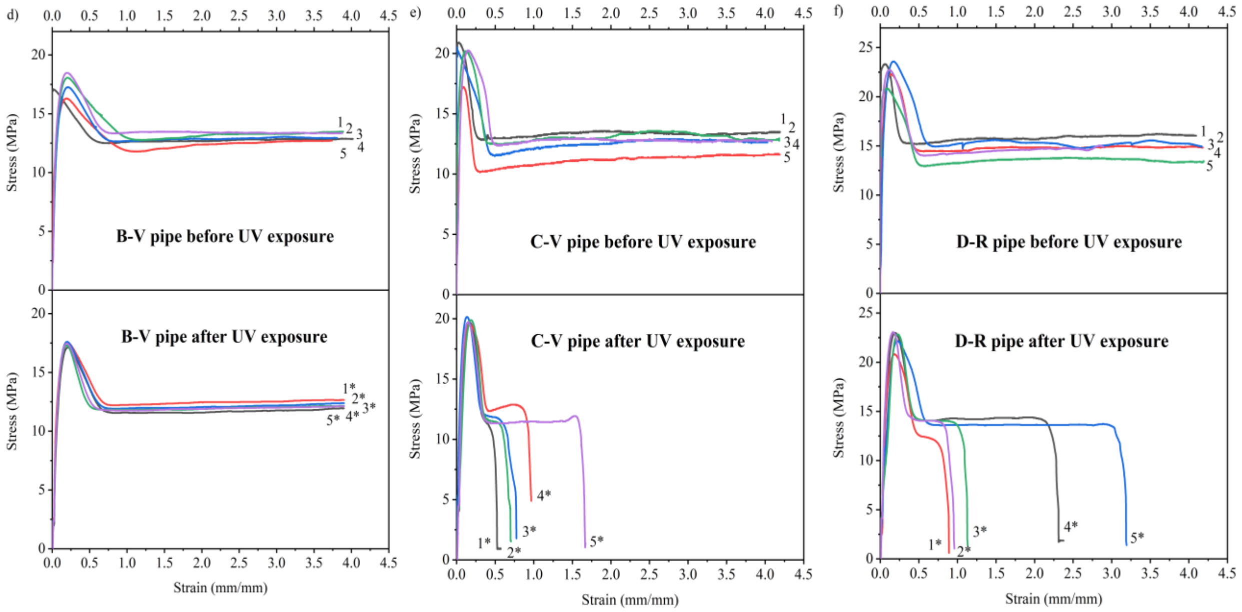


Figure 4.8 Typical tensile stress–strain behaviors of the HDPE pipes before and after UV exposure: a) A-R, b) A-V, c) B-R, d) B-V, e) C-V, and f) D-R.

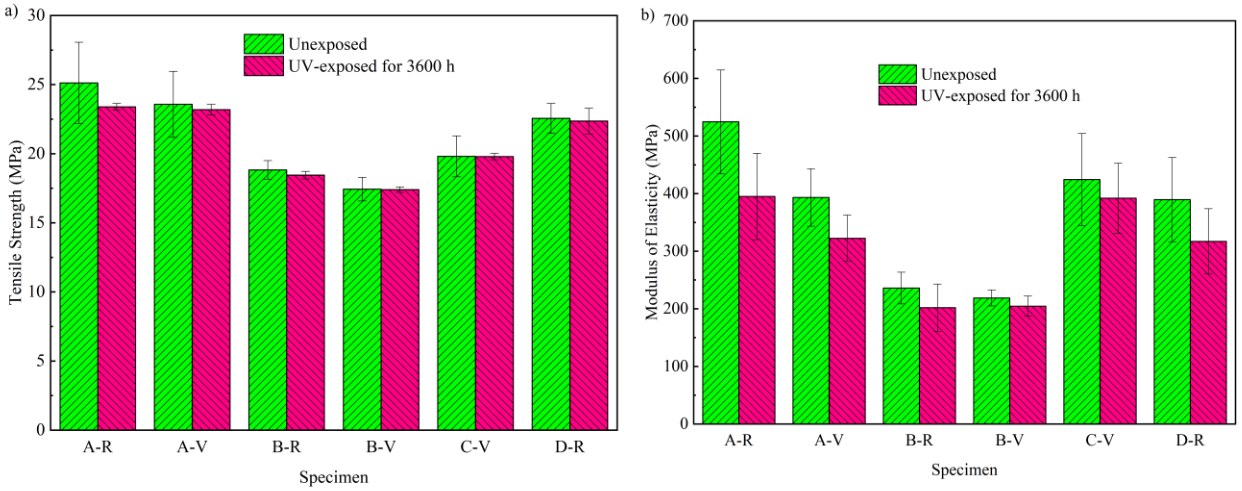


Figure 4.9 Mechanical properties of the HDPE pipes before and after UV exposure: a) tensile strength and b) modulus of elasticity.

Table 4.4 gives the results of the hardness analysis; hardness was measured with a Shore D durometer. Table 4.4 shows that a slight decrease in hardness was observed for all specimens. For semicrystalline polymers such as HDPE, the factors affecting hardness act mainly on the crystalline region. In fact, polymer chains in the crystalline region are denser than those in the amorphous region [139]. Consequently, the slight differences in hardness values between the HDPE pipes before and after UV exposure were caused by variations in the contents of the crystalline phase (Table 4.2).

Table 4.4 Hardness of the HDPE pipes before and after UV exposure

Specimen	Hardness (HD)	
	Before UV Exposure	After UV Exposure
A-R	56	51
A-V	55	49
B-R	55	53
B-V	60	56
C-V	65	61
D-R	61	57

4.3.5 Statistical Analysis

Table 4.4 and Fig. 4.9 show that there was variation in tensile strength, modulus of elasticity and hardness of pipes before and after UV exposure. Therefore, a statistical analysis was performed to clarify if there were any significant differences between the measurements. In the present study, one-way analysis of variance (ANOVA) was used. In general, there is no significant difference between measurements if they meet the following conditions: (1) the F-value is less than the F-critical, or (2) the P-value is greater than the selected alpha level (0.05). As shown in Tables 4.5, 4.6 and 4.7, the F value is less than the critical F value and the P value is greater than 0.05 for tensile strength, modulus of elasticity and hardness before and after exposure to UV. It is important to emphasize that there was no statistically significant difference in these measurements before and after UV exposure. Therefore, the tensile strength, modulus of elasticity and hardness of pipes can be considered relatively unchanged after UV exposure.

Table 4.5 One-way ANOVA of tensile strength before and after UV exposure

Specimen	Description	Sum of squares	df	Mean square	F-value	P-value	F-critical
A-R	Between groups	7.5	1	7.5	1.726	0.225	5.318
	Within groups	34.9	8	4.4	–	–	–
	Total	42.4	9	–	–	–	–
A-V	Between groups	0.4	1	0.4	0.130	0.728	5.318
	Within groups	23.4	8	2.9	–	–	–
	Total	23.8	9	–	–	–	–
B-R	Between groups	0.4	1	0.4	1.329	0.282	5.318
	Within groups	2.1	8	0.3	–	–	–
	Total	2.5	9	–	–	–	–
B-V	Between groups	0.004	1	0.004	0.011	0.921	5.318
	Within groups	3.047	8	0.381	–	–	–
	Total	3.051	9	–	–	–	–
C-V	Between groups	0.001	1	0.001	0.001	0.979	5.318
	Within groups	8.939	8	1.117	–	–	–
	Total	8.940	9	–	–	–	–
D-R	Between groups	0.1	1	0.1	0.092	0.769	5.318
	Within groups	8.1	8	1.0	–	–	–
	Total	8.2	9	–	–	–	–

Table 4.6 One-way ANOVA of elastic modulus before and after UV exposure

Specimen	Description	Sum of squares	Df	Mean square	F-value	P-value	F-critical
A-R	Between groups	34052.7	1	34052.7	4.959	0.068	5.987
	Within groups	41204.7	6	6867.5	–	–	–
	Total	75257.4	7	–	–	–	–
A-V	Between groups	11166.1	1	11166.1	5.585	0.051	5.591
	Within groups	13995.3	7	1999.3	–	–	–
	Total	25161.4	8	–	–	–	–
B-R	Between groups	2189.6	1	2189.6	1.589	0.254	5.987
	Within groups	8269.9	6	1378.3	–	–	–
	Total	10459.5	7	–	–	–	–
B-V	Between groups	382.5	1	382.5	1.399	0.282	5.987
	Within groups	1639.7	6	273.3	–	–	–
	Total	2022.2	7	–	–	–	–
C-V	Between groups	1871.9	1	1871.9	0.403	0.549	5.987
	Within groups	27856.6	6	4642.8	–	–	–
	Total	29728.5	7	–	–	–	–
D-R	Between groups	7505.4	1	7505.4	2.061	0.211	6.608
	Within groups	18209.5	5	3641.9	–	–	–
	Total	25714.9	6	–	–	–	–

Table 4.7 One-way ANOVA of hardness before and after UV exposure

Specimen	Description	Sum of squares	Df	Mean square	F-value	P-value	F-critical
A-R	Between groups	40.8	1	40.8	2.065	0.201	5.987
	Within groups	118.7	6	19.8	–	–	–
	Total	159.5	7	–	–	–	–
A-V	Between groups	70.5	1	70.5	4.342	0.082	5.987
	Within groups	97.5	6	16.2	–	–	–
	Total	168.0	7	–	–	–	–
B-R	Between groups	7.5	1	7.5	1.875	0.220	5.987
	Within groups	24	6	4.0	–	–	–
	Total	31.5	7	–	–	–	–
B-V	Between groups	22.5	1	22.5	4.588	0.076	5.987
	Within groups	29.5	6	4.9	–	–	–
	Total	52.0	7	–	–	–	–
C-V	Between groups	28.0	1	28.0	5.708	0.054	5.987
	Within groups	29.5	6	4.9	–	–	–
	Total	57.5	7	–	–	–	–
D-R	Between groups	21.1	1	21.1	3.992	0.093	5.987
	Within groups	31.8	6	5.3	–	–	–
	Total	52.9	7	–	–	–	–

4.4 Discussion

The use of recycled HDPE pipes is of current interest given the sources of recycling material. Compared to virgin pipes, recycled pipes are more sustainable and cost-effective. Using recycled plastics can reduce a product's carbon footprint. Indeed, a product with 100% recycled content would have a carbon footprint 24% smaller [60]. Recycling products can reduce the use of raw materials, energy requirements, water consumption, and greenhouse-gas emissions in the production process [60,62]. Our study investigated the effect of UV radiation on the properties of recycled and virgin HDPE pipes. Table 4.8 shows that UV radiation produced only small changes in the pipe surface, and it did not significantly affect the pipes made with virgin or recycled material.

- FTIR analysis shows that negligible structural modifications occurred on the top surface after UV exposure. In the case of manufacturer, A, the peak of carbonyl stretching vibration at 1717 cm^{-1} was observed with virgin pipes (A-V). In contrast, the peak of carbonyl stretching vibration did not appear for either recycled or virgin pipes from manufacturer B. This peak was detected for recycled pipes from manufacturer D. In addition, the peak of polyhydroxyl stretch at 3380 cm^{-1} was detected in all the pipes. The SEM results were similar for specimens after UV exposure. When the scanning width was 10 µm, some cracks appeared on the surface of virgin pipes (A-V) from manufacturer A and recycled pipes (D-R) from manufacturer D. This was not observed for either recycled or virgin pipes from manufacturer B.

- DSC analysis shows that the melting points for the recycled and virgin pipes from the same manufacturer (only A and B) were the same before and after UV exposure. The degree of crystallinity remained constant for both recycled and virgin pipes after 3600 hours of UV exposure. In general, the presence of impurities during the manufacturing process caused the recycled pipes to easily absorb UV radiation compared to the virgin pipes. However, based on OIT measurements, it appears that antioxidants were consumed as a result of UV exposure. In other words, they prevented the formation of free radicals. Consequently, the thermal properties of recycled and virgin pipes remained constant under the effect of UV radiation.
- The tensile strength, elastic modulus, and hardness of the HDPE pipes made with recycled or virgin material were relatively unaffected after UV exposure. In contrast, the elongation at break decreased from 12% to 87% of the initial elongation at break for both recycled and virgin pipes.

Based on the results, the pipes are made from recycled or virgin resins were relatively unaffected by UV radiation. These results are in good agreement with the finding from other studies [149-152,161]. It should be emphasized that the presence of carbon black, antioxidants, and UV stabilizers had a synergistic effect on the UV stabilization of the recycled and virgin HDPE pipes.

Table 4.8 The effect of UV radiation on the pipes made from recycled or virgin HDPE

Type of HDPE	Specimen	Before and After UV Exposure						
		FTIR	SEM	Degree of crystallinity	Tensile strength	Elongation at break	Elastic modulus	Hardness
Recycled	A-R	√	o	o	o	√	o	o
	B-R	√	o	o	o	o	o	o
	D-R	√	√	o	o	√	o	o
Virgin	A-V	√	√	o	o	√	o	o
	B-V	√	o	o	o	o	o	o
	C-V	√	o	o	o	√	o	o

Note: o = no changes or remained constant; √ = negligible changes.

4.5. Conclusions

The effects of UV radiation on the chemical, microstructure, thermal, and tensile properties of new corrugated HDPE pipes made with or without recycled resins used in road drainage systems were assessed. The following conclusions can be drawn from the study:

1. The incorporation of carbon black, antioxidants, UV stabilizers helped limit the formation of free radicals during the aging process. Therefore, it prevented the degradation of the HDPE pipes during UV exposure.
2. Before being exposed to UV radiation, the recycled and virgin pipes had the same chemical and morphological structures. Small changes, probably due to the presence of a contaminant on the surface of certain pipes, were observed in FTIR and SEM analysis after UV exposure.
3. While the antioxidants in the pipes were partly consumed due to UV exposure, the minimum 20-minute OIT requirement for withstanding oxidation was met. In addition, statistical analysis using one-way analysis of variance (ANOVA) shows that there was no significant difference between measures of tensile strength, modulus of elasticity and hardness before and after exposure to UV. Therefore, it can be considered that the pipes made from virgin or recycled resins maintained adequate thermal (melting point, degree of crystallinity) and mechanical (tensile strength, elastic modulus, hardness) properties after exposure to UV radiation. The elongation at break was, however, reduced. Achieving this level of UV protection required a minimum of 2% to 4% of carbon black added to the resin compound, depending on the presence or absence of other antioxidants or UV stabilizers.
4. The findings of the current study would be the premise upon which recycled HDPE becomes one of the most widely used materials in the future. On the other hand, recycled HDPE should be used for floating solar applications where the material is subjected to direct sunlight.

Data Availability Statement

Some or all data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This research received financial support from the Natural Science and Engineering Research Council of Canada (NSERC), the Ministry of Transportation of Quebec (MTQ), and the University of Sherbrooke Research Centre on Composite Materials (CRUSMaC). The authors are also grateful to the technical staff of the structural laboratory at the University of Sherbrooke, especially Jérôme Lacroix and Steven MacEachern, for their technical assistance.

5 CHAPITRE 5: COMPORTEMENT DE FLUAGE À LONG TERME DES TUYAUX EN PEHD

5.1 Introduction

Afin de déterminer la sensibilité du PEHD à la rupture par fluage, deux méthodes d'essais ont été initialement proposées, à savoir la méthode de fluage conventionnelle et la méthode isotherme par étapes (SIM : *Stepped Isothermal Method*). Compte tenu du nombre d'essais à réaliser et du temps nécessaire, la méthode conventionnelle n'a pas été retenue puisqu'elle prendrait plusieurs années. Par conséquent, la méthode adoptée pour déterminer le fluage est la méthode SIM qui utilise des paliers de température et des temps d'arrêt (temps entre les paliers de température) pour accélérer la rupture par fluage. Un seul échantillon est utilisé pour tous les paliers de température, ce qui évite toute variabilité et donne un résultat plus précis. Pour obtenir la courbe maîtresse, la méthode emploie des facteurs de translation à une température de service définie et nécessite un décalage vertical puisque le comportement de l'échantillon ne peut pas être attribué uniquement au fluage, mais également aux contraintes thermiques dues au chauffage.

En service, les tuyaux sont soumis au fluage lorsqu'ils sont soumis à une charge constante pendant une longue période. En raison de sa ductilité, le PEHD subit des déformations excessives pendant le fluage. En cas de présence de défauts tels qu'une fissure, le fluage peut faire croître cette fissure et entraîner la rupture. La rupture par fluage est critique puisqu'elle se produit à des contraintes relativement faibles. Dans cet essai, les paliers de température et les temps d'arrêt ne doivent pas être inférieurs à 10 000 secondes pour accélérer la rupture et un pourcentage de la limite d'élasticité en traction est appliqué. Les données obtenues sont utilisées pour déterminer le comportement de fluage à long terme des matériaux en utilisant les facteurs de décalage. La figure 5.1 montre l'appareil MTS de l'Université de Sherbrooke utilisé pour les essais SIM. La machine est composée d'une chambre faisant varier la température et d'un système de chargement en traction classique. Il est à noter que ces essais sont assez rapides et fournissent des résultats en moins de 24 heures. La figure 5.2 montre les spécimens et la figure 5.3 la configuration d'essai et une vue rapprochée des spécimens testés. Le mode de rupture des éprouvettes est illustré à la figure 5.4, qui est le mode de rupture typique des tuyaux en PEHD en phase I.

Figure 5.1 Machine MTS et contrôleur pour les essais SIM

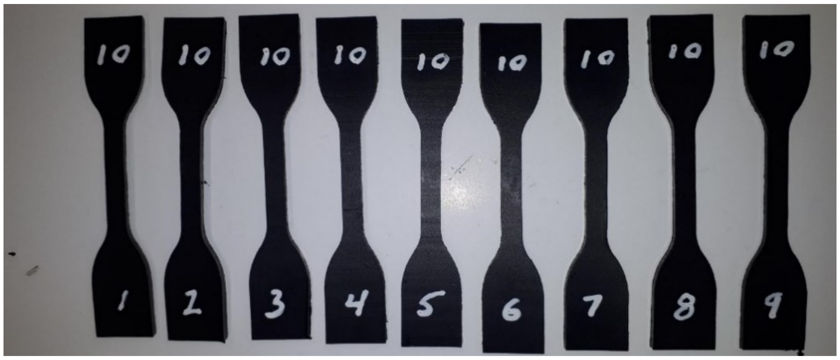


Figure 5.2 Échantillons A-V pour les essais SIM

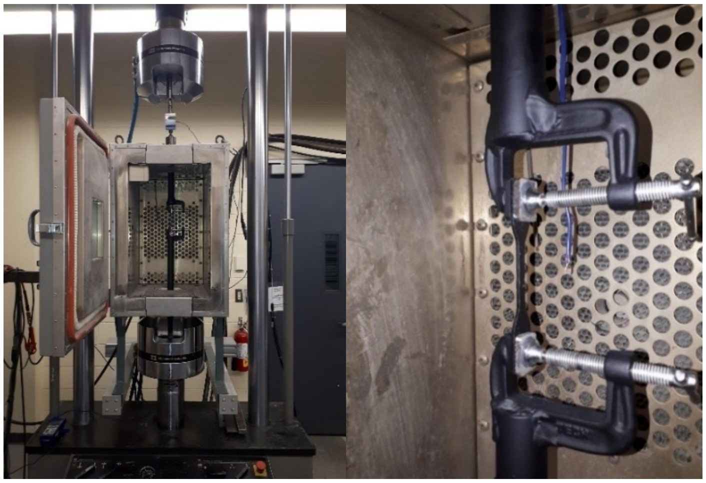


Figure 5.3 Configuration des essais SIM et A-V spécimen en cours d’essai.

Figure 5.4 Mode de rupture typique des spécimens en PEHD après l'essai SIM

5.2 Matériaux et méthodes

Au total, six tuyaux de 900 mm de diamètre, dont trois fabriqués selon la classe A, BNQ 3624-120 [19] avec de la matière vierge (A-V, B-V, C-V), deux avec différentes proportions de matière recyclée (A-R, D-R) et un avec de la matière réutilisée (B-R) ont été testés. Il convient de noter que les proportions exactes de matière recyclée n'ont pas été divulguées par les fabricants. La matière réutilisée est générée uniquement en interne plutôt que de provenir d'une source externe. Pour l'essai SIM, tous les échantillons sont découpés directement sur la paroi intérieure des tuyaux à l'aide d'un dispositif approprié en acier inoxydable selon les dimensions standards des normes ASTM D638-14 [20] pour le fluage.

Pour les essais SIM, les échantillons ont une forme d'haltère (ASTM D638, type IV) comme ceux de l'essai de traction. L'essai débute par un fluage de 1740 secondes à 23^0 ou 25^0C. Ensuite, la température est augmentée rapidement par palier de 7^0C jusqu'à atteindre 60°C pour les essais de fluage subséquents. Une fois les données brutes collectées, l'analyse des données de déformation est réalisée avec pour objectif de prendre chacun des segments de température supra-ambiante et de les déplacer à la température ambiante (référence à 23^0 et 10^0C). Il existe trois parties types de déplacement pour analyser les données de SIM et construire les courbes maîtresses de fluage : (1) le déplacement vertical, (2) le rééchelonnement et (3) le déplacement horizontal.

- (1) le déplacement vertical donne une courbe continue en déformation par soustraction des déformations élastiques à chaque température comme illustré à la figure 5.5a.

- (2) la courbe de fluage d'une seule étape est extrapolée à déformation de fluage nulle, puis décalée sur l'axe des temps par la différence entre l'heure de début réelle de cette étape et l'heure de début extrapolée comme le montre les figures 5.5b et c. On notera que la première étape de l'analyse consiste à déterminer ce que l'on appelle le temps de démarrage virtuel (t') pour chaque étape suivante l'étape précédente (figure 5.5b). Ceci explique les effets du fluage produit à basse température. Cette étape est nécessaire, car l'éprouvette a «mémorisé» l'étape de fluage précédente. Cela permet également de redimensionner les courbes de fluage individuelles et de les tracer sur une échelle de temps commune.

- (3) les courbes de fluage individuelles mises à l'échelle sont décalées horizontalement pour former une seule courbe maîtresse à l'aide de l'équation WLF (équation 1) comme le montre la figure 5.5d. Les paramètres universels C_1 = 18K et C_2 = 52K sont utilisés. Il est à noter qu'il existe une brève période (de 25^0 à 32^0C) pendant laquelle les données ne correspondent pas au modèle de courbe maîtresse. Cette déviation est causée par un changement de température trop lent du fait de la proximité de cette plage de température avec la température ambiante. Il ne s'agit donc pas d'une déviation par rapport au modèle, mais simplement d'un problème de contrôle de température.

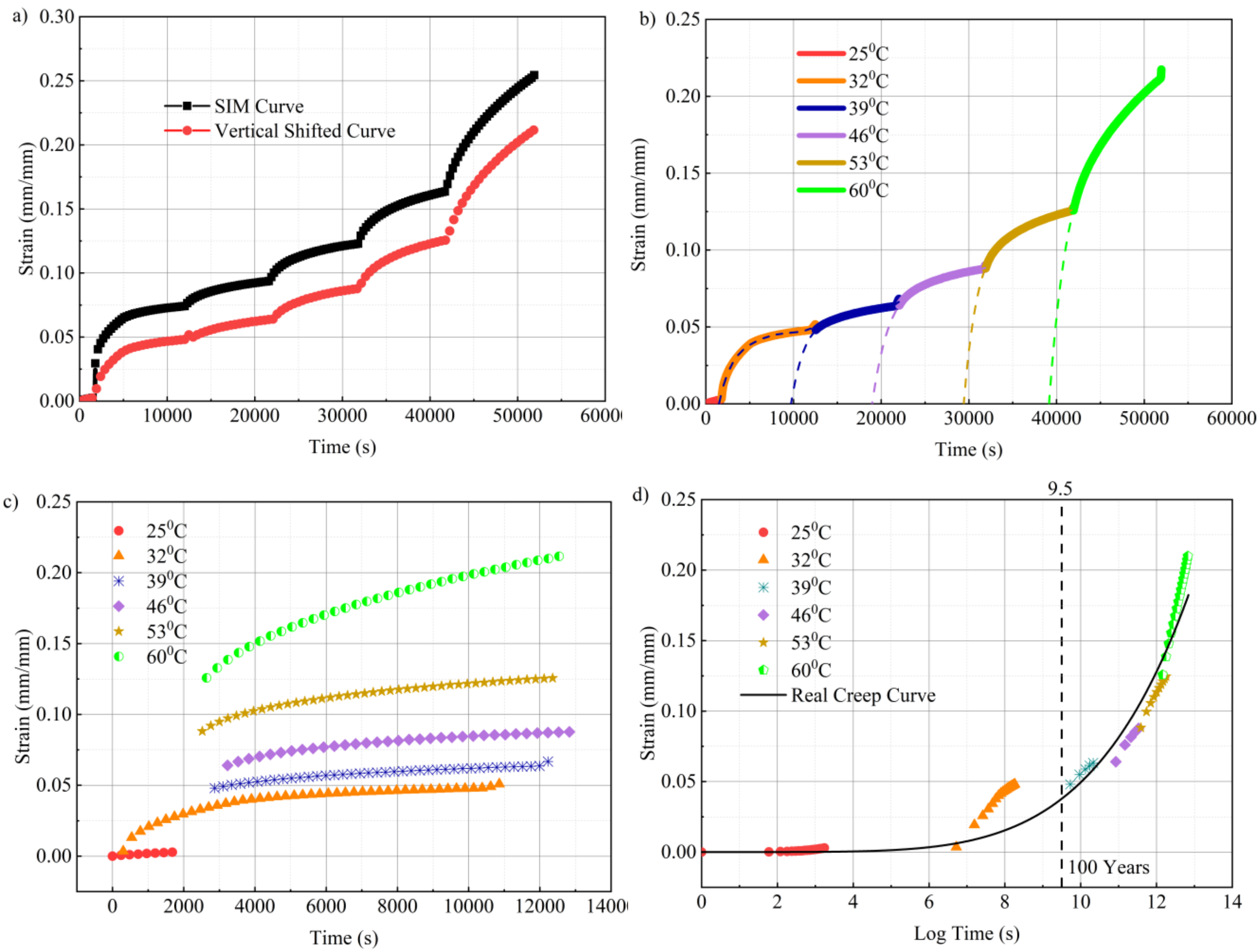


Figure 5.5 Étapes de construction des courbes maîtresses de fluage a) déplacement vertical b) temps de démarrage virtuel c) rééchelonnement d) déplacement horizontal.

5.3 Résultats et conclusions

La figure 5.6 présente les courbes maîtresses de fluage (déformation-temps) des tuyaux à 23^0 et 10^{0}C. L'essai SIM peut également être effectué sous des charges plus élevées pour créer un environnement de rupture par fluage. Dans cette étude, les tuyaux ont été testés avec trois charges différentes pour créer la courbe maîtresse de fluage (contrainte – temps). Puisqu'il est difficile de localiser exactement l'endroit de la rupture, il existe une convention qui détermine une limite de contrainte. Dans ce cas, une limite de déformation de 35% (tuyaux A-R, A-V, B-R, B-V) et 15% (tuyaux C-V, D-R) a été choisie pour définir la durée de vie. Pour construire la courbe maîtresse de rupture par fluage, il suffit de tracer la contrainte logarithmique en fonction du temps logarithmique comme illustré à la figure 5.7. D'après les courbes obtenues, il peut être conclu que la durée de vie des tuyaux en PEHD excède 100 ans à 23^0 et 10^{0}C (figures 5.6a, b, c, d, e, et f). Les résultats montrent que la limite élastique à 100 ans est d'au moins 8 MPa (1160 lb/po^2) à 23^{0}C et 10 MPa (1450 lb/po^2) à 10^{0}C comme indiqué dans le tableau 5.1.

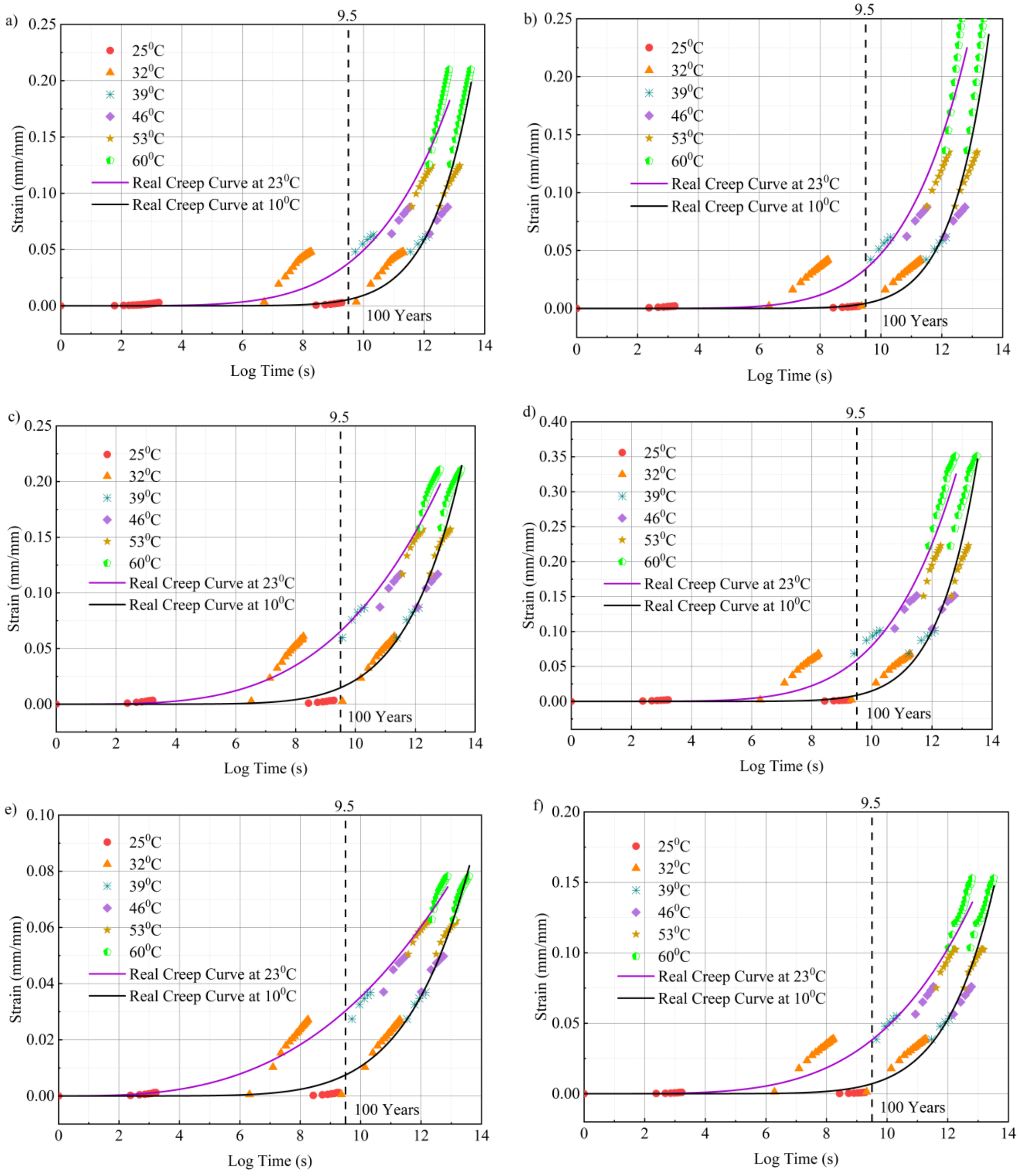


Figure 5.6 Courbes maîtresses de fluage (déformation-temps) à 23^0 et 10^0C a) A-R b) A-V c) B-R d) B-V e) C-V f) D-R.

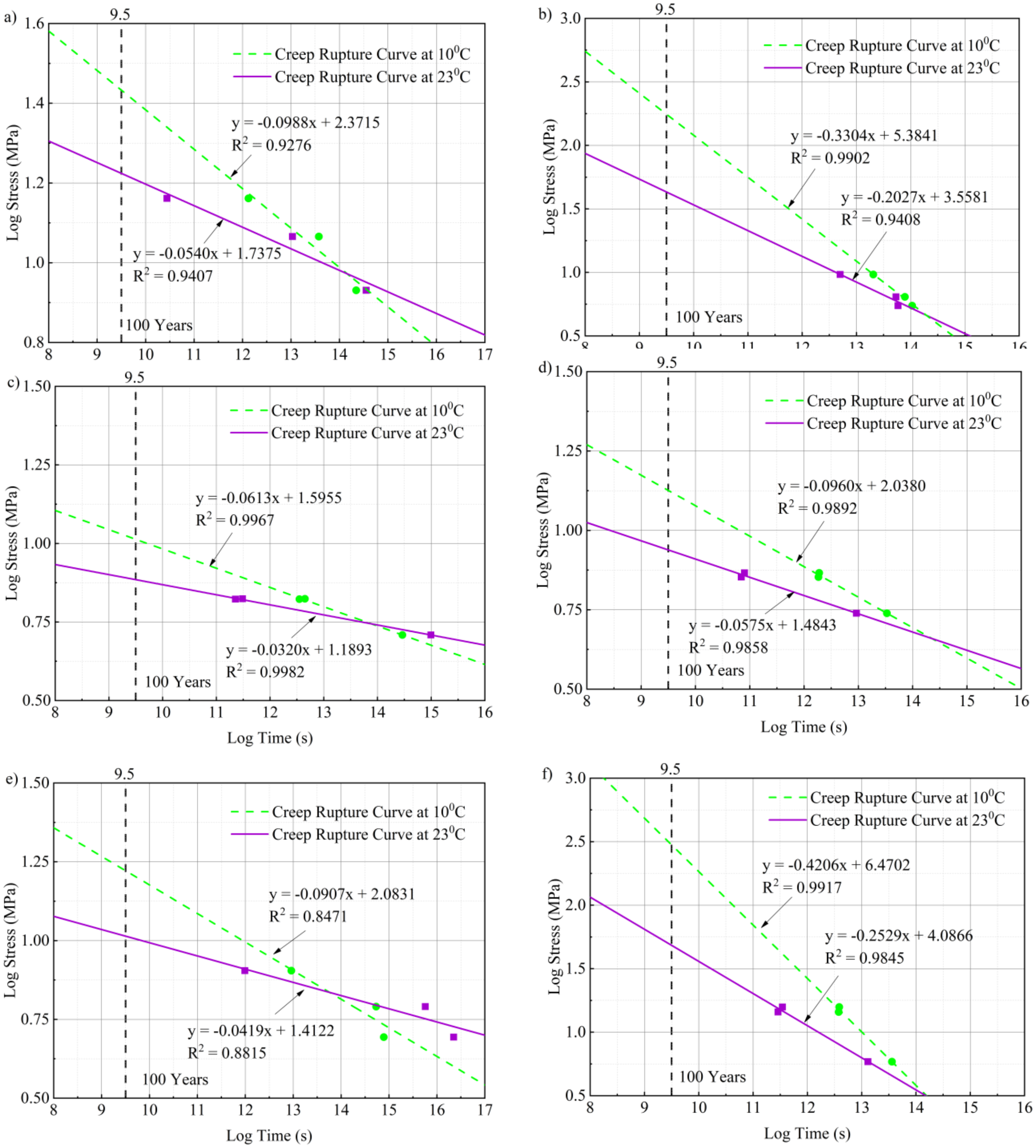


Figure 5.7 Courbes maîtresses de fluage (contrainte-temps) à 23^0 et 10^0C a) A-R b) A-V c) B-R d) B-V e) C-V f) D-R.

Tableau 5.1 Contraintes estimées des tuyaux en PEHD à 23^0C et 10^0C

Échantillon	Durée de vie (année)	Contrainte à 10^0C (MPa) (Québec)	Contrainte à 23^0C (MPa) (Floride)
A-R	> 100	27	17
A-V	> 100	176	43
B-R*	> 100	10	8
B-V	> 100	13	9
C-V	> 100	17	10
D-R	> 100	298	48

6 CHAPITRE 6: RÉSISTANCE AUX FISSURES SOUS CONTRAINTE ET PRÉDICTION DE LA DURÉE DE VIE DES TUYAUX ONDULÉS RECYCLÉS ET VIERGES EN PEHD UTILISÉS DANS LES SYSTÈMES DE DRAINAGE-ROUTIER : UNE ÉTUDE DE CAS AU QUÉBEC, CANADA

Avant-propos

Auteurs et affiliation:

Khanh Q. Nguyen: Étudiant au doctorat, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Khaled Mohamed: Stagiaire postdoctoral, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Patrice Cousin: Chercheur associé, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Mathieu Robert: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Adel El-Safty: Professeur, Département de génie civil, Université de North Florida, Jacksonville, Florida, USA.

Brahim Benmokrane: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Date de soumission: 16 décembre 2021

Titre du journal: ASCE Journal of Materials in Civil Engineering

Titre en anglais: Stress Crack Resistance and Life Prediction of Corrugated Recycled and Virgin HDPE Pipes Used in Road Drainage Systems: A Case Study in Quebec, Canada.

Résumé en français

La durée de vie de conception de 100 ans est une exigence essentielle pour les qualités de tuyaux modernes en polyéthylène à haute densité (PEHD) utilisées dans les systèmes de drainage-routier. La question de savoir si les tuyaux recyclés garantissent des performances et peuvent être substitués aux tuyaux vierges est également un enjeu en cours de clarification. De plus, la variété des caractéristiques de surface et les conditions de service spécifiques des tuyaux affectent considérablement la durée de vie des tuyaux. Ainsi, l'Université de Sherbrooke et le ministère des Transports du Québec (MTQ) ont récemment collaboré pour réaliser un projet sur la performance à long terme des tuyaux en PEHD recyclés et vierges fournis par les fabricants locaux. Ce projet comprend notre étude portant sur la résistance à la fissuration sous contrainte (SCR) et prédisant la durée de vie des nouveaux tuyaux en PEHD fabriqués par les fabricants canadiens ainsi que des tuyaux en PEHD en service sur le terrain au Québec sous vieillissement naturel. Des spécimens entaillés ont été découpés directement dans le liner de tuyau en PEHD pour évaluer leur SCR. De plus, quatre conditions d'exposition ont été utilisées: les cycles de gel-dégel et l'exposition à des solutions salines, acides et alcalines pour prédire la durée de vie restante des tuyaux. La SCR a été étudiée avec la méthode d'essai Florida dans de l'eau à 450 lb/po^2 et 80^{0}C, 650 lb/po^{2}psi et 80^{0}C, et 650 lb/po^2 et 70^{0}C conformément à FM 5-573. Les points de données à des températures élevées ont ensuite été décalés pour prédire la durée de vie des tuyaux dans des conditions de service inférieures avec la méthode de décalage de Popelar (PSM) et la méthode de processus de débit (RPM), qui sont toutes deux des méthodes d'extrapolation. Les résultats montrent qu'avant immersion dans différentes solutions, le recyclé pouvait garantir 100 ans de durée de vie en tant que tuyaux vierges sous des conditions de service de 10^{0}C et 500 lb/po^2. Après immersion, la diminution de SCR était liée aux deux étapes de température élevée dans le test conditionné et le test Florida, ainsi qu'à l'environnement hostile du test conditionné. De plus, RPM s'est avéré plus fiable que PSM. Les points de données étaient presque dispersés et non linéaires avec PSM.

Mots-clés: Tuyaux en polyéthylène haute densité (PEHD); Vieillissement par accélération ; Vieillissement naturel; Résistance à la fissuration sous contrainte ; Méthode de décalage de Popelar (PSM); Méthode de Rate Processing (RPM) ; Tuyaux en PEHD recyclé ; La durée de vie.

Abstract

The design service life of 100 years is an essential requirement for modern corrugated high-density polyethylene (HDPE) pipe grades used in road drainage systems. Whether the recycled pipes guarantee performance and can be substituted for virgin pipes is also an issue in the process of being clarified. Furthermore, the variety of area characteristics and the specific serving conditions of the pipes significantly affect pipe service life. Therefore, the University of Sherbrooke and the Quebec Ministry of Transportation (MTQ) recently collaborated to conduct a project on the long-term performance of recycled and virgin HDPE pipes supplied by local manufacturers. This project included investigating stress crack resistance (SCR) and predicting the service life of new corrugated HDPE pipes fabricated by Canadian manufacturers as well as corrugated HDPE pipes in field service in Quebec under natural aging. Notched specimens were cut directly from the HDPE pipe liner to evaluate their SCR. In addition, four exposure conditions were used: freeze–thaw cycles and exposure to saline, acidic, and alkaline solutions to predict the remaining lifetime of pipes. The SCR was investigated with the Florida Test Method in water at 450 psi (3.10 MPa) and 80°C, 650 psi (4.48 MPa) and 80°C, and 650 psi (4.48 MPa) and 70°C in compliance with FM 5-573. The data points at elevated temperatures were then shifted to predict the service life of pipes at lower service conditions with the Popelar's Shift Method (PSM) and the Rate Process Method (RPM), both of which are extrapolation methods. The results show that, before immersion in different solutions, the recycled could guarantee 100 years of service life as virgin pipes under service conditions of 10°C and 500 psi (3.45 MPa). After immersion, the decrease in SCR was related to the two steps of elevated temperature in the conditioned test and the Florida test, along with the harsh environment of the conditioned test. Moreover, RPM proved better than PSM, because the latter yielded nearly scattered, nonlinear data points.

Keywords: High-density polyethylene (HDPE) pipes; Acceleration aging; Natural aging; Stress crack resistance; Popelar's Shift Method (PSM); Rate Processing Method (RPM); Recycled HDPE pipes; service life.

6.1 Introduction

Corrugated high-density polyethylene (HDPE) pipes have become one of the most attractive materials used in underground structures such as for drainage and sewer applications due to their good properties. They are flexible, lightweight, and corrosion resistant. Moreover, they are easy to install with or without trenchless and relining techniques [1,4,34,35] and involve low initial and maintenance costs. All these characteristics combined with the possibility of manufacturing HDPE pipes from recycled resins are outstanding advantages for them over steel and concrete pipes [43,45-47]. Some concerns about the long-term behavior of HDPE still exist after several years of service. Moreover, it has not been fully established that certain degradations (such as the appearance of cracks due to high loads or some oxidation) will not occur during pipe service life. Pipe service conditions vary widely depending on geographic location, installation techniques, temperature, type of surrounding soil, and traffic load. Therefore, it is important to understand the corrugated pipe degradation mechanism to avoid any long-term performance problems in road drainage systems. In general, there are three types of pipe failure depending on time and stress level. They are ductile failure (stage I), brittle failure (slow crack growth: stage II), and brittle failure (chemical degradation: stage III) (Fig. 2.2) [116]. Stage I (ductile failure) occurs once HDPE pipes have been subjected to high stresses (normally >30% of yield). Unlike with pressure pipes, however, stage I failure does not usually occur with pipes used in road drainage systems. Their service stresses are usually lower than the equilibrium yield stress [46]. Stages II and III failure occur with no apparent yield deformation and many cracks starting at the same time [11]. In these stages, chemical degradation occurs when the pipes are subjected to an environment generating oxygen radicals. Using additives (e.g., carbon black, antioxidants, and UV stabilizers) during pipe manufacturing can delay stage III initiation formation [79,80]. Stage III oxidation/degradation is, however, independent of time. Hence, stage II involving slow crack growth (SCG) under intermediate stress is the primary concern for road drainage systems. This failure stage is mainly dominated by mechanical stress or a combination of both stress and a chemically aggressive environment [116]. That is why critical failure mechanisms for long-term applications are found in stage II. Pipe service lifetime is related to slow crack growth by (1) crack initiation and (2) crack propagation phases [162]. The inevitable initial defects such as impurities and material inhomogeneities cause the formation of the crack initiation phase. In contrast, the crack propagation phase is related to the stress crack resistance (SCR) of pipes [163,164].

A 100-year service life is considered as one of the requirements for modern corrugated PE pipe grades for road drainage systems. Pipes manufactured from recycled resins have also been a concern in recent years [11,43,45-47,64,116,165]. As recycled materials are available from consumer HDPE products (detergent bottles and milk jugs), the pipes made from recycled HDPE materials are becoming increasingly sustainable and cost competitive. Recycled products can reduce the carbon footprint, the energy consumption in producing raw materials, and greenhouse-gas emissions compared to virgin products [60,62,166]. Therefore, it is important to evaluate the stress crack resistance for recycled pipes in order to estimate their service life. In addition, attention should be paid to estimating the remaining lifetime of a pipeline after a period of use to assess the influence of service conditions on pipe degradation [167,168]. The long-term stress crack resistance (SCR) of corrugated HDPE pipes has been investigated with many accelerated laboratory tests: environmental stress crack resistance (ESCR) [24,78], the Pennsylvania Notched Test (PENT) [23,91,169,170], the Notched Constant Ligament Stress (NCLS) [22,46,80,92], the Un-Notched Constant Ligament Stress (UCLS) [47,107,171], and the Florida Test Method [18,25,106]. All these testing methods have pros and cons. The Florida Test Method [25] is becoming more common because of its short testing time and correlated to the field conditions as opposed to the other methods [116]. Hsuan and McGrath (2005) [18] first conducted this test method to estimate the 100-year service life of HDPE pipe. The stress crack resistance (SCR) of pipe liners and junctions was performed in water at 650 psi and 70°C, as well as at 650 and 450 psi and 80°C. The high-temperature rupture times were then extrapolated using Popelar's Shift Method (PSM) and the Rate Process Method (RPM) to determine long-term life under service conditions [172]. The results of these studies provided the foundation for establishing the specification for 100-year stress crack resistance of corrugated HDPE pipes.

Only a few studies have focused on the stress crack resistance and life prediction of recycled HDPE corrugated pipes used in road drainage systems [11,43,45-47,64,116,165]. According to NCHRP Research Report 870 [47], AASHTO M294 [3] and ASTM F2306/F2306M [65] were updated in 2018, and in 2019, respectively, to establish specific requirements for recycled HDPE corrugated pipes. In addition, past studies [46,47] have concluded that recycled pipes can guarantee 100 years of service just as virgin pipes, as long as certain test results for UCLS and elongation at break are met. A specification of the 100-year SCR for virgin corrugated HDPE pipes was also established based on research by Hsuan et al. (2007) [172]. Recycled pipes were not however mentioned in this specification. Moreover, the long-term service life of HDPE pipes depends on the manufacturing, installation process, area characteristics, and specific service conditions. The objective of our study was to investigate the stress crack resistance of recycled and virgin HDPE corrugated pipes provided by Canadian manufacturers and used in road drainage systems. Furthermore, the effect of service conditions on pipe lifetime was also investigated by aging pipe specimens under laboratory conditions including freeze–thaw cycles as well as exposure to saline (NaCl), acidic (H_2SO_4), and alkaline (NaOH) solutions. The study also investigated the stress crack resistance of HDPE pipes in field service in Quebec under natural aging. The Florida Test Method was used in our study. The data points from SCR tests were shifted to generate a master curve using both the Popelar's Shift Method (PSM) and the Rate Process Method (RPM). Based on the extrapolation results, the 100-year service life and remaining lifetime of specimens exposed to different solutions were predicted.

6.2 Materials and Test Conditions

6.2.1 Materials

A total of six corrugated HDPE pipes from four different Canadian manufacturers, used for nonpressure road drainage systems were investigated. Figure 3.1 shows the corrugated HDPE pipes under investigation. In the present study, these pipes are manufactured according to class A of BNQ 3624-120 (2016) [19] for roads and highways (Table 6.1). It should be noted that these pipes are manufactured by local manufacturers in Quebec province. The requirements for these pipes are therefore out of the AASHTO M294 (2018) [3] range (Table 6.2). In accordance with the ASTM D3350 (2014) classification [29], the pipes had a density greater than 0.941 g/cm^3. The pipe materials were stabilized with 2% to 4% carbon black, antioxidants, and a UV absorber. Two of the pipes were manufactured with postconsumer recycled resins (A-R and D-R), one with postindustrial recycled resin (B-R), and three with virgin resins (A-V, B-V, and C-V). The letters A, B, C, and D designate the manufacturers; R is for recycled resin; and V for virgin resin. Postconsumer recycled (PCR) materials come from household waste (detergent bottles and milk jugs). They are shredded, washed, stabilized, and pelletized by the pipe manufacturers. In contrast, postindustrial recycled (PIR) “reworked” materials come from products damaged during the manufacturing process. The regrind pipe is produced by shredding, mixing with the raw material, and drawing by an extrusion process. This type of pipe is considered a pseudo-source of recycled resins. The external diameter and length of each pipe were 900 mm and 3000 mm, respectively. Table 4.1 summarizes the detailed properties of the pipes from the results of our previous studies [173]. It should be highlighted however that some of the characteristics of B-R, B-V, and C-V are not compatible with the class classification by the ASTM D3350 [29].

Table 6.1 Classification of investigated HDPE pipes according to BNQ 3624-120 standard

Characteristics	Class A	Class B
Nominal Diameter	75 mm to 1500 mm	300 mm to 900 mm
Category	R320, R140, R125, R110, and R95	R210
Classification by properties of ASTM D3350	PE435400	PE424420

Table 6.2 Cell classification limits of HDPE pipes for BNQ 3624-120 and AASHTO M294

Property	AASHTO M294	BNQ 3624-120
Density, g/cm^3	> 0.947 – 0.955	> 0.945
Melt flow index (MFI), g/10min	< 0.4	< 0.15
Carbon black, %	2 – 4	2 - 4
Ash, %	< 0.5	< 2
Polypropylene, %	< 5	< 5
Flexural modulus, MPa (psi)	> 896 (> 130000)	552 – 758 (80000 – 110000)
Yield stress, MPa (psi)	> 24 (> 3500)	21 – 24 (3000 – 3500)
NCLS, hours	36	> 17
Oxidation induction time (OIT), minutes	50	10

6.2.2 Laboratory-Accelerated Aging Tests

To simulate the service condition of exposure in Quebec (Canada), SCR specimens taken from the pipe liner were conditioned in different chemical solutions. The dimension and geometry of the specimens are provided below. Four exposure conditions were used: freeze–thaw cycles and expose to saline, acidic, and alkaline solutions. Freeze–thaw cycles are a major concern for infrastructure in Quebec. The cold condition could create additional internal stresses (shrinkage and expansion) and causes internal cracks in materials. Specimens were immersed in water for 625 cycles between -40^{0}C and +40^{0}C. Each cycle consisted in 8 hours of freezing and 6 hours of thawing in water. To simulate de-icing salts, specimens were immersed in saline solution (1 M NaCl). For the acidic and alkaline service conditions, sulfuric acid (H_2SO_4) at a pH of 2 and sodium hydroxide (NaOH) at a pH of 10 were used, respectively. For immersion tests, the different chemical solutions were conditioned at 60^{0}C. As mentioned in past studies, acceleration tests are valid when only the temperature affects the rate of degradation [108,116,174,175]. It should be pointed out, that the temperature selected for accelerated aging tests was not high enough to modify the nature of the material or to consume carbon black and antioxidants. Also, it doesn’t modify the degradation mechanism. To prevent this from occurring, the upper temperature limit for immersion tests of HDPE pipes should not exceed the softening point of 60^{0}C. After 12 months, the specimens were removed from the solutions and the stress crack resistance was determined to estimate the remaining lifetime of the pipes.

6.2.3 HDPE Pipes in Field Service in Quebec under Natural Aging

Five commercial corrugated HDPE pipes (P1, P2, P3, P4, and P5) made of virgin resin were collected from different field locations in Quebec (Canada) (Fig. 6.1). These pipes have been in service for 5 to 14 years. They were manufactured in Canada according to the provisions for class A in BNQ 3624-120 (2016) [19]. The pipes had a density greater than 0.941 g/cm^3 and were stabilized with 2% to 4% carbon black. The SCR specimens taken from the pipe liner with the dimension and geometry are provided below. It should be noted that the pipes exposed to Quebec weather conditions were examined to provide an overview of the stress crack resistance in laboratory-accelerated aging and in real-time in situ. The present study focused on the climate impact of Canada's winter (e.g., low temperature, temperature variations between day and night, and freeze-thaw cycles) on the SCR of pipes. Table 6.3 summarizes the information of the in-service duration, installation time, and repair time for five pipes in field service in Quebec

Figure 6.1 HDPE pipes in field service in Quebec

Table 6.3 Technical parameters of HDPE pipes in field service in Quebec

Sample	Location (Quebec province)	Diameter (mm)	Year of construction	Year rehabilitation	Sample collection year	Years of use
P1	Nicolet	900	2013	-	2018	5
P2	Grand-Saint-Esprit	1200	2010	-	2018	8
P3	Chesterville	1200	2003	-	2018	15
P4	Bouchette	900	-	2004	2018	14
P5	Bois-Franc	900	-	2004	2018	14

6.2.4 Stress Crack Resistance (SCR) Tests

Specimens were cut from the pipe liner and adjusted in the longitudinal direction of pipes (Figs. 6.2a and 6.2b) to evaluate the stress crack resistance of the pipes. This should be highlighted as the polymer molecule orientation is stretched in the longitudinal direction which is the fabrication direction. Therefore, SCR specimens were punched directly from the pipe liner with a stainless-steel die. The dimension and geometry of the SCR specimens were the same as defined in ASTM F2136 (2018) [22] with a notch (Fig. 6.2c). The notch depth was 20% of the average thickness of the pipe liner in order to control the initial cracks. The thickness of the specimens cut from pipes A-R, A-V, B-R, B-V, C-V, and D-R were 4.80, 4.40, 7.50, 7.60, 2.70, and 3.80 mm, respectively. For the pipes in field service, the specimen thickness of pipes P1, P2, P3, P4, and P5 were 4.40, 4.80, 4.50, 7.00, and 7.80 mm, respectively.

The SCR tests were performed according to FM 5-572 (2008) [106], procedure C, using the Florida Test Method (FM 5-573 2008) [25]. The failure time of conditioned and unconditioned SCR specimens was measured in water at 650 psi and 70^0C and at 650 and 450 psi at 80^0C (Fig. 6.2d). Five replicates were tested for each condition of pressure/temperature. All 15 data points (5 x 3 = 15) were then shifted to obtain a master curve using both the Popelar's Shift Method (PSM) and the Rate Process Method (RPM).

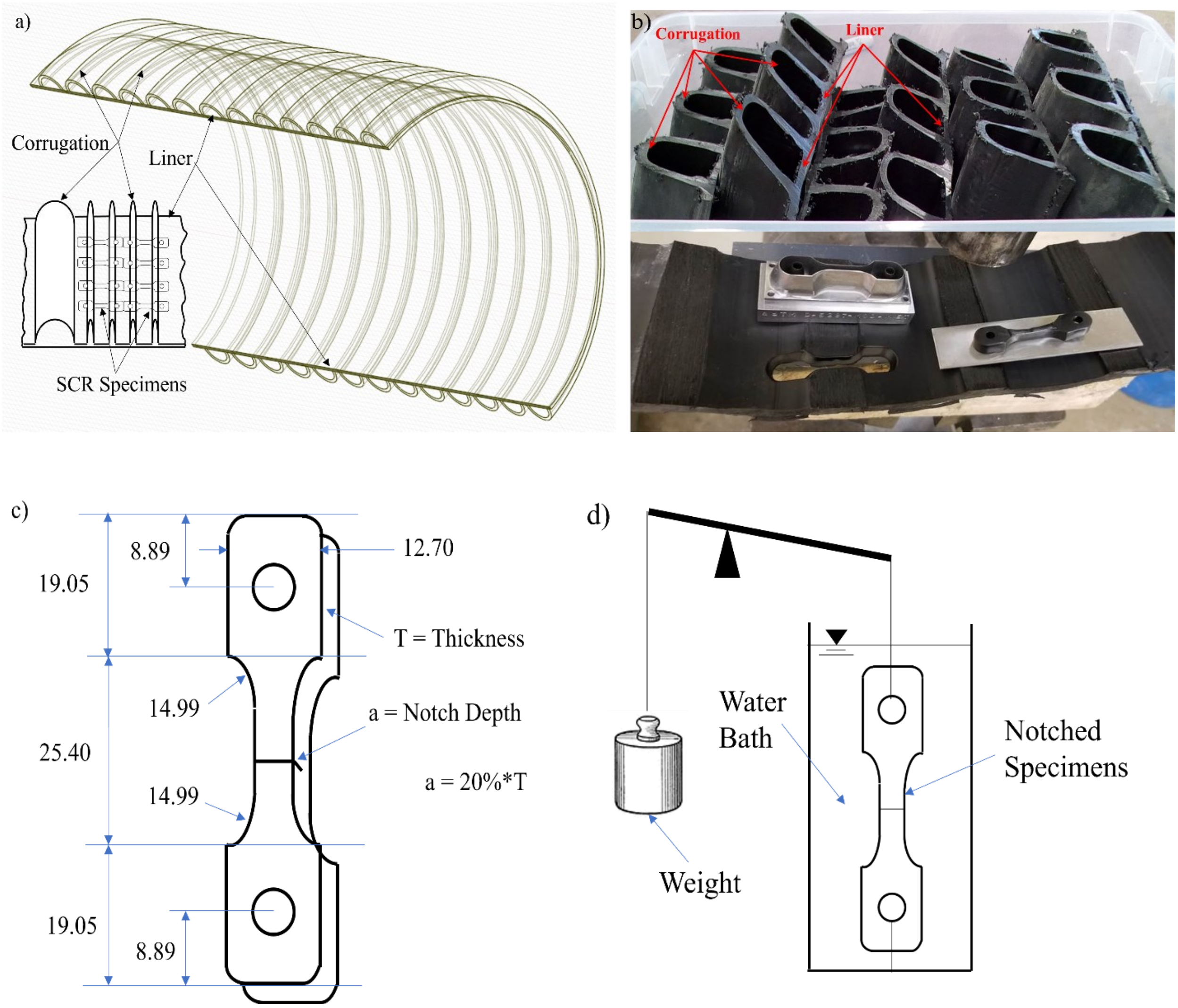


Figure 6.2 SCR measurement a) Sectional view of HDPE pipe, b) SCR specimens c) Dimension of SCR specimens (mm), d) Experimental setup

6.3 Analytical Methods

Two extrapolation methods—Popelar's Shift Method (PSM) and the Rate Process Method (RPM)—were used to estimate the service life of HDPE pipes. These methods can be used to shift data from the elevated temperature to the service temperature (reference temperature) to produce a master curve. From the historical weather record in Quebec, the annual average temperature is 10^0C [176]. Figure 6.3 gives the variation in minimum and maximum daily temperature and snowfall records at the Quebec measuring station from 2000 to 2020. In our study, service conditions at 500 psi and 10^0C were therefore used to estimate a 100-year service life.

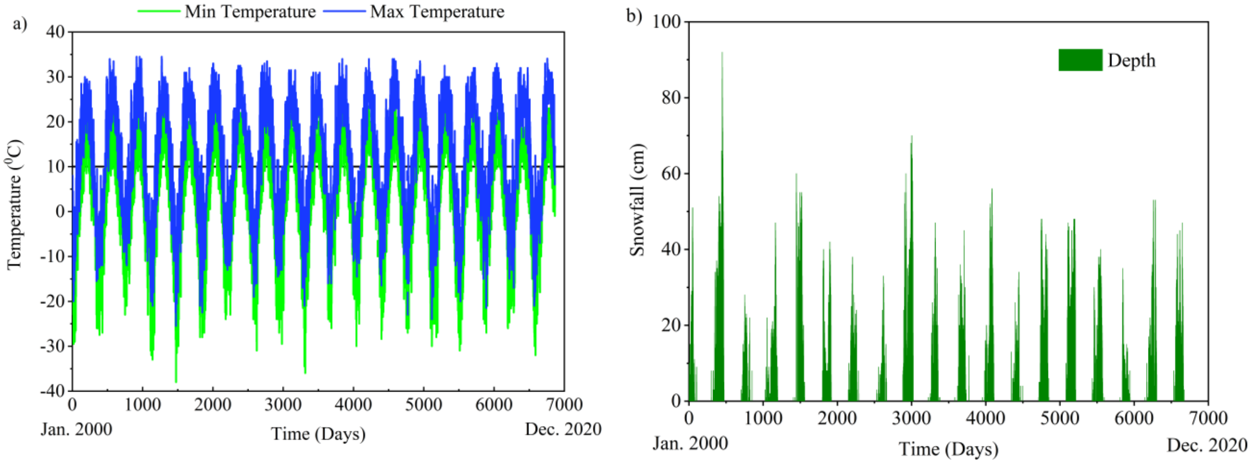


Figure 6.3 The historical weather record in Quebec a) the variation in temperature extremities, b) snowfall at the Drummondville climatological monitoring station (2000–2020) [176].

6.3.1 Popelar's Shift Method (PSM)

Popelar et al. (1991) [96] developed the horizontal shift factor (a_T) and the vertical shift factor (b_T) based on the stress relaxation testing of MDPE (medium-density polyethylene) and HDPE gas pipes, as shown in Eqns. 2.2 and 2.3. The failure time at elevated temperatures can be shifted along time and stress axes. The horizontal shift factor was established based on the time–temperature superposition principle, whereas the vertical shift factor involved changes in crystalline structure with temperature. The failure time at elevated temperature can be shifted to a lower temperature using the (a_T) factor. In contrast, the (b_T) factor was applied to shift the stress at elevated temperature to a lower temperature.

$$a_T \text{ (time shift factor)} = \exp[0.109(T-T_R)] \tag{2.2}$$

$$b_T \text{ (stress shift factor)} = \exp[0.0116(T-T_R)] \tag{2.3}$$

where a_T is the horizontal shift factor, b_T is the vertical shift factor, T is the temperature (K), and T_R is the service temperature (reference temperature) (K).

6.3.2 Rate Process Method (RPM)

The Rate Process Method (RPM) is well-known as a common method for estimating the lifetime of gas pipes. The theory of this method was developed based on the principle of time–temperature superposition. Data extrapolation is performed assuming that the failure time conforms to an Arrhenius equation, where the energy responds linearly to the logarithm of stress. Nowadays, RPM is also applied to predict the service life of corrugated pipes used in transportation infrastructure applications. This method can be used to evaluate the pipe SCR based on ISO 9080 (2012) [72] or ASTM D2837-13e1 (2013) [73]. A four-coefficient model is used in ISO 9080, while three coefficients are applied in ASTM D2837 to extrapolate the data. In our study, three coefficients as expressed in Eq. 2.4 are solved by using a least-squares multivariable linear regressions method from the SCR data points. Once these constants are known, a master curve can be generated to estimate the service life of pipes.

$$\log(t) = A + B/T + C.\log(\sigma)/T \qquad (2.4)$$

where t is the failure time (hours); σ is the applied stress (psi); T is the test temperature (K); and A, B, and C are constants.

6.4 Results and Discussions

6.4.1 SCR Results

The failure time of pipe-liner specimens before and after immersion in various chemical solutions was determined with the Florida Test Method in water at 450 psi and 80^0C, at 650 psi and 80^0C, and at 650 psi and 70^0C, as summarized in Tables 6.4 and 6.5.

Table 6.4 Time to failure of pipes before immersion in the various solutions.

Sample	Condition	Failure Time (hours)					Avg. (h)	SD (h)	COV (%)
		1	2	3	4	5			
A-R	450 psi/80°C	10.7	9.6	9.7	10.0	11.4	10.28	0.76	7.39
	650 psi/80°C	4.3	4.3	4.2	3.8	4	4.12	0.22	5.26
	650 psi/70°C	17.9	16.8	17.8	17.8	18.2	17.70	0.53	2.30
A-V	450 psi/80°C	145.2	120.9	123.2	126.1	145.0	132.08	12.02	9.11
	650 psi/80°C	90.9	199.0	81.6	123.0	101.7	119.24	47.18	39.57
	650 psi/70°C	297.4	292.2	632.0	338.2	377.9	387.54	141.0	36.38
B-R	450 psi/80°C	>2491	>2491	>2491	>2491	>2491	>2491	N/A	N/A
	650 psi/80°C	113.6	164.3	113.7	113.4	113.3	123.66	22.72	18.37
	650 psi/70°C	>2706	>2706	>2706	>2706	>2706	>2706	N/A	N/A
B-V	450 psi/80°C	>2491	>2491	>2491	>2491	>2491	>2491	N/A	N/A
	650 psi/80°C	>2491	>2491	>2491	>2491	>2491	>2491	N/A	N/A
	650 psi/70°C	>2491	>2491	>2491	>2491	>2491	>2491	N/A	N/A
C-V	450 psi/80°C	230.6	164.3	119.2	123.5	98.3	147.18	52.40	35.61
	650 psi/80°C	82.9	113.5	88.8	73.9	42.9	80.40	25.60	31.83
	650 psi/70°C	111.1	119.8	123.6	110.9	110.9	115.26	6.03	5.23
D-R	450 psi/80°C	225.3	205.2	230.2	387.5	261.2	261.88	73.03	27.89
	650 psi/80°C	14.6	20.1	18.3	24.4	21.7	19.82	3.68	18.56
	650 psi/70°C	99.8	85.3	84.9	78.5	137.6	97.22	23.88	24.57

Note: N/A = not available; SD = standard deviation; COV = coefficient of variation.

Table 6.5 Time to failure of pipes after immersion in the various solutions.

Sample	Condition	Saline			Alkaline			Acidic			Freeze–thaw		
		Avg. (h)	SD (h)	COV (%)	Avg. (h)	SD (h)	COV (%)	Avg. (h)	SD (h)	COV (%)	Avg. (h)	SD (h)	COV (%)
A-R	450 psi/80°C	12.30	4.89	39.7	25.20	13.62	54.0	9.30	3.89	41.9	12.60	7.62	60.5
	650 psi/80°C	6.30	1.49	23.7	6.50	4.39	67.5	5.50	3.73	67.8	7.10	4.12	58.1
	650 psi/70°C	19.97	4.81	24.1	18.26	7.89	43.2	18.15	4.48	24.7	18.29	16.89	92.3
A-V	450 psi/80°C	120.90	20.85	17.2	145.1	23.41	16.1	93.30	64.25	68.9	144.77	63.31	43.7
	650 psi/80°C	83.00	25.89	31.2	116.1	22.41	19.3	118.30	40.94	34.6	106.33	39.35	37.0
	650 psi/70°C	201.90	20.83	10.3	202.0	47.47	23.5	387.50	102.10	26.4	304.40	51.54	16.9
B-R	450 psi/80°C	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A
	650 psi/80°C	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A
	650 psi/70°C	>300	N/A	N/A	>300	N/A	N/A	>300	N/A	N/A	>300	N/A	N/A
B-V	450 psi/80°C	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A
	650 psi/80°C	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A	>1400	N/A	N/A
	650 psi/70°C	>500	N/A	N/A	>500	N/A	N/A	>500	N/A	N/A	>500	N/A	N/A
C-V	450 psi/80°C	60.46	13.69	22.6	46.57	12.40	26.6	71.27	13.31	18.7	98.7	63.05	63.9
	650 psi/80°C	14.52	4.07	28.0	33.98	5.23	15.4	13.64	12.17	89.2	13.67	15.90	116
	650 psi/70°C	46.5	30.37	65.3	62.72	14.31	22.8	47.13	15.63	33.2	33.46	17.57	52.5
D-R	450 psi/80°C	123.47	38.92	31.5	145.1	28.58	19.7	118.30	21.64	18.3	133.92	3.71	2.8
	650 psi/80°C	68.09	19.08	28.0	116.1	25.96	22.4	93.30	4.59	4.9	117.18	20.91	17.8
	650 psi/70°C	214.25	70.86	33.1	202.0	15.79	7.8	387.50	34.98	9.0	304.40	90.71	29.8

Note: N/A = not available; SD = standard deviation; COV = coefficient of variation

The results show that the time to failure of pipes made with recycled resins was faster than those made with virgin resins in the case of manufacturers A and B. Adding carbon black reduced the SCR of the pipes. Recycled pipes from both manufacturers A and B had higher carbon-black contents than virgin pipes (Table 3). Variations in the SCR test results for pipes from different manufacturers are inevitable. This can also be true for specimens from the same manufacturer. Many studies [10,80,172] have reported this variation. Hsuan et al. (2007) [172] found the coefficient of variation of failure time among test specimens can be as high as ±60%. In our study, this coefficient of variation was found to be less than 40% for unincubated specimens and greater than 60% for incubated specimens exposed to various chemical solutions. In addition, the manufacturing process also significantly affects pipe SCR. This information is, however, considered an industrial secret and not available to the authors. In addition, this variation could also be due to machining of the notches (ASTM F2136 [22]). The Florida SCR testing method specifies that notched or unnotched specimens can be used for the test. Notching the specimens works as a crack initiator and induces rupture at the notching location. Thomas (2011) [46] reported that notching HDPE pipes manufactured with recycled resins could alter their SCR. Therefore, the authors recommended using unnotched specimens for the SCR of HDPE pipes made with recycled resins. Nevertheless, in this study, notched samples were used to estimate the SCR of all pipes since the research project included virgin resins. This was decided because unnotched specimens might not rupture for a long time. Moreover, all B-R and B-V specimens experienced plastic deformation (yield) before and after immersion in the solutions. This type of behavior might be due to the fact that the applied stresses were greater than those producing brittle fractures. It should be noted that specimens B-R and B-V had low tensile strength (Table 3.1), which could explain this behavior.

Tables 6.4 and 6.5 show that exposure environment and high temperature are factors that can alter pipe SCR. The two steps of elevated temperature: (1) incubation of the specimens in the laboratory for 12 months at 60^0C, and (2) the Florida test at high temperature (70^0C, 80^0C), indicates that the time to failure of the incubated specimens was shorter than that of the unincubated specimens. The time to failure of specimens in the various solutions could be similar. Table 6.6, however, shows that the failure time was shortest with the saline environment. This value is (1.20–1.49), (1.04–1.38), and (1.02–1.41) times slower for the alkaline, acidic solutions, and freeze–thaw cycles than for the saline solution, respectively.

Table 6.6 Failure time ratio between solutions.

Sample	Condition	Failure Time Ratio		
		Alkaline/saline	Acidic/saline	Freeze–thaw/saline
A-R	450 psi/80°C	2.05	0.76	1.02
	650 psi/80°C	1.03	0.87	1.13
	650 psi/70°C	0.91	0.91	0.92
	Average	1.33	0.85	1.02
	SD	0.62	0.08	0.11
	COV	0.47	0.09	0.10
A-V	450 psi/80°C	1.20	0.77	1.20
	650 psi/80°C	1.40	1.43	1.28
	650 psi/70°C	1.00	1.92	1.51
	Average	1.20	1.37	1.33
	SD	0.20	0.58	0.16
	COV	0.17	0.42	0.12
C-V	450 psi/80°C	0.77	1.18	1.63
	650 psi/80°C	2.34	0.94	0.94
	650 psi/70°C	1.35	1.01	0.72
	Average	1.49	1.04	1.10
	SD	0.79	0.12	0.48
	COV	0.53	0.12	0.43
D-R	450 psi/80°C	1.18	0.96	1.08
	650 psi/80°C	1.71	1.37	1.72
	650 psi/70°C	0.94	1.81	1.42
	Average	1.27	1.38	1.41
	SD	0.39	0.43	0.32
	COV	0.31	0.31	0.23

6.4.2 Popelar's Shift Method (PSM)

As discussed earlier, each data point at the elevated temperature can be shifted to the lower service temperature using the horizontal and vertical shift factors, as shown in Eqns. 2.1 and 2.2. With PSM, the shift factors $b_T(80^0C)$ of 2059.050 and $b_T(70^0C)$ of 692.287 are used to shift the stress from 80^0C and 70^0C to 10^0C, while the shift factors $a_T(80^0C)$ of 2.252 and $a_T(70^0C)$ of 2.006 were used to shift the failure time from 80^0C and 70^0C to 10^0C. The data points at the elevated temperature of the investigated specimens before and after immersion in the various solutions were shifted to service conditions at 10^0C and 500 psi to estimate pipe lifetime as presented in Tables 6.7, 6.8, 6.9, and 6.10.

Table 6.7 Average failure test data of A-R shifted to 10°C and 500 psi with PSM.

Solutions	Condition	Avg. Failure Time (h)	Shifted Stress (psi)	Shifted Time (h)	Shifted Stress – Log (psi)	Shifted Time – Log (h)
Nonconditioned	450 psi/80°C	10.28	1013.584	21167.034	3.006	4.326
	650 psi/80°C	4.12	1464.065	8483.286	3.166	3.929
	650 psi/70°C	17.70	1303.714	12253.472	3.115	4.088
Saline	450 psi/80°C	12.30	1013.584	25326.315	3.006	4.404
	650 psi/80°C	6.30	1464.065	12972.015	3.166	4.113
	650 psi/70°C	19.97	1303.714	13824.963	3.115	4.141
Alkaline	450 psi/80°C	25.20	1013.584	51888.061	3.006	4.715
	650 psi/80°C	6.50	1464.065	13383.825	3.166	4.127
	650 psi/70°C	18.26	1303.714	12641.153	3.115	4.102
Acidic	450 psi/80°C	9.30	1013.584	19149.165	3.006	4.282
	650 psi/80°C	5.50	1464.065	11324.775	3.166	4.054
	650 psi/70°C	18.15	1303.714	12565.001	3.115	4.099
Freeze–thaw	450 psi/80°C	12.60	1013.584	25944.030	3.006	4.414
	650 psi/80°C	7.10	1464.065	14619.255	3.166	4.165
	650 psi/70°C	18.29	1303.714	12661.922	3.115	4.102

Table 6.8 Average failure test data of A-V shifted to 10°C and 500 psi with PSM.

Solutions	Condition	Avg. Failure Time (h)	Shifted Stress (psi)	Shifted Time (h)	Shifted Stress – Log (psi)	Shifted Time – Log (h)
Nonconditioned	450 psi/80°C	132.08	1013.584	271959.327	3.006	5.435
	650 psi/80°C	119.24	1464.065	245521.124	3.166	5.390
	650 psi/70°C	387.54	1303.714	268288.741	3.115	5.429
Saline	450 psi/80°C	120.90	1013.584	248939.147	3.006	5.396
	650 psi/80°C	83.00	1464.065	170901.152	3.166	5.233
	650 psi/70°C	201.90	1303.714	139772.660	3.115	5.145
Alkaline	450 psi/80°C	145.10	1013.584	298768.158	3.006	5.475
	650 psi/80°C	116.10	1464.065	239055.707	3.166	5.378
	650 psi/70°C	202.00	1303.714	139841.889	3.115	5.146
Acidic	450 psi/80°C	118.30	1013.584	243585.617	3.006	5.387
	650 psi/80°C	93.30	1464.065	192109.367	3.166	5.284
	650 psi/70°C	387.50	1303.714	268261.049	3.115	5.429
Freeze–thaw	450 psi/80°C	144.77	1013.584	298088.671	3.006	5.474
	650 psi/80°C	106.33	1464.065	218938.787	3.166	5.340
	650 psi/70°C	304.40	1303.714	210732.034	3.115	5.324

Table 6.9 Average failure test data of C-V shifted to 10°C and 500 psi with PSM.

Solutions	Condition	Avg. Failure Time (h)	Shifted Stress (psi)	Shifted Time (h)	Shifted Stress – Log (psi)	Shifted Time – Log (h)
Nonconditioned	450 psi/80°C	147.18	1013.584	303050.982	3.006	5.482
	650 psi/80°C	80.40	1464.065	165547.622	3.166	5.219
	650 psi/70°C	115.26	1303.714	79792.951	3.115	4.902
Saline	450 psi/80°C	60.46	1013.584	124490.164	3.006	5.095
	650 psi/80°C	14.52	1464.065	29897.406	3.166	4.476
	650 psi/70°C	46.50	1303.714	32191.326	3.115	4.508
Alkaline	450 psi/80°C	46.57	1013.584	95889.959	3.006	4.982
	650 psi/80°C	33.98	1464.065	69966.520	3.166	4.845
	650 psi/70°C	62.72	1303.714	43420.214	3.115	4.638
Acidic	450 psi/80°C	71.27	1013.584	146748.495	3.006	5.167
	650 psi/80°C	13.64	1464.065	28085.442	3.166	4.448
	650 psi/70°C	47.13	1303.714	32627.466	3.115	4.514
Freeze-thaw	450 psi/80°C	98.7	1013.584	203228.237	3.006	5.308
	650 psi/80°C	13.67	1464.065	28147.214	3.166	4.449
	650 psi/70°C	33.46	1303.714	23163.909	3.115	4.365

Table 6.10 Average failure test data of D-R shifted to 10°C and 500 psi with PSM.

Solutions	Condition	Avg. Failure Time (h)	Shifted Stress (psi)	Shifted Time (h)	Shifted Stress – Log (psi)	Shifted Time – Log (h)
Nonconditioned	450 psi/80°C	261.88	1013.584	539224.019	3.006	5.732
	650 psi/80°C	19.82	1464.065	40810.371	3.166	4.611
	650 psi/70°C	97.22	1303.714	67304.101	3.115	4.828
Saline	450 psi/80°C	123.47	1013.584	254230.906	3.006	5.405
	650 psi/80°C	68.09	1464.065	140200.716	3.166	5.147
	650 psi/70°C	214.25	1303.714	148322.399	3.115	5.171
Alkaline	450 psi/80°C	145.10	1013.584	298768.158	3.006	5.475
	650 psi/80°C	116.10	1464.065	239055.707	3.166	5.378
	650 psi/70°C	202.0	1303.714	139841.889	3.115	5.146
Acidic	450 psi/80°C	118.30	1013.584	243585.617	3.006	5.387
	650 psi/80°C	93.30	1464.065	192109.367	3.166	5.284
	650 psi/70°C	387.50	1303.714	268261.049	3.115	5.429
Freeze–thaw	450 psi/80°C	133.92	1013.584	275747.979	3.006	5.441
	650 psi/80°C	117.18	1464.065	241279.481	3.166	5.383
	650 psi/70°C	304.40	1303.714	210732.034	3.115	5.324

6.4.3 Rate Process Method (RPM)

For the RPM method, the three constants—A, B, and C—in Eq. 6.1 were calculated to predict the failure time with 97.5% lower confidence at 10°C and 500 psi. In this study, a least-squares multivariable linear regression was used to determine these constants from all data points, as listed in Table 6.11. In other words, coefficients A, B, and C can be solved using matrix algebra, as shown in Eq. 6.1.

$$\begin{pmatrix} \log(t_1) \\ \log(t_2) \\ \log(t_3) \end{pmatrix} = \begin{pmatrix} 1 & 1/353 & \log(450)/353 \\ 1 & 1/353 & \log(650)/353 \\ 1 & 1/343 & \log(650)/343 \end{pmatrix} \begin{pmatrix} A \\ B \\ C \end{pmatrix} \tag{6.1}$$

where t_1, t_2, and t_3 are the failure time (hours) at 450 psi and 80^0C, at 650 psi and 80^0C, and at 650 psi and 70^0C, respectively; A, B, and C are constants.

Table 6.11 RPM coefficients.

Sample	Coefficient	Nonconditioned	Saline	Alkaline	Acidic	Freeze–thaw
A-R	A	-21.112	-16.421	-15.776	-18.828	-11.173
	B	10135.532	7807.529	9639.731	8480.103	5707.120
	C	-876.771	-615.758	-1352.963	-566.625	-529.302
A-V	A	-15.645	-11.833	-6.124	-19.241	-14.247
	B	6658.868	5938.035	3505.043	8128.460	6477.214
	C	-146.218	-387.209	-219.170	-227.897	-263.063
C-V	A	-4.173	-13.729	-7.434	-20.648	-17.926
	B	3774.612	9128.795	3967.149	12774.275	12909.486
	C	-581.741	-1378.040	-285.664	-1821.734	-2222.375
D-R	A	-22.298	-15.065	-6.402	-19.212	-11.848
	B	15263.825	7551.674	3594.278	8088.617	5301.216
	C	-2466.097	-565.423	-216.147	-217.395	-138.817

6.4.4 Predicting SCR Service Life

This study used two extrapolation methods—PSM and RPM—in order to predict the service life of corrugated HDPE pipes at a service condition of 10^0C and 500 psi. Figures 6.4 (a, b, c, and d) and Figures 6.5 (a, b, c, and d) present the brittle curve slopes as a function of the stress and failure time at 10^0C for the unincubated and incubated specimens, respectively.

RPM revealed that, prior to immersion in the various solutions, the pipes had lifetimes exceeding 100 years (except for pipe C-V). PSM also yielded similar results for pipes C-V and D-R. However, dispersions of test data results are found in PSM. The coefficient of determination (R-squared) was low for pipes A-V (0.67) and C-V (0.39) (Table 6.12). In addition, pipes B-R and B-V were not analyzed since there was insufficient data of failure time under these test conditions.

RPM revealed that, after immersion in the various solutions, the pipes had shorter lifetimes than before immersion. Table 6.12 shows that a 12-month incubation and elevated testing temperatures exceeding 60°C (the softening temperature of HDPE pipes) in the various chemical solutions changed the SCR of the pipes. It should be noted that laboratory aging conditions are harsher than actual field conditions (natural aging). In addition, all sides of the specimens were in direct contact with the solutions. Therefore, the lifetime of most of the aged specimens was less than 100 years. Specimens A-V, C-V, and D-R, however, exhibited longer lifetimes after exposure to the acidic solution than the unincubated specimens. This can be explained by the large deviation between the experimental results; the coefficient of variation in all the measurements was greater than 60% (Table 6.5). PSM also revealed that incubated specimens had shorter predicted lifetimes than unincubated specimens, with exception of A-V specimens. Incubated specimens A-V, C-V, and D-R had predicted lifetimes in excess of 100 years. Nevertheless, PSM yielded scattered and nonlinear data. In most of the experimental results, the coefficient of variation was greater than 60% and the coefficient of determination (R-squared) less than 0.5.

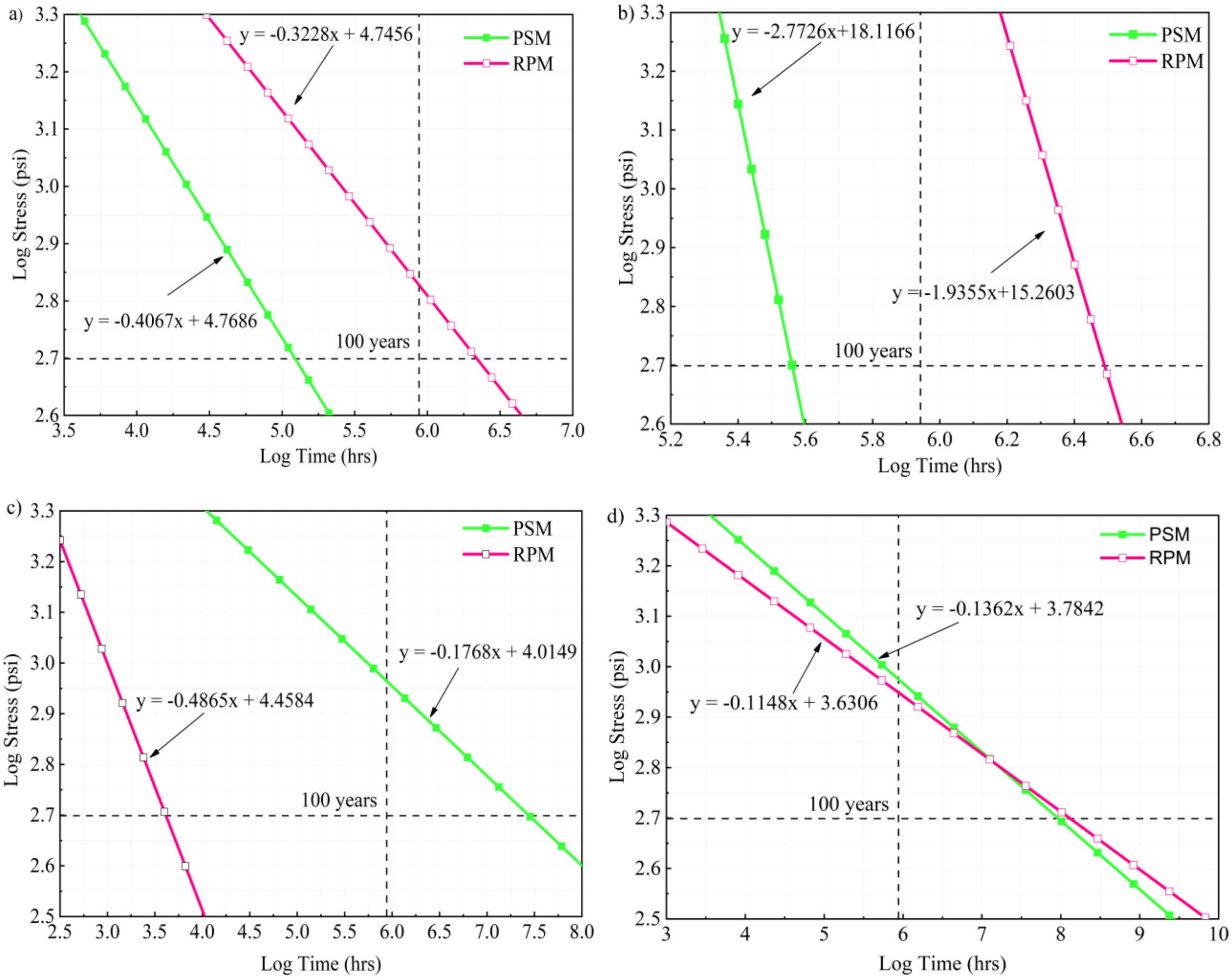


Figure 6.4 SCR data shifted to 10°C and 500 psi with PSM and RPM a) A-R, b) A-V, c) C-V, d) D-R.

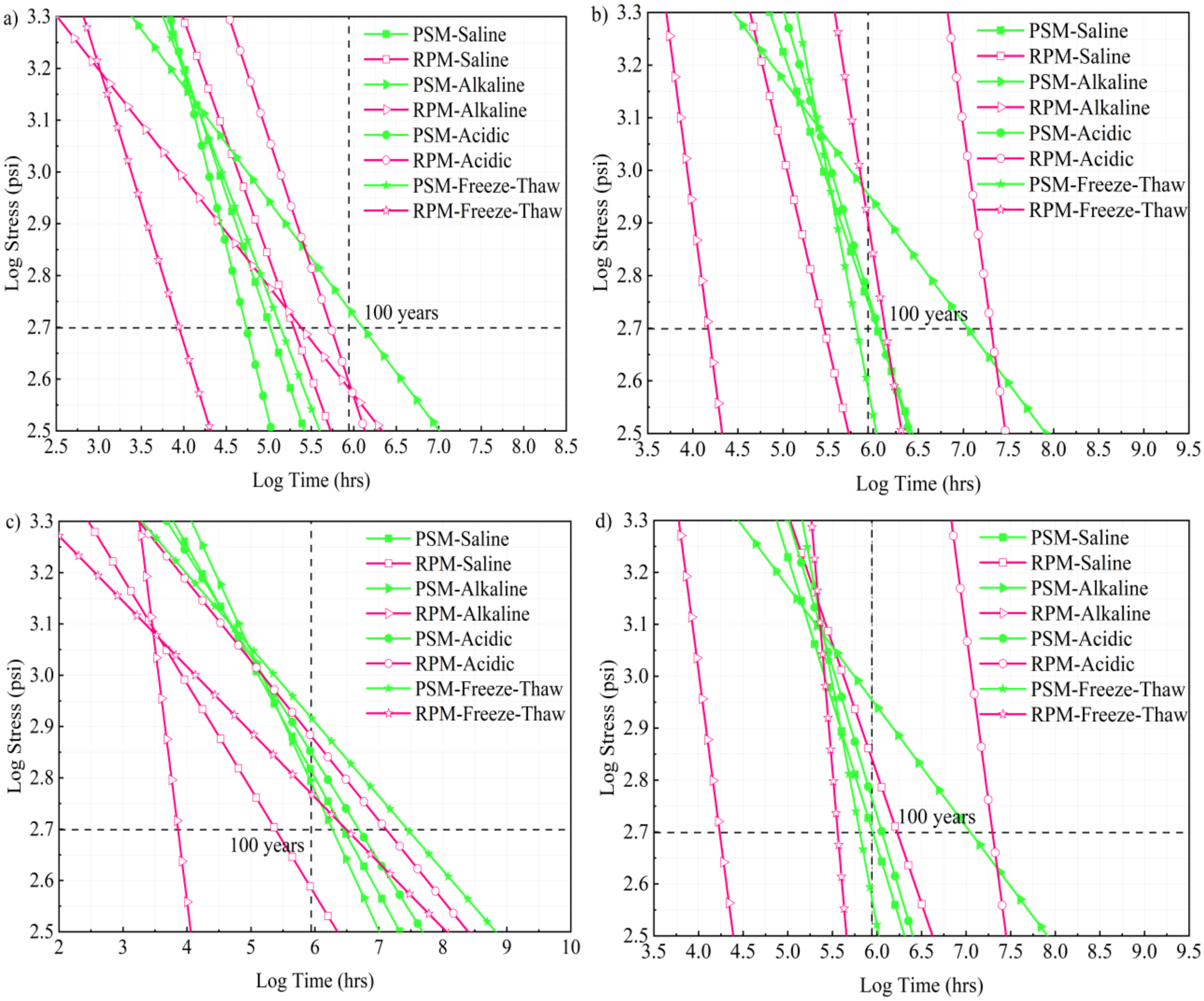


Figure 6.5 SCR data shifted to 10°C and 500 psi with PSM and RPM after immersion in the various solutions a) A-R, b) A-V, c) C-V, and d) D-R.

Table 6.12 presents a comparison of PSM and RPM in predicting pipe service life. It shows that RPM was more reliable than PSM. Since a multivariable least-squares linear regression was used with 97.5% lower confidence in the RPM method, the data points are linear (R-squared equals 1) for all pipes. In contrast, with the use of horizontal and vertical shift factors in PSM, the data points are almost scattered and nonlinear (R-squared less than 0.5). Furthermore, it should be concluded that recycled pipes can guarantee 100 years of service life just like virgin pipes under service conditions of 10^0C and 500 psi.

Table 6.12 Predicted failure times at service conditions of 10°C and 500 psi for both PSM and RPM shifting.

Sample	Slope		Y-intercept		R-squared		Predicted Time to Failure at 500 psi and 10°C (years)	
	PSM	RPM	PSM	RPM	PSM	RPM	PSM	RPM
Nonconditioned								
A-R	-0.4067	-0.3228	4.7686	4.7456	0.99	1	14	250
A-V	-2.7726	-1.9355	18.1166	15.2603	0.67	1	42	353
C-V	-0.1768	-0.4865	4.0149	4.4584	0.39	1	3166	4135 (h)
D-R	-0.1362	-0.1148	3.7842	3.6306	0.98	1	10603	14885
Conditioned								
A-R								
Saline	-0.4960	-0.4596	5.1882	5.1325	0.95	1	12	23
Alkaline	-0.221	-0.2092	4.0490	3.8250	0.88	1	146	28
Acidic	-0.6705	-0.4994	5.8749	5.5624	0.98	1	6	62
Freeze-thaw	-0.4337	-0.5347	4.9288	4.8085	0.77	1	16	1
A-V								
Saline	-0.5054	-0.7309	5.7527	6.6871	0.62	1	126	33
Alkaline	-0.2309	-1.2912	4.3267	8.0848	0.23	1	1279	2
Acidic	-0.5743	-1.2418	6.1774	11.7740	0.28	1	130	2320
Freeze–thaw	-0.9050	-1.0758	7.9638	9.2955	0.84	1	75	155
C-V								
Saline	-0.2258	-0.2054	4.1550	3.8050	0.93	1	320	28
Alkaline	-0.2723	-0.9907	4.4086	6.5228	0.33	1	217	1
Acidic	-0.2001	-0.1554	4.0379	3.8046	0.95	1	561	1502
Freeze–thaw	-0.1444	-0.1273	3.7754	3.5262	0.85	1	3251	360
D-R								
Saline	-0.5574	-0.5005	6.0167	5.8156	0.95	1	102	193
Alkaline	-0.2309	-1.3093	4.3267	8.2468	0.23	1	1279	2
Acidic	-0.5743	-1.3018	6.1774	12.1973	0.28	1	130	2257
Freeze–thaw	-0.9320	-2.0387	8.1119	14.0345	0.44	1	75	41

6.4.5 HDPE pipes in field service in Quebec under natural aging

The dimensions of specimens and the procedure were similar to those of the SCR testing of the six pipes (A-R, A-V, B-R, B-V, C-V, and D-R). Table 6.13 provides the results for the five pipes collected in the field. The results show that the time to failure is varied between pipes. As studied earlier, the variation in the SCR test results was also reported for specimens from the same manufacturer. The coefficient of variation was found to be 20% for P2 specimens. It should be noted that the time to failure of specimens in field service varies considerably, which can be explained, as already indicated, by the service life at the time of testing, resin properties, the manufacturing process, and the environmental conditions to which the pipes were exposed.

Table 6.13 Time to failure of five pipes in field service in Quebec.

Sample	Condition	Average failure time (h)	SD (h)	COV (%)
P1	450 psi/80°C	187.25	21.85	11.67
	650 psi/80°C	76.50	1.70	2.22
	650 psi/70°C	412.80	28.14	6.82
P2	450 psi/80°C	65.50	3.96	6.05
	650 psi/80°C	2.05	0.21	10.35
	650 psi/70°C	6.10	1.27	20.87
P3	450 psi/80°C	194.80	9.33	4.79
	650 psi/80°C	11.70	0.28	2.42
	650 psi/70°C	36.40	1.56	4.27
P4	450 psi/80°C	-	N/A	N/A
	650 psi/80°C	> 1684	N/A	N/A
	650 psi/70°C	-	N/A	N/A
P5	450 psi/80°C	-	N/A	N/A
	650 psi/80°C	> 1684	N/A	N/A
	650 psi/70°C	-	N/A	N/A

Note: N/A = not available; SD = standard deviation; COV = coefficient of variation.

For five pipes in field service, both methods PSM and RPM were also used to estimate the remaining life after a period of use. With PSM, the shift factors are used to shift the data points at the elevated temperature to service conditions at 10^0C and 500 psi, as shown in Table 6.14. With RPM, the three constants—A, B, and C—were calculated to predict the failure time with 97.5% lower confidence at 10°C and 500 psi, as indicated in Table 6.15. Figures 6.6 (a, b, and c) present the brittle curve slopes as a function of the stress and failure time at 10^0C. It should be noted that the estimated life was only determined for pipes P1, P2, and P3 (Table 6.16) because there was not enough data for the others to make a reliable prediction. Again, the data points are linear (R-squared equals 1) for all pipes with RPM. In contrast, with the use of horizontal and vertical shift factors in PSM, the data points are almost scattered (R-squared less than 0.90). It can be concluded that the remaining lifetime of most of the specimens would be in excess of 100 years after 5 to 14 years of use. The limitation of the present study, however, is that a correlation between the degradation time accelerated in the laboratory and in real time in situ is not mentioned. The initial pipe composition, antioxidants, and specific service conditions are not available from the manufacturers.

Table 6.14 Average failure test data of five pipes in field service in Quebec shifted to 10°C and 500 psi with PSM.

Sample	Condition	Avg. Failure Time (h)	Shifted Stress (psi)	Shifted Time (h)	Shifted Stress – Log (psi)	Shifted Time – Log (h)
P1	450 psi/80°C	187.25	1013.584	385557.116	3.006	5.586
	650 psi/80°C	76.50	1464.065	157517.327	3.166	5.197
	650 psi/70°C	412.80	1303.714	285775.899	3.115	5.456
P2	450 psi/80°C	65.50	1013.584	134867.776	3.006	5.130
	650 psi/80°C	2.05	1464.065	4221.053	3.166	3.625
	650 psi/70°C	6.10	1303.714	4222.948	3.115	3.626
P3	450 psi/80°C	194.80	1013.584	401102.944	3.006	5.603
	650 psi/80°C	11.70	1464.065	24090.885	3.166	4.382
	650 psi/70°C	36.40	1303.714	25199.231	3.115	4.401

Table 6.15 RPM coefficients for five pipes in field service in Quebec.

Sample	Coefficient	Value
P1	A	-23.227
	B	11281.090
	C	-859.309
P2	A	-15.932
	B	15088.330
	C	-3325.493
P3	A	-15.839
	B	13562.390
	C	-2699.768

Table 6.16 Predicted failure times at service conditions of 10°C and 500 psi for five pipes in field service in Quebec with both PSM and RPM shifting.

Sample	Slope		Y-intercept		R-squared		Predicted Time to Failure at 500 psi and 10°C (years)	
	PSM	RPM	PSM	RPM	PSM	RPM	PSM	RPM
P1	-0.3802	-0.3293	5.1536	5.4787	0.8493	1	326	31515
P2	-0.0894	-0.0851	3.4646	3.1814	0.9049	1	41840	53
P3	-0.115	-0.1048	3.6302	3.3632	0.9128	1	25665	249

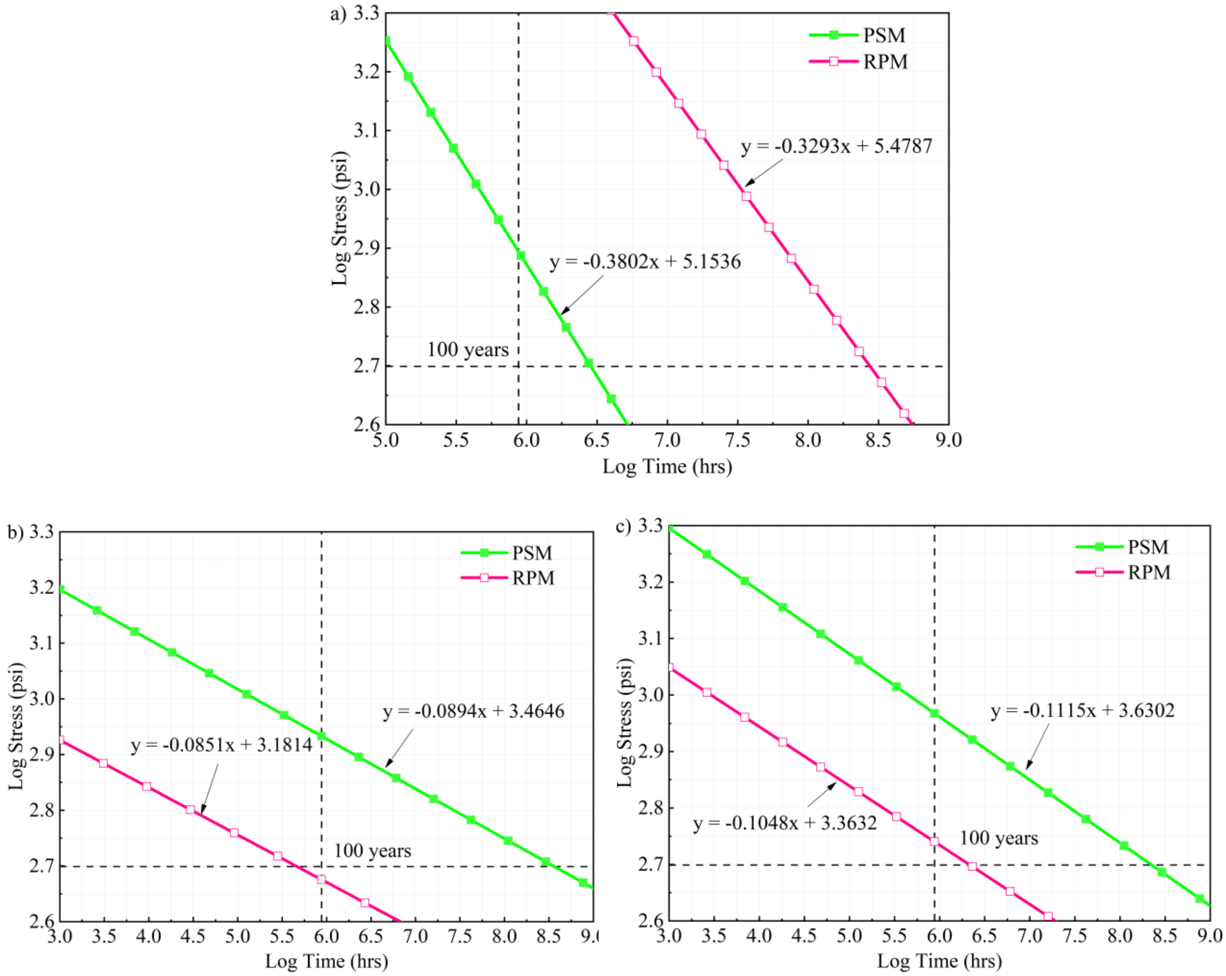


Figure 6.6 SCR data shifted to 10°C and 500 psi for five pipes in field service in Quebec with PSM and RPM a) P1, b) P2, c) P3.

6.5 Discussion

The stress crack resistance (SCR) of corrugated HDPE pipes was investigated. SCR specimens taken from the 6 new corrugated HDPE pipe liners (A-R, A-V, B-R, B-V, C-V, and D-R) were examined before and after immersion in saline, acidic, or alkaline solutions at 60°C, and freeze–thaw cycles (from -40°C to +40°C) for 12 months. In addition, SCR specimens from 5 corrugated HDPE pipes in field service in Quebec (P1, P2, P3, P4, and P5) for 5 to 14 years were investigated to estimate the service life of pipes. The failure time of the pipe-liner specimens was determined with the Florida Test Method (FM 5-573) in water at 450 psi and 80°C, at 650 psi and 80°C, and at 650 psi and 70°C. Two extrapolation methods—Popelar's Shift Method (PSM) and the Rate Process Method (RPM)—were used to estimate the service life of HDPE pipes at service conditions of 10°C and 500 psi.

New Corrugated HDPE Pipes

The time to failure of pipes made with recycled resin was faster than those made with virgin resin (manufacturers A and B) before and after immersion in the different solutions. This can be explained by the recycled pipe containing more carbon black than the virgin pipe (Table 3). The addition of carbon black reduced the SCR of the pipes. In addition, the recycling process also significantly affects the properties of recycled pipe (e.g., presence of impurities or other polymers). Moreover, all B-R and B-V specimens experienced plastic deformation. It should be noted that B-R was regrind pipe manufactured from "reworked" materials of products damaged during the manufacturing process. Therefore, carbon black and regrind materials affected the SCR of pipes. That might be due to the applied stresses being greater than those producing brittle fractures. Furthermore, specimens B-R and B-V had low tensile strength (Table 3), which could explain this behavior. Variations in the SCR test results for pipes from the same or different manufacturers were observed before and after immersion in various solutions. This can be taken into account when machining notches. In the four test environments, the saline solution resulted in the greatest acceleration of the pipe SCR.

Before immersion in various solutions, the recycled and virgin pipes could have achieved a 100-year service life under service conditions of 10°C and 500 psi (3.45 MPa). This value would be lower after immersion. This could be related to the two steps of elevated temperature in the conditioned test (60°C) and the Florida test (70°C and 80°C), along with the harsh environment of the conditioned test. A testing temperature of 80°C might result in the melting of some of the poorly formed crystallites. It should be noted that the laboratory aging conditions were harsher than the actual field conditions. Moreover, all sides of the specimens were in direct contact with the solutions in the laboratory. The RPM method could be more reliable than the PSM in predicting a pipe service life of 100 years. PSM yielded data points that were almost scattered and nonlinear.

Corrugated HDPE Pipes in Field Service in Quebec under Natural Aging

Variations in SCR test results was also noted for specimens from the same or different manufacturers. As mentioned earlier, the time to failure of the specimens in field service varied considerably. That can be explained by the service life at the time of testing, resin properties, manufacturing process, and the environmental conditions to which the pipes were exposed. It can, however, be concluded that the remaining lifetime of most of the specimens would exceed 100 years after 5 to 14 years of use. It should be kept in mind that the data points were almost scattered and nonlinear with PSM. Based on the preliminary results of the current study, RPM could be a reasonable method for predicting pipe service life. Supplemental testing and other studies should be conducted to optimize the extrapolation of this approach in more accurately predicting the lifetime of corrugated HDPE pipes.

6.6 Conclusions

In order to assess the 100-year service life of corrugated recycled and virgin HDPE pipes used in road drainage systems, the SCR was investigated with the Florida Test Method. The SCR tests were performed on pipe liners in water at 450 psi and 80°C, at 650 psi and 80°C, and at 650 psi and 70°C in compliance with FM 5-573 [25]. The SCR of new pipes before and after immersion in various solutions (saline, alkaline, and acidic) and freeze–thaw cycles for 12 months at 60°C was investigated. The remaining lifetime of in-service pipes in the field under natural after 5 to 14 years of use was also calculated. The data points at elevated temperatures were then shifted to predict the service life of pipes under lower service conditions using PSM and RPM, both extrapolation methods. Based on the results of this study, the following conclusions can be made.

1. Before immersion in the various solutions, the recycled pipes could achieve a 100-year service life just like virgin pipes under service conditions of 10°C and 500 psi.
2. After immersion in the various solutions, the service life of the recycled and virgin pipes was less than 100 years. The decrease in SCR is related to the two steps of elevated temperature in the conditioned test and the Florida test, along with the harsh environment of the conditioned test. The time to failure of pipes decreased faster in the saline solution rather than other solutions. It should be noted that laboratory aging conditions are harsher than actual field conditions. In addition, all sides of the specimens are in direct contact with the solutions in the laboratory.
3. RPM could be a reasonable alternative to PSM in predicting 100 years of pipe service life. The data points were almost scattered and nonlinear with PSM. The increase in the SCR after immersion resulted from scattered data and a large deviation between SCR measurements.
4. The pipes exposed under in-service field and natural aging conditions in Quebec were examined to estimate their remaining life. The results show that the remaining life of the investigated pipes installed in the field would be in excess of 100 years after 5 to 14 years of use.
5. The findings of this study will further promote the use of recycled HDPE materials in the future. The long-term performance along with the 100-year service life of recycled pipes holds promise for replacing virgin pipes for road drainage systems. Furthermore, future studies should also consider using recycled HDPE materials for various applications. They can be used for floating solar applications where working conditions are not too harsh and non-pressure as road drainage applications.

Data Availability Statement

All data, models, and code supporting the findings of this study are available from the corresponding author upon reasonable request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This research received financial support from the Natural Science and Engineering Research Council of Canada (NSERC), the NSERC Research Chair in Innovative FRP Reinforcement for

Sustainable Concrete Infrastructure, the Tier-1 Canada Research Chair in Composite Materials for Civil structures, the Fonds Québécois de la recherche sur la nature et les technologies (FQRNT), the Ministry of Transportation of Quebec (MTQ), and the University of Sherbrooke Research Centre on Composite Materials (CRUSMaC). The donation of HDPE pipes by Soleno Inc. (Saint-Jean-sur-Richelieu, QC, Canada), Armtec Inc. (Saint-Augustin-de-Desmaures, QC, Canada), ADS Canada (Saint-Germain-de-Grantham, QC, Canada) and Infra Pipe Solutions Ltd (Mississauga, ON, Canada) to support this investigation are greatly appreciated. The authors are also grateful to the technical staff of the structural laboratory at the University of Sherbrooke, especially Jérôme Lacroix and Steven MacEachern, for their technical assistance. In addition, the authors are grateful to Mario Parades and TRI/Environmental, Inc. (TRI, Texas, USA) for their valuable advice and performing SCR tests.

7 CHAPITRE 7: EFFETS DES CONDITIONS D'EXPOSITION SUR L'ÉPUISEMENT DES ANTIOXYDANTS, LA RÉSISTANCE À LA TRACTION ET LE MODULE À LONG TERME DES TUYAUX ONDULÉS EN PEHD FABRIQUÉS AVEC ET SANS RÉSINES RECYCLÉES

Avant-propos

Auteurs et affiliation:

Khanh Q. Nguyen: Étudiant au doctorat, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Patrice Cousin: Chercheur associé, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Khaled Mohamed: Stagiaire postdoctoral, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Mathieu Robert: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Brahim Benmokrane: Professeur, Département de génie civil et de génie du bâtiment, Faculté de génie, Université de Sherbrooke, 2500, boul. de l'Université, Sherbrooke (Québec) J1K 2R1, Canada.

Date de soumission: 19 janvier 2022

Titre du journal: Journal of Polymers and the Environment

Titre en anglais: Effects of Exposure Conditions on Antioxidant Depletion, Tensile Strength, and Long-term Modulus of Corrugated HDPE pipes made With or Without Recycled Resins.

Résumé en français

La durée de vie des tuyaux ondulés en polyéthylène à haute densité (PEHD) utilisés dans les applications d'infrastructure de transport a fait l'objet de plusieurs études au fil des ans.

Cependant, les facteurs spécifiques aux conditions climatiques nord-américaines tels que des cycles de gel-dégel, des sels de déglaçage et de l'abrasion sur la durée de vie des canalisations n'ont pas été abordés. Des essais de vieillissement accéléré en laboratoire ont été menés pour examiner la dégradation thermo-oxydante ainsi que la résistance à la traction et le module à long terme des tuyaux en PEHD fabriqués avec et sans résines recyclées. Les échantillons ont été immergés dans la solution saline (NaCl), la solution acide (H_2SO_4), la solution alcaline (NaOH) à 60^0C et dans de l'eau pour des cycles de gel-dégel de -40^0C à $+40^0$C. En mesurant le temps d'induction oxydative (OIT) à divers intervalles de temps, qui quantifie les quantités d'antioxydants présents dans les tuyaux, on constate que le taux d'épuisement des antioxydants dépend du milieu environnant. Le taux d'épuisement est le plus rapide dans la solution saline et le plus lent dans les cycles de gel-dégel. Pour les spécimens en gel-dégel, l'épuisement est environ 1,12 à 1,91 inférieur à celui de l'acide, 1,25 à 2,64 fois inférieur à celui de l'alcaline et 1,43 à 3,25 fois inférieur à celui de la solution saline. Cependant, ces tuyaux en PEHD satisfont tous aux exigences minimales de 20 minutes de l'OIT. De plus, l'analyse de la variance à un facteur (ANOVA) et les méthodes statistiques HSD de Tukey indiquent que la résistance à la traction et les valeurs de module à long terme des tuyaux en PEHD recyclés et vierges sont inchangées avant et après immersion dans différentes solutions.

Mots-clés: Tuyau ondulé en polyéthylène haute densité (PEHD); résines recyclées; conditions d'exposition; temps d'induction oxydative (OIT); l'épuisement des antioxydants, la résistance à la traction et le module à long terme ; ANOVA et HSD de Tukey analyses.

Abstract

The service lifetime of corrugated high-density polyethylene (HDPE) pipes used in transportation infrastructure applications has been the subject of several studies over the years. How factors specific to North American climate conditions—such as freeze/thaw cycles, deicing salts, and abrasion—affect pipe service life have not been addressed. In this study, laboratory accelerated-aging tests were conducted to examine the thermo-oxidative degradation as well as tensile strength and long-term modulus of HDPE pipes made with or without recycled resins. Specimens were immersed in saline solution (NaCl), acidic solution (H_2SO_4), alkaline solution (NaOH) at 60°C, or in water and exposed to freeze/thaw cycles ranging from -40°C to +40°C. Measuring the oxidative induction time (OIT) at various time intervals quantified the amounts of antioxidants present in the pipes, revealing that the rate of antioxidant depletion depended on the surrounding environment. The depletion rate was faster in the saline solution and slower in the freeze/thaw cycles. For specimens exposed to freeze/thaw cycles, the depletion was 1.12 to 1.91 lower than in the acidic solution, 1.25 to 2.64 times lower than in the alkaline solution, and 1.43 to 3.25 times lower than in saline solution. Nevertheless, the HDPE pipes all met the OIT requirements of a minimum of 20 minutes. Furthermore, the one-way analysis of variance (ANOVA) and Tukey's HSD test indicate that the tensile strength and long-term modulus values of the recycled and virgin HDPE pipes were unchanged before and after immersion in the different solutions.

Keywords: Corrugated high-density polyethylene (HDPE) pipe; recycled resins; exposure conditions; oxidative induction time (OIT); antioxidant depletion, tensile strength, and long-term modulus; ANOVA and Tukey's HSD tests.

7.1 Introduction

Corrugated high-density polyethylene (HDPE) pipes used for transportation infrastructure applications are usually buried underground. Many studies [177-180] have shown that the properties of polymer materials can change overtime and due to exposure conditions (e.g., sunlight, temperature, thermal cycles, and oxygen …). Understanding long-term performance is important in estimating a pipe's lifetime. In general, the long-term service lifetime of HDPE follows three aging phases [8,181]. In the first phase (I), the pipes mainly fail in a ductile manner under significant loads (normally >30% of yield). In the second phase (II), pipes are subjected to intermediate loads, which initiates microcracks at the junction of the liner and corrugated part, leading to brittle failures. Lastly, in the third phase (III), failures are generally brittle under lower stresses when the antioxidants have been consumed and the pipe undergoes oxidation.

Three main factors control the lifetime of a plastic pipe: material (resins, additives), environment (chemical agents, UV radiation, microbiological effects), and loading (static and dynamic loading) [182]. In stark contrast to pressure pipes, it can be assumed that stress cracking is not a major problem for buried pipes. They could be in a state of relaxing bending stress under an occasional load or a dynamic load of vehicles [183]. In fact, the choice of resin type, additives, or manufacturing process can be planned early on. In contrast, environmental factors are a major issue with buried pipes. The water in culvert and sewer systems contains heavy materials, synthetic chemicals, petroleum, or gas. Moreover, the surrounding soil might contain significant amounts of transition metal and moisture that cause the degradation of the pipes [181]. Oxidation occurs once the antioxidant (AO) content has been exhausted. Oxidation can decrease of the molecular weight of the polymer due to chain scission, making the pipe brittle over time [184]. Therefore, oxidative degradation is an important issue affecting pipe degradation. Antioxidants (AOs) and stabilizers incorporated into HDPE pipes retard or halt the oxidation process [174]. Primary and secondary antioxidants are two common types for antioxidant packages [185]. A primary antioxidant's function is to act as free radical (ROO•, RO•, OH•) scavengers and form ROOH, ROH, and H_2O. Secondary antioxidants convert carboxylic acids (ROOH) to inactive alcohol (ROH) [8,186]. In general, the oxidative degradation process, which can be evaluated by measuring OIT values, occurs in three stages, as shown in Fig. 7.1 [8,53,187]. The total loss of antioxidants takes place in stage t_1. Stage t_2 is the induction time to the onset of material degradation. Stage t_3 is the process of material degradation with a reduction of properties. The time taken for a material property to drop to a specified value (typically 50% of its initial value) is defined as the material's lifetime(t_{life}). For HDPE pipes, this is a combined duration of the three stages ($t_1 + t_2 + t_3$).

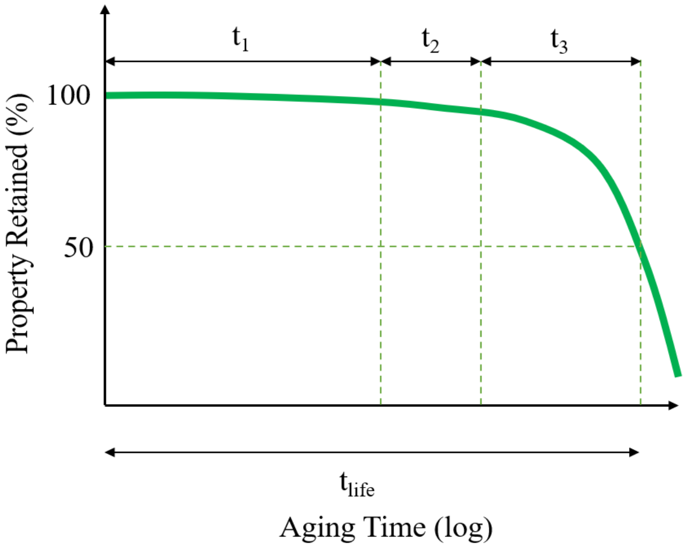


Figure 7.1 Three stages conceptual model of chemical aging of HDPE (adopted from Hsuan and Koerne [8])

Other studies have evaluated the antioxidant depletion and mechanical properties of pipes with accelerated aging tests to simulate the long-term exposure and estimate pipe durability. Gedde et al. [188] studied the long-term behavior of polyolefin pipes used in hot water environments. Thomas [46] studied the long-term performance of corrugated pipe manufactured with recycled polyethylene content. Most of the work done in the past focused on HDPE water pipes. Hoang and Lowe [158] studied the depletion of antioxidants and initiation of thermo-oxidative degradation of a PE100 blue water pipe. They had been exposed to a hydrostatic pressure at 20^0C, 40^0C, 60^0C, or 80^0C. The results indicated that the antioxidant content decreased by 95% after 2500 h of immersion at 80^0C. Whelton et al. [189] evaluated the impact of the aging of HDPE water pipes by exposing them to chlorinated water. The results demonstrated that after 141 days, the OIT value was reduced by 69% during the first 90 days and remained relatively unchanged afterwards. They also reported unchanged tensile strength. In addition, Hsuan and Wong [187] evaluated the oxidation degradation of corrugated HDPE pipe. The tested specimens contained 2.5wt% of carbon black (CB) and an antioxidant package that consisted of 0.05% Irganox 1010 and 0.1% Irgafox 168. Specimens were incubated in a forced-air oven at temperature of 65^0C, 75^0C, and 85^0C and in a water bath at 85^0C. The results also indicated that the OIT value decreased with incubation time. Krushelnitzky's work pointed out that the antioxidant depletion rate of HDPE pipes was faster when the pipes were immersed in a synthetic municipal solid-waste leachate rather than in the air [183]. Recent works have pointed out that antioxidant depletion rates were faster for HDPE geomembranes exposed to leachate than that exposed in water or air [174,175]. Jeon's work also indicated that the antioxidant depletion time of HDPE geomembranes was longer in an acidic solution and shorter in an alkaline solution [190]. Furthermore, the effects of different exposure conditions on the mechanical properties (tensile strength, long-term modulus) have also been investigated. Rowe and Rimal [191] reported that tensile at yield and break was almost unchanged after 35 months of incubation in synthetic leachates at 85^0C for an HDPE geomembrane in a composite liner. Rowe et al [192,193] had similar results for the unchanged tensile strength of an HDPE geomembrane under landfill conditions. HDPE geomembranes, however, are different than HDPE pipes because of their constituents and conditions of use. The literature contains little about the effect of exposure conditions on antioxidant depletion, tensile strength, and long-term modulus of corrugated HDPE pipe for transportation infrastructure applications. In contrast, no research has evaluated the antioxidant depletion rate, or the mechanical properties of corrugated HDPE pipes made with virgin and recycled resins exposed to North American climate conditions.

The exposure conditions for HDPE pipe in past studies do not reflect the actual conditions in North American, including their durability characteristics in freeze-thaw cycles, de-icing salts, and abrasion conditions. In our study, the failure of corrugated HDPE pipe in phase III (oxidation) was assessed by determining the level of antioxidants necessary to prevent the chemical attack due to the ageing conditions (freeze-thaw cycles, saline, acidic, and alkaline solutions). The study aimed at comparing of the antioxidant depletion rate between these different aging durations. Moreover, the effects of different solutions on the tensile strength and long-term modulus of recycled and virgin HDPE pipes were identified.

7.2 Materials and Methods

7.2.1 Materials

The material used had been described elsewhere [173]. It consists of six corrugated HDPE pipes, two (A-R, D-R) manufactured with post-consumer recycled resins (household waste), one (B-R) with post-industrial recycled (pipe's scrap), and three with virgin resins (A-V, B-V, C-V) from four different North American manufacturers (Fig. 3.1). These pipes, used for transportation infrastructure applications (e.g., storm drainage and storm sewers), were manufactured according to BNQ 3624-120, Class A [19]. Their density was greater than 0.945 g/cm^3 [29] and they were stabilized with 2-4 % carbon black and antioxidants. The letters A, B, C, and D designate the manufacturers, R stands for recycled resin, and V for virgin resin. Each pipe had an external diameter of 900 mm and length of 3000 mm. As shown in Table 4.1, the detailed properties and parameters used for this study were summarized in a past study [173].

7.2.2 Laboratory-accelerated Ageing Tests

Polyethylene (PE) is well-known for its low chemical reactivity with polar fluids and solvents—such as water, acids, and bases— due to its hydrophobicity and chemical structure [4,194]. HDPE pipes are therefore resistant to chemical corrosion [34]. The available literature related to short-term applications does not address road infrastructure application. In addition, current environments—including exposure to detergents, leachate, hot water, chlorinated water—do not simulate Northern conditions. Our study focused on the impact of Canada's winter (e.g., low temperature, variations in daytime and nighttime temperatures, and freeze-thaw cycles) on antioxidant depletion, tensile strength, and long-term modulus. Historical weather records for Quebec indicate that the average highest and lowest temperatures annually are around 40^{0}C and -40^{0}C, respectively. Weather characteristics with long, cold winters, and short summers, as well as the impact of sunlight at certain times (installation, repair, etc), are among the factors that affect the long-term performance of pipelines. To simulate the Northern climate, specimens taken from the pipe liner (Fig. 4.1) were conditioned in different chemical solutions. Specimen consisting of 40 x 10 x 4 mm^3 plates were used to investigate antioxidant depletion and long-term modulus. Dogbone specimens were used to evaluate the tensile strength. The following sections provide details on the dimension of test specimens. Four exposure conditions were applied: freeze-thaw cycles, and exposure to saline, acidic, or alkaline solutions. It is well known that freeze-thaw cycles are one of the problems facing transportation infrastructures in North America. These conditions could cause internal cracks by inducing additional internal stresses (shrinkage and expansion). To simulate freeze-thaw cycles, specimens were immersed in water for 625 cycles at temperatures between -40^0C and 40^0C. Each cycle consisted of 8 h of freezing and 6 h of thawing. To simulate deicing-salt, acidic and alkaline service conditions, specimens were immersed in either a saline solution (NaCl 1M), sulfuric acid (H_2SO_4, pH 2), or sodium hydroxide (NaOH, pH 10), respectively. The immersion tests were performed at 60^0C. It should be noted that acceleration tests are valid when only the temperature affected the rate of degradation [108,116,174,175]. The selected temperature for accelerated aging tests is not so high that it modifies the nature of the material and consumes the antioxidants. Hence, it is recommended that the upper-temperature limit for laboratory-accelerated aging tests of HDPE pipes not exceed the softening point at 60°C. Specimens were retrieved at various time intervals, including 4, 8, and 12 months. The oxidative induction time was monitored to evaluate the rate of antioxidant depletion. Tensile properties were also investigated. Due to the availability of the conditioning chamber, specimens for long-term modulus were investigated after 6.5 months.

7.2.3 Oxidative Induction Time

Oxidative induction time is a standardized test to determine the amount of AO in the pipes. This test was conducted with differential scanning calorimetry (DSC 6000 from Perkin-Elmer) in accordance with ASTM D3895-19 [28]. Specimens weighing 5-10 mg were heated under nitrogen from room temperature to 200^0C at 30^0C/min. Once the higher temperature was reached, oxygen was introduced and the value of OIT was taken as the onset of the exothermic peak appearing when the sample oxidized. Fig. 4.3 provides a typical evaluation of OIT from the recorded time-based thermal curve.

7.2.4 Tensile Tests

The tensile properties of HDPE pipes before and after immersion in the different solutions were investigated according to ASTM D638 [20] with type IV dog-bone specimens. The specimens were punched directly from the pipe liner using a stainless-steel die. The specimen geometry and dimension are described in ASTM D638 as illustrated in Fig. 4.4. The thickness of test specimens cut from pipes A-R, A-V, B-R, B-V, C-V, and D-R was 4.80, 4.40, 7.50, 7.60, 2.70, and 3.80 mm, respectively. The test was performed at 23^0C on an MTS (Material Testing Systems) universal testing machine with a constant crosshead speed at 50 mm/min. The specimen extension was measured with a 634.12F-24 extensometer. At least four replicates were performed to evaluate the tensile strength before and after immersion.

7.2.5 Time-temperature superposition (TTS)/DMA Tests

Time-temperature superposition (TTS) is the acceleration test most often used to investigate the viscoelastic behavior and the long-term modulus with elevated temperature. The relaxation curves obtained at high frequency and temperature steps can be shifted to generate a master curve at the reference temperature. The master curve is then extrapolated to predict the long-term modulus of materials. In the current study, the tests were carried out using a DMA (DMA 8000 from Perkin-Elmer). The specimens—measuring 40 x 10 x 4 mm^3—were tested using the single cantilever mode with a span length of 17.5 mm. The temperature ranged from 5^0 to 100^0C, with 5^0C steps. Frequencies from 0.01Hz to 100Hz were applied. A heating rate of 5^0C/min and an isotherm of 3 minutes for each temperature step were applied. The strain applied was 0.05%. At least three replicates were performed with a test duration of 10 hours.

7.2.6 Statistical Analysis

Variations between the measured values of the specimens before and after have been immersed in solutions are normal. Therefore, a one-way analysis of variance (ANOVA) was used to evaluate whether the differences between measurements were significant. This method indicates no significant difference between measurements (treatments) if the F-value is less than the F-critical value or the P-value is greater than the alpha level (0.05). The F-value is determined as the ratio of the variance of the group means (means square between) to the mean of the group variance (mean squared error). The F-critical is determined by the numerator (df1) and denominator (df2) degrees of freedom with a specific significance level (alpha level) from the F-distribution table [195]. The P-value is the probability value describing how likely it is that the data could have occurred under the null hypothesis (no significant difference between measurements). In contrast, if one or more treatments are significantly different, post-hoc tests are then applied to clarify which pairs of treatments are significantly different. In this study, the Tukey's HSD test was applied as a post-hoc test. The first issue is to determine the critical value of the Tukey's HSD (q-critical) based on the number of treatments and degree of freedom for the error term and for the alpha level (0.05) from the tables of the inverse Studentized Range Distribution [196]. Next, Tukey's HSD q-statistic is calculated by taking the absolute value of the difference between pairs of means and dividing it by the standard error of the mean, as shown in Equations 7.1 and 7.2.

$$q_{i,j} = \frac{|x_i - x_j|}{SE} \tag{7.1}$$

$$SE = \sqrt{\frac{1}{2} MS_W \left(\frac{1}{n_i} + \frac{1}{n_j}\right)} \tag{7.2}$$

where, $q_{i,j}$ is Tukey's HSD q-statistic; $x_i - x_j$ is the difference between pairs of means; SE is the standard error of a group mean; MS_w is the mean square within group; n is the group; and i,j are the columns labeled i and j.

For Tukey's post-hoc test, if the q-statistic value is less than q-critical, there is no significant difference between the pair of treatments and vice versa.

7.3 Results and Discussions

7.3.1 Rates of antioxidant depletion

Although OIT measurements cannot identify the types of antioxidants, the relative concentration of antioxidant retained in the aging specimen can be determined. A higher OIT value indicates a greater amount of AO [197]. In other words, the OIT is proportional to the antioxidant concentration. In our investigation, the AO depletion rate followed a first-order decay model, which can be expressed as Eqs. 7.3 and 7.4 [8,53].

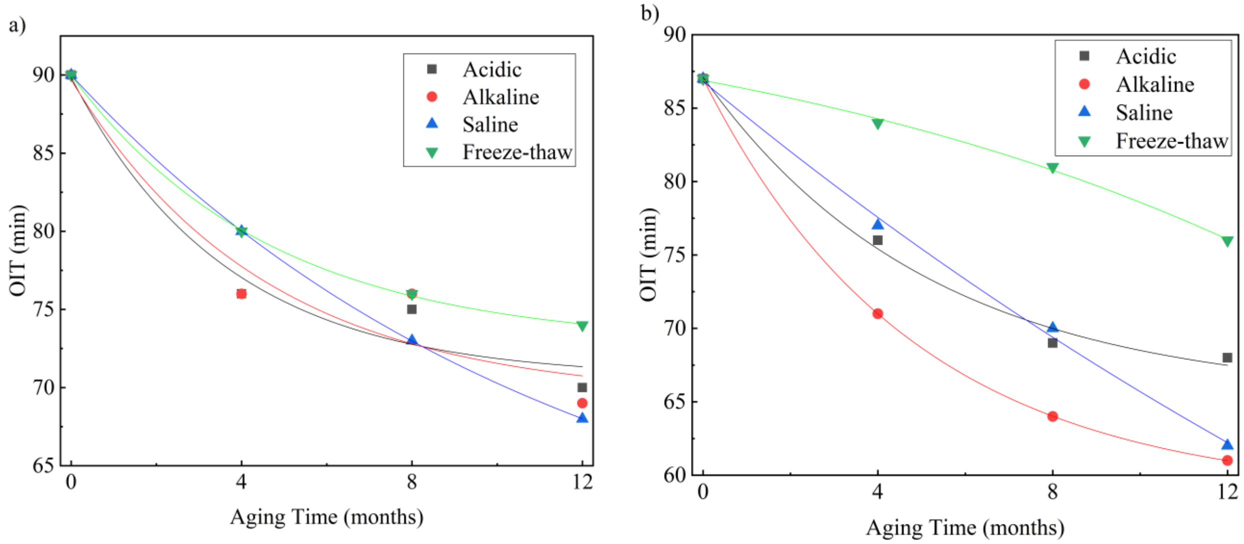
$$\mathrm{OIT}_t = \mathrm{OIT}_0.e^{-st} \tag{7.3}$$

or taking logarithm on both sides,

$$\ln(\mathrm{OIT}_t) = -st + \ln(\mathrm{OIT}_0) \tag{7.4}$$

where, OIT_t is the OIT at time t (in minutes), OIT_0 the initial OIT value (in minutes), s the rate of AO depletion (in month^{-1}), and t the aging time (in months).

Figures 7.2 (a-f) show the OIT value as a function of time and exposure conditions for six corrugated HDPE pipes. The OIT was measured at various time intervals with three replicate specimens for each pipe. The exponential curve fitting was used to fit experimental data. The results indicate that, after 12 months of aging in different solutions, the OIT percent retained was above 55%. Therefore, all the pipes met the OIT minimum of 20 min according to ASTM D3895-19 [28]. Hsuan and Koerner [8] observed that the OIT decreases with time and different exposure conditions. In other words, antioxidant depletion is dependent on chemical reactions and leaching. Reaction with oxygen and oxidizers as well as diffusion/extraction processes cause the consumption of AO [198].



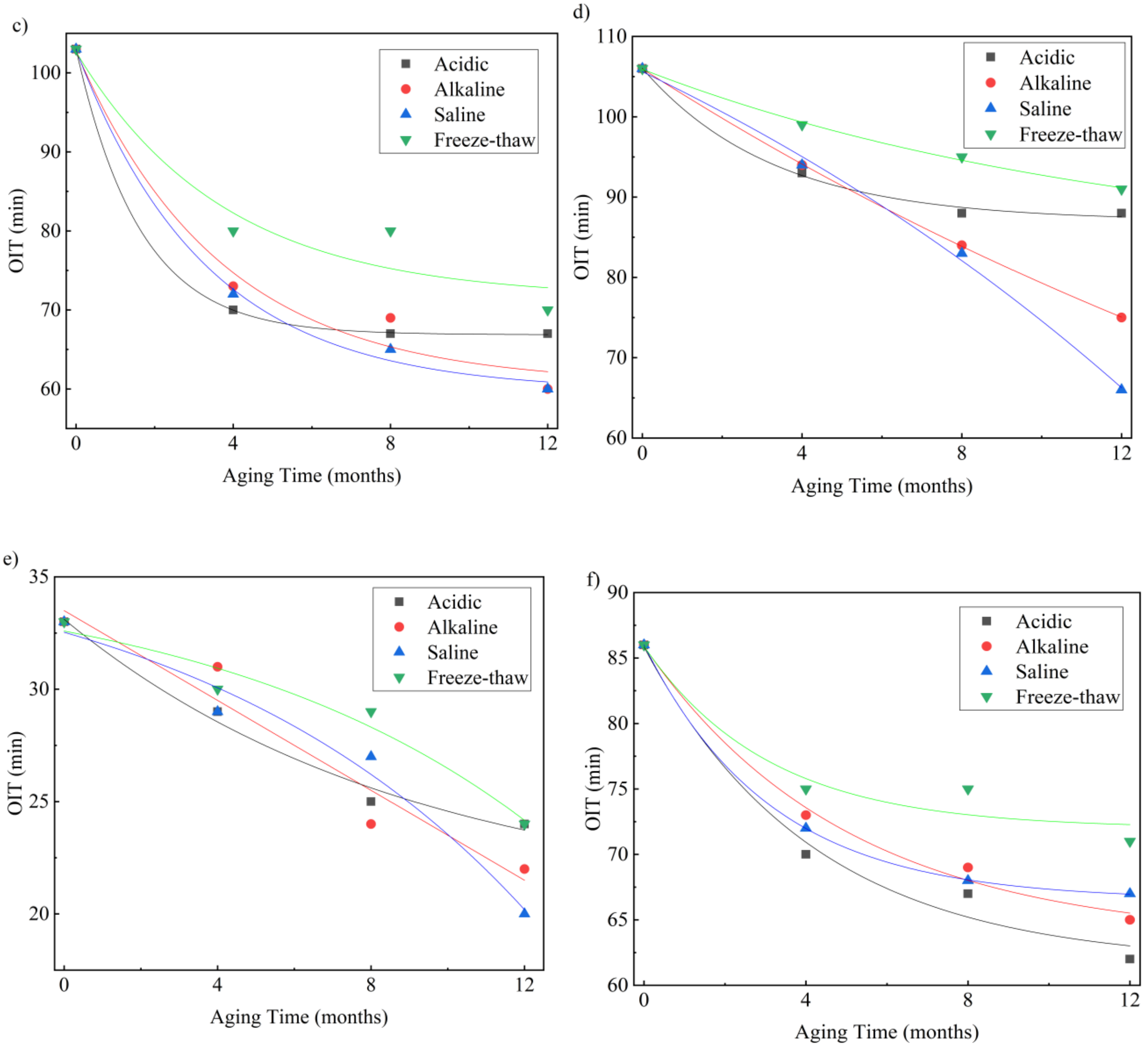
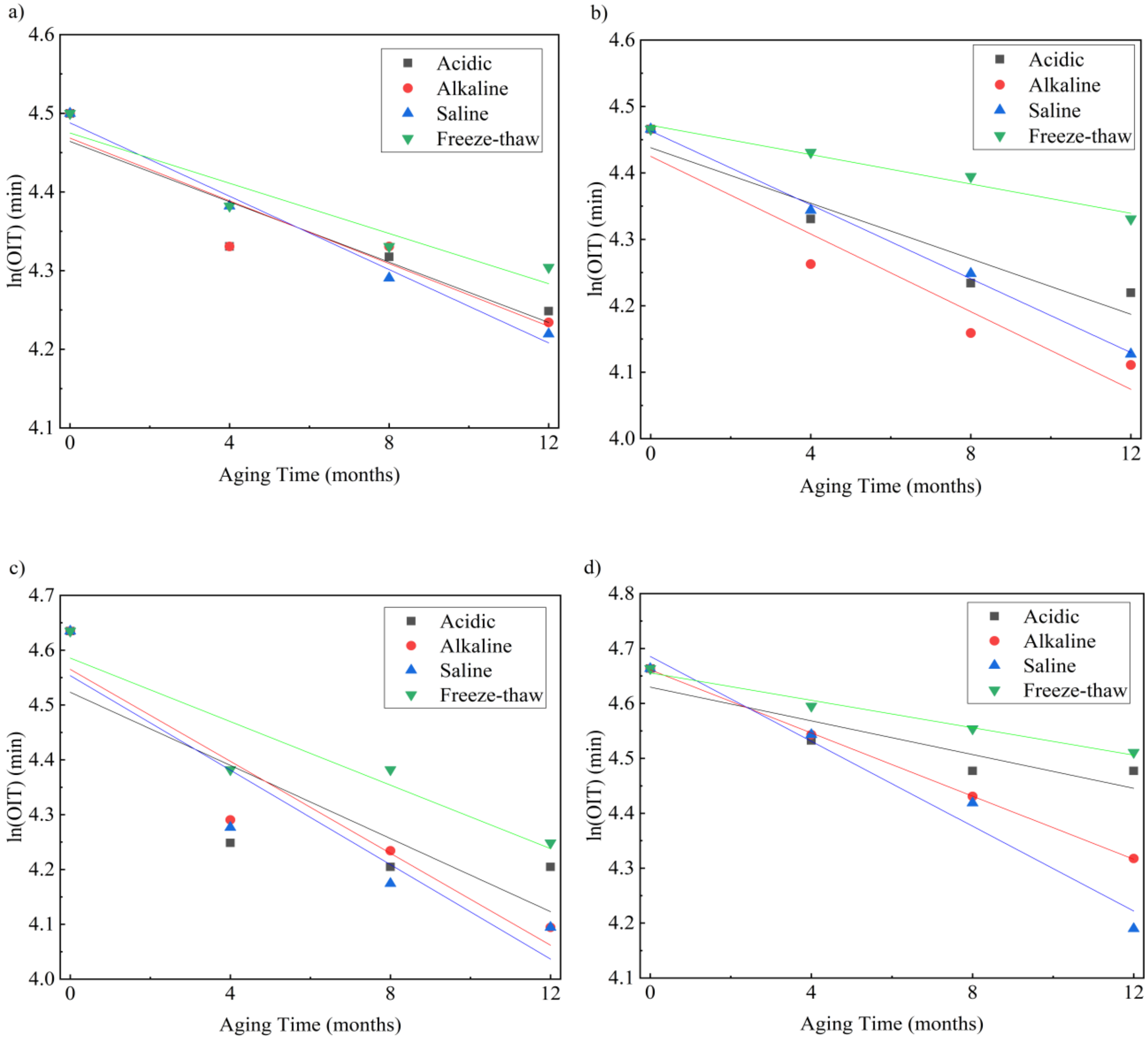


Figure 7.2 OIT value of HDPE pipes with aging time under different exposure conditions a) A-R b) A-V c) B-R d) B-V e) C-V f) D-R

Figures 7.3 (a-f) presents the typical plot of ln(OIT) with aging time for pipes immersed in saline, acidic, and alkaline solutions, and subjected to freeze-thaw cycles. With a linear regression of data (Eq. 7.3), the AO depletion rate corresponds to the slopes of the graph. Table 7.1 presents the rate of antioxidant depletion values determined for these conditions. The results show that the lowest depletion rates are observed with the freeze-thaw cycles, while the highest depletion rates were obtained with the saline solution. A comparison between the AO depletion rate after immersion in the acidic, alkaline, and saline solutions with freeze-thaw cycles indicates that the depletion rate in saline solution varies from 1.43 to 3.25 times the depletion time after freeze-thaw cycles (Table 7.1). It should be highlighted that the initial OIT value is different from a pipe to the next because of the composition of their antioxidant and stabilizer package, which is exclusive and proprietary for each manufacturer. The pipes might have been stabilized with phenolic and phosphates antioxidants, which would probably make them more susceptible to chemical solutions than freeze-thaw cycles. The thickness of the pipe wall could also be a factor influencing the antioxidant depletion [33,183]. In our study, the specimens were all cut from pipe liners with the same thickness (4 mm). Therefore, the pipe-wall thickness factor is not mentioned herein. In addition, both sides of the investigated specimens were in contact with the solutions. Lastly, the AO depletion rates in recycled and virgin pipes cannot be compared because they contain different and unknown antioxidants, which do not react in the same way with their environment.



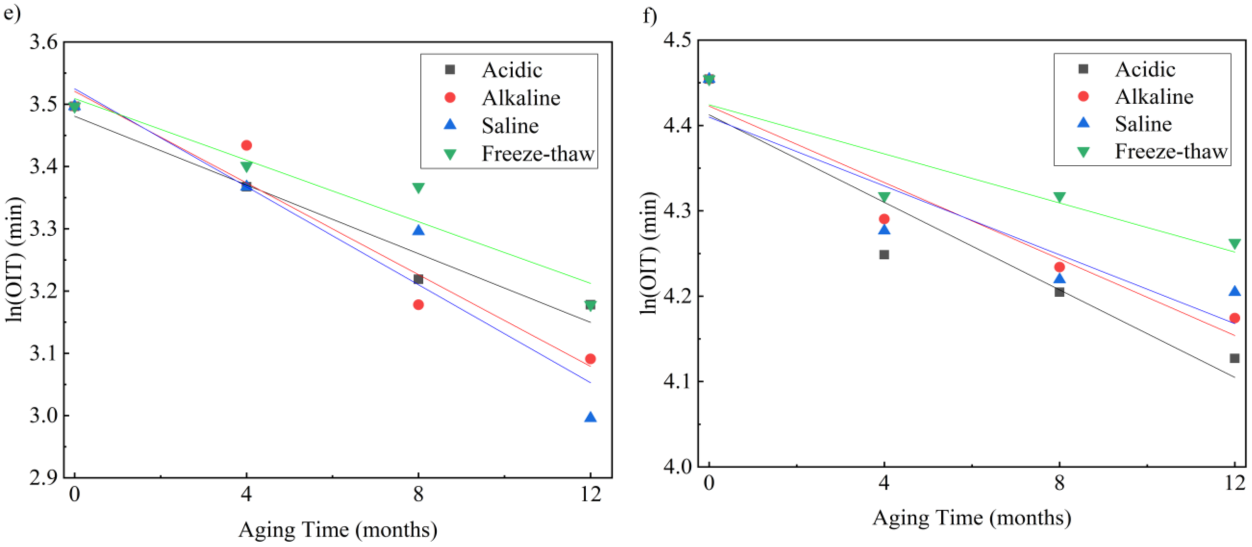


Figure 7.3 Plot of ln(OIT) with aging time under different exposure conditions a) A-R b) A-V c) B-R d) B-V e) C-V f) D-R

Table 7.1 AO depletion rate of HDPE pipes under different exposure conditions

Specimen	Exposure conditions				Comparison		
	Acidic	Alkaline	Saline	Freeze-thaw	Acidic/ Freeze-thaw	Alkaline/ Freeze-thaw	Saline/ Freeze-thaw
A-R	0.019	0.020	0.023	0.016	1.19	1.25	1.44
A-V	0.021	0.029	0.028	0.011	1.91	2.64	2.55
B-R	0.033	0.042	0.043	0.029	1.14	1.45	1.48
B-V	0.015	0.029	0.039	0.012	1.25	2.42	3.25
C-V	0.028	0.037	0.039	0.025	1.12	1.48	1.56
D-R	0.026	0.022	0.020	0.014	1.86	1.57	1.43

7.3.2 Tensile Strength

Figure 7.4 shows the average tensile strength of original and aged HDPE pipes for 12 months. The standard deviation is represented by vertical bars. The stress-strain behavior of specimens is similar to that in another study [173] in which three regions were observed: elastic, neck propagation, and plastic. The tensile strength of the specimens before immersion is considered the reference. There was no significant degradation in pipe tensile strength before and after immersion in the different solutions.

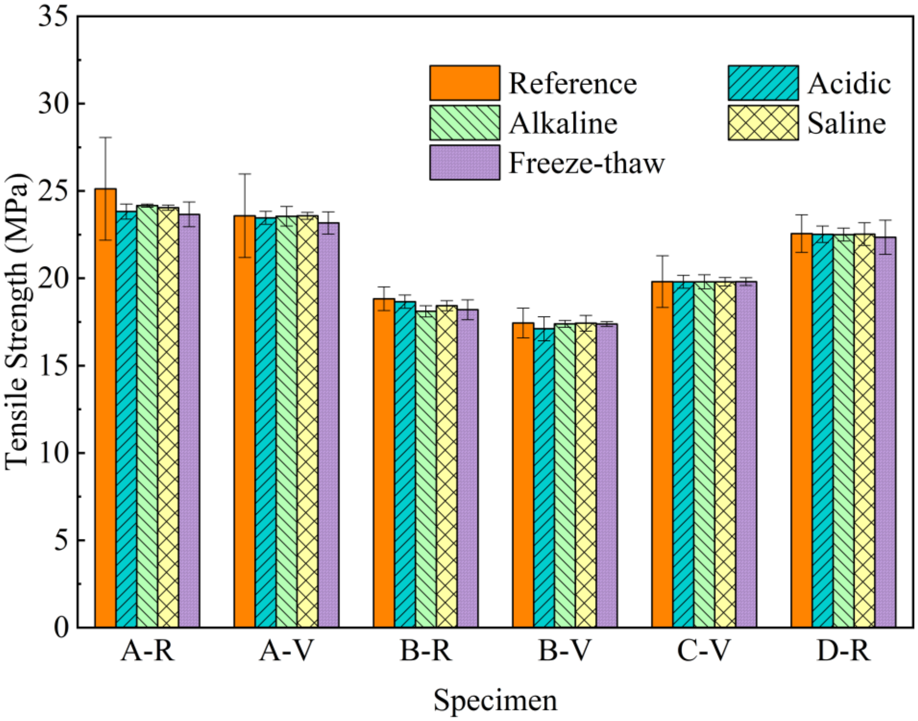


Figure 7.4 Tensile strength of HDPE pipes before and after exposure to different conditions

There was, however, a small variation between the measurements. Therefore, a one-way analysis of variance (ANOVA) was used to clarify if there are significant differences between measurements. Table 7.2 shows that the F-value was less than the F-critical value and the P-value was greater than 0.05 for tensile strength. Therefore, under laboratory conditions including conditioning in saline, acidic, or alkaline solutions, as well as freeze-thaw cycles, it can be concluded that the tensile strength was not significant affected. Furthermore, the tensile strength values for the recycled pipes exposed to the different solutions were relatively unchanged compared to the reference values.

Table 7.2 One-way ANOVA of tensile strength before and after immersion

Specimen	Description	Sum of squares	df	Mean square	F-value	P-value	F-critical
A-R	Between groups	6.1	4	1.5	0.665	0.625	3.007
	Within groups	36.8	16	2.3	–	–	–
	Total	42.9	20	–	–	–	–
A-V	Between groups	0.5	4	0.1	0.076	0.988	3.007
	Within groups	25.5	16	1.6	–	–	–
	Total	26.0	20	–	–	–	–
B-R	Between groups	1.6	4	0.4	1.684	0.203	3.007
	Within groups	3.8	16	0.2	–	–	–
	Total	5.4	20	–	–	–	–
B-V	Between groups	0.3	4	0.1	0.243	0.910	3.007
	Within groups	5.1	16	0.3	–	–	–
	Total	5.4	20	–	–	–	–
C-V	Between groups	0.0004	4	9E-05	0.0002	1.000	3.007
	Within groups	9.9519	16	0.6	–	–	–
	Total	9.9523	20	–	–	–	–
D-R	Between groups	0.1	4	0.03	0.043	0.996	3.007
	Within groups	9.9	16	0.6	–	–	–
	Total	10	20	–	–	–	–

7.3.3 Long-term Modulus

Time-temperature superposition (TTS) is an extrapolation techniques widely applied to predict the long-term modulus of materials. The theory of this technique is based on the empirical Williams-Landed-Ferry (WLF) relationship [199]. Although this technique was ninitially developed for amorphous polymers, Nielsen and Landel [200] recommended that it could also be employed for semicrystalline polymers (HDPE). The Wicket plot [201,202] was used to confirm that the materials in this study were suitable for analysis with the WLF relationship. A specimen measuring 40 x 10 x 4 mm^3 was heated from 0 to 100^{0}C with a heating rate of 5^{0}C/min at 1 Hz using the single cantilever mode. As shown in Fig. 7.5, the Wicket-plot is almost symmetrical, which is suitable for applying the principle of TTS.

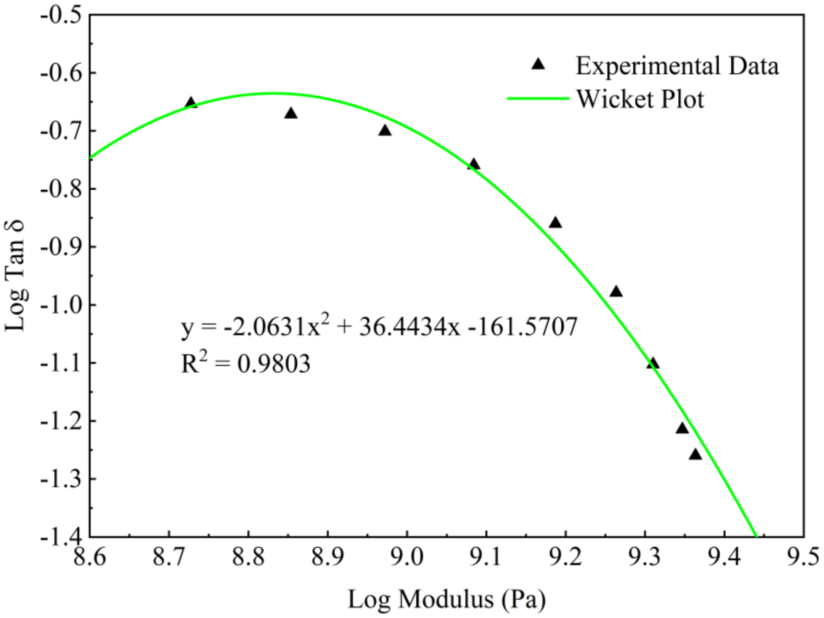


Figure 7.5 Theoretical Wicket plot

As discussed above, the effects of temperature and frequency variations are interchangeable according to the TTS principle. Therefore, data obtained over a limited frequency range can be extended by applying a shift factor to data obtained over a range of temperatures. First, a specimen was heated from 5 to 100^0C, at steps of 5^0C along a range of frequencies between 0.01Hz and 100Hz (Fig. 7.6a). The curves obtained were then shifted to generate a master curve (Fig. 7.6b), using a reference temperature and shift factor as illustrated in the WLF equation (Eq. 2.1). A reference temperature of 50^0C was used, which is just below the softening temperature of the materials. With the TTS principle, temperatures below the reference temperature are shifted to higher frequencies, while temperatures above are shifted to lower frequencies.

$$\log (a_T) = \frac{-C_1(T-T_r)}{C_2+T-T_r} \qquad (2.1)$$

where a_T is the shift factor, C_1 and C_2 the WLF coefficients (K), T the temperature (K), and T_r the reference temperature (K).

Figure 7.6c shows the long-term modulus of HDPE pipes before and after immersion in different solutions for 6.5 months. A higher variation in the long-term modulus values was observed. Therefore, a one-way analysis of variance (ANOVA) was used to determine if there was a significant difference between the measurements before and after immersion.

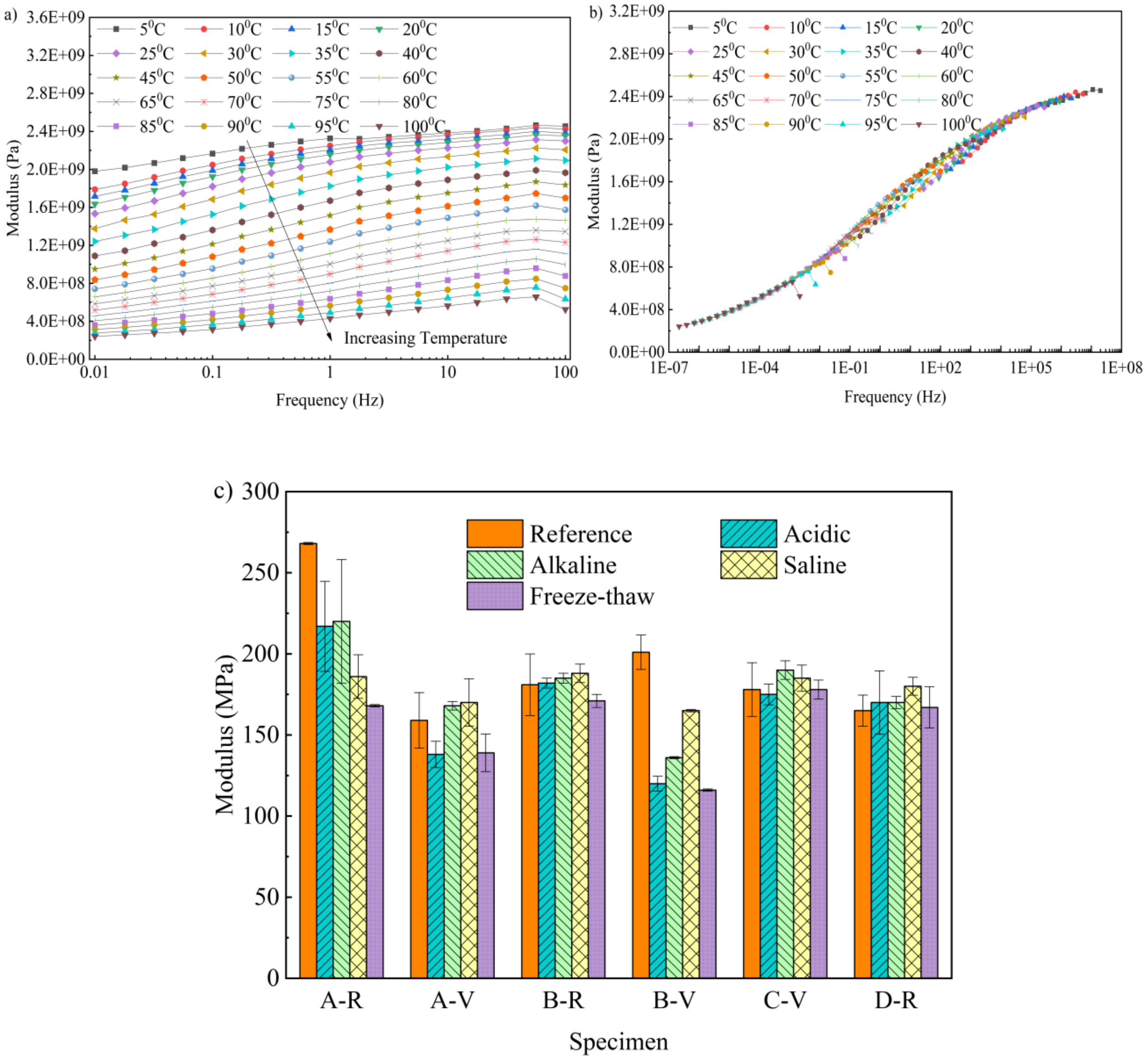


Figure 7.6 TTS/DMA analysis a) Temperature-frequency sweep b) A typical of master curve for A-R pipe c) Long-term modulus of HDPE pipes before and after exposure to different conditions

Table 7.3 indicates that the F-value is higher than the F-critical value for A-R, A-V, and B-V pipes, which suggests that one or more pairs of measurements were significantly different. In contrast, the long-term modulus values for B-R, C-V, and D-R pipes were relatively unchanged before and after immersion. The Tukey's HSD test was then applied to confirm whether the pairs of measurements are indeed significantly different. Table 7.4 shows no significant difference between the pair of measurements for A-R and A-V pipes as the q-statistic value is less than the q-critical value. The difference is mainly observed with the B-V pipe before and after immersion in the different solutions. Based on the results, it can be concluded that the long-term modulus of

HDPE pipes was not significantly affected by the saline, acidic, or alkaline solutions, or the freeze-thaw cycles.

Table 7.3 One-way ANOVA of long-term modulus before and after immersion

Specimen	Description	Sum of squares	df	Mean square	F-value	P-value	F-critical
A-R	Between groups	11802.1	4	2950.5	4.445	0.042	4.120
	Within groups	4646.2	7	663.7	–	–	–
	Total	16448.3	11	–	–	–	–
A-V	Between groups	2778.3	4	694.6	4.848	0.020	3.478
	Within groups	1432.7	10	143.3	–	–	–
	Total	4211.0	14	–	–	–	–
B-R	Between groups	496.4	4	124.1	1.448	0.289	3.478
	Within groups	857.3	10	85.7	–	–	–
	Total	1353.7	14	–	–	–	–
B-V	Between groups	13824.8	4	3456.2	90.217	4E-06	4.120
	Within groups	268.2	7	38.3	–	–	–
	Total	14093.0	11	–	–	–	–
C-V	Between groups	451.6	4	112.9	1.261	0.347	3.478
	Within groups	895.3	10	89.5	–	–	–
	Total	1346.9	14	–	–	–	–
D-R	Between groups	386.9	4	96.7	0.709	0.604	3.478
	Within groups	1364.7	10	136.5	–	–	–
	Total	1751.6	14	–	–	–	–

Table 7.4 Post-hoc Tukey's HSD test

Specimen	Treatment pair	Tukey's HSD q-statistic	Tukey's HSD q-critical	Tukey's HSD inference
A-R	Ref vs Acidic	3.0367	5.0615	insignificant
	Ref vs Alkaline	2.8362	5.0615	insignificant
	Ref vs Saline	4.5012	5.0615	insignificant
	Ref vs Freeze-thaw	5.4893	5.0615	**significant**
	Acidic vs Alkaline	0.2241	5.0615	insignificant
	Acidic vs Saline	1.8942	5.0615	insignificant
	Acidic vs Freeze-thaw	2.9765	5.0615	insignificant
	Alkaline vs Saline	2.0946	5.0615	insignificant
	Alkaline vs Freeze-thaw	3.1770	5.0615	insignificant
	Saline vs Freeze-thaw	0.9881	5.0615	insignificant
A-V	Ref vs Acidic	3.0388	4.6552	insignificant
	Ref vs Alkaline	1.2541	4.6552	insignificant
	Ref vs Saline	1.4953	4.6552	insignificant
	Ref vs Freeze-thaw	2.8941	4.6552	insignificant
	Acidic vs Alkaline	4.2930	4.6552	insignificant
	Acidic vs Saline	4.5341	4.6552	insignificant
	Acidic vs Freeze-thaw	0.1447	4.6552	insignificant
	Alkaline vs Saline	0.2412	4.6552	insignificant
	Alkaline vs Freeze-thaw	4.1483	4.6552	insignificant
	Saline vs Freeze-thaw	4.3894	4.6552	insignificant
B-V	Ref vs Acidic	22.7602	5.0615	**significant**
	Ref vs Alkaline	16.4778	5.0615	**significant**
	Ref vs Saline	9.2192	5.0615	**significant**
	Ref vs Freeze-thaw	21.4837	5.0615	**significant**
	Acidic vs Alkaline	3.8796	5.0615	insignificant
	Acidic vs Saline	11.1381	5.0615	**significant**
	Acidic vs Freeze-thaw	1.1263	5.0615	insignificant
	Alkaline vs Saline	6.6261	5.0615	**significant**
	Alkaline vs Freeze-thaw	4.5697	5.0615	insignificant
	Saline vs Freeze-thaw	11.1959	5.0615	**significant**

7.4 Conclusion

The antioxidant depletion rate of six HDPE pipes was evaluated with accelerated aging in acidic, alkaline, and saline solutions and under freeze-thaw cycles. The results show that the OIT value decreased with aging time in the solution. The OIT percent retained was above 55% for all solutions. After 12 months of aging had been completed, the antioxidant depletion rate was: Saline > Alkaline > Acidic > Freeze-thaw. For specimens subjected to freeze-thaw cycles, the depletion was about 1.12-1.91 lower than that in acidic solution, 1.25-2.64 times lower than that in alkaline solution, and 1.43-3.25 times lower than that in saline solution. The antioxidant depletion rate herein refers only to the first stage (t_1) of the degradation process and does not consider stages t_2 and t_3 corresponding to the degradation with a reduction in properties. The HDPE pipes all met the OIT minimum of 20 min according to ASTM D3895-19, regardless of the manufacturing process and antioxidant package. Moreover, it must be pointed out that the laboratory conditionings were more corrosive than the actual environment of the pipes in the field. Based on one-way analysis of variance (ANOVA) and Tukey's HSD tests, the specimens exhibited no significant changes in tensile strength or long-term modulus after immersion in the different solutions. Whether produced with recycled or virgin resins, the pipes maintained good mechanical properties after laboratory aging. The findings from this study again confirm the appropriateness of using pipes made with recycled HDPE for transportation infrastructure applications. Recycled pipes can deliver short- and long-term performance when they contain appropriate antioxidants and an improved recycling process (e.g., super-clean recycling).

Data Availability Statement

Some or all data, models, or code that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This research received financial support from the Natural Science and Engineering Research Council of Canada (NSERC), the NSERC Research Chair in Innovative FRP Reinforcement for Sustainable Concrete Infrastructures, the Tier-1 Canada Research Chair in Composite Materials for Civil structures, the Fonds Québécois de la recherche sur la nature et les technologies (FQRNT), the Ministry of Transportation of Quebec (MTQ), and the University of Sherbrooke Research Centre on Composite Materials (CRUSMaC).

8 CHAPITRE 8: ÉTUDE SUR DES TUYAUX EN PEHD PRÉLEVÉS DU TERRAIN

8.1 Matériaux

Le MTQ tient à établir une corrélation entre le temps de dégradation accéléré en laboratoire des tuyaux et le temps réel in situ nécessaire pour atteindre ce même niveau de dégradation. Pour ce faire, dix tuyaux fabriqués avec des résines vierges ont été prélevés sur le terrain par le Ministère à l'automne 2018 en Outaouais et au Centre-du-Québec (Figure 6.1). Les paramètres de chaque tuyau sont indiqués dans le tableau 8.1.

Tableau 8.1 Paramètres des tuyaux prélevés sur le terrain en 2018

Tuyau	Profil	Diamètre – épaisseur des parois (mm)	Année de construction	Année de réhabilitation	Échantillon prélevé en	Années en service
T1	Ouvert	900 – 4,00	2013	-	2018	5
T2	Ouvert	1200 – 4,80	2010	-	2018	8
T3	Ouvert	1200 – 4,50	2003	-	2018	15
T4	Ouvert	900 – 5,00	1993	-	2018	25
T5	Fermé	1200 – 8,25	2013	-	2018	5
T6	Fermé	1900 – 10,85	-	2008	2018	10
T7	Fermé	1200 – 8,90	-	2005	2018	13
T8	Fermé	900 – 7,00	-	2004	2018	14
T9	Fermé	900 – 7,80	-	2004	2018	14
T10	Fermé	1800 – 10,40	-	2000	2018	18

8.2 Essais et résultats

Les essais effectués sur ces tuyaux sont similaires à ceux effectués sur les six tuyaux fabriqués avec et sans résines recyclées (A-R, A-V, B-R, B-V, C-V, D-R) :

- Résistance à long terme à la traction.
- Résistance à long terme à la fissuration sous contrainte.
- Module à long terme.
- Oxydation à long terme.
- Chimie de surface : FTIR

8.2.1 Résistance à la traction

La dimension de l'échantillon d'essai et la procédure de l'essai sont similaires à ceux de l'essai de résistance à court terme à la traction des six tuyaux neufs. Le tableau 8.2 montre les valeurs moyennes de résistance à la traction résiduelle des tuyaux prélevés sur le terrain. Il existe une différence de valeur de résistance à la traction entre les tuyaux. Cette valeur dépend de la durée en service, des propriétés de la résine, du procédé de fabrication et des conditions environnementales auxquelles les tuyaux sont soumis.

Tableau 8.2 Valeur de résistance à la traction des tuyaux prélevés sur le terrain

Spécimen	Profil	Année de service	Limite d'élasticité (MPa)
T1	Ouvert	5 ans	19,91
T2	Ouvert	8 ans	25,87
T3	Ouvert	15 ans	24,24
T4	Ouvert	25 ans	12,29
T5	Fermé	5 ans	18,75
T6	Fermé	10 ans	19,19
T7	Fermé	13 ans	19,00
T8	Fermé	14 ans	19,71
T9	Fermé	14 ans	18,67
T10	Fermé	18 ans	18,50

8.2.2 Module à long terme

La dimension des échantillons et la procédure de l'essai sont similaires à ceux de l'essai de module à long terme des 6 tuyaux neufs. Le tableau 8.3 montre que les valeurs de module à long terme des tuyaux sont différentes. Cette différence dépend non seulement de la durée d'utilisation mais aussi de la composition de la résine, du procédé de fabrication et des différentes conditions de service. De plus, les valeurs de module à long terme des tuyaux sur le terrain sont assez similaires aux valeurs de module à long terme obtenues avec les six tuyaux neufs conditionnés dans différentes solutions (entre 138 et 220 MPa).

Tableau 8.3 Module à long terme des tuyaux prélevés sur le terrain

Spécimen	Profil	Année de service	E' (MPa)
T1	Ouvert	5 ans	184
T2	Ouvert	8 ans	226
T3	Ouvert	15 ans	230
T4	Ouvert	25 ans	152
T5	Fermé	5 ans	154
T6	Fermé	10 ans	237
T7	Fermé	13 ans	189
T8	Fermé	14 ans	223
T9	Fermé	14 ans	214
T10	Fermé	18 ans	207

8.2.3 Oxydation à long terme

La dimension des échantillons et la procédure de l'essai sont similaires à ceux de l'essai d'oxydation à long terme des 6 tuyaux neufs. Cependant, des échantillons ont été découpés dans les parois intérieure et extérieure pour déterminer le taux d'épuisement des antioxydants ainsi que l'effet des conditions environnementales sur les deux surfaces de la paroi du tuyau [203]. Le tableau 8.4 montre les valeurs d'OIT sur les parois intérieure et extérieure des tuyaux sur le terrain. Les résultats montrent que l'oxydation a essentiellement lieu sur la paroi externe puisqu'une diminution des valeurs d'OIT et donc de la teneur des antioxydants est observée.

Tableau 8.4 Valeurs d'OIT sur les parois intérieure et extérieure des tuyaux prélevés sur le terrain

Spécimen	Profil	Année d'utilisation	OIT (min)	
			Paroi intérieure	Paroi extérieure
T1	Ouvert	5 ans	22	17
T2	Ouvert	8 ans	38	27
T3	Ouvert	15 ans	25	22
T4	Ouvert	25 ans	50	21
T5	Fermé	5 ans	76	20
T6	Fermé	10 ans	93	47
T7	Fermé	13 ans	77	40
T8	Fermé	14 ans	76	46
T9	Fermé	14 ans	96	22
T10	Fermé	18 ans	46	32

8.2.4 Chimie de surface

Les dimensions des échantillons et la procédure sont similaires à ceux de l'essai de chimie de surface des 6 tuyaux neufs. Les spectres FTIR ont été réalisées entre 4000 et 600 cm^{-1} à une résolution de 4 cm^{-1}. Les résultats pour les parois extérieure et intérieure sont présentés à la figure 8.1. La présence de groupements hydroxyles est observée à 3380 cm^{-1}. Les pics à 2913 et 2847 cm^{-1} sont des bandes typiques du groupe C–H du PEHD et ceux à 1465 et 722 cm^{-1} correspondent aux groupements CH_2 [155,204,205]. La présence de groupes carbonyle cétoniques à 1717 cm^{-1} de même que celle de groupements C–OH et C–O–C à 1040 cm^{-1} témoigne d'une oxydation [156]. Par ailleurs, on observe que l'intensité du pic à 1717 cm^{-1} de l'échantillon de la paroi extérieure est plus élevée que celui de la paroi intérieure. Une explication possible de ce phénomène est la formation d'esters provoquée par l'oxydation [156].

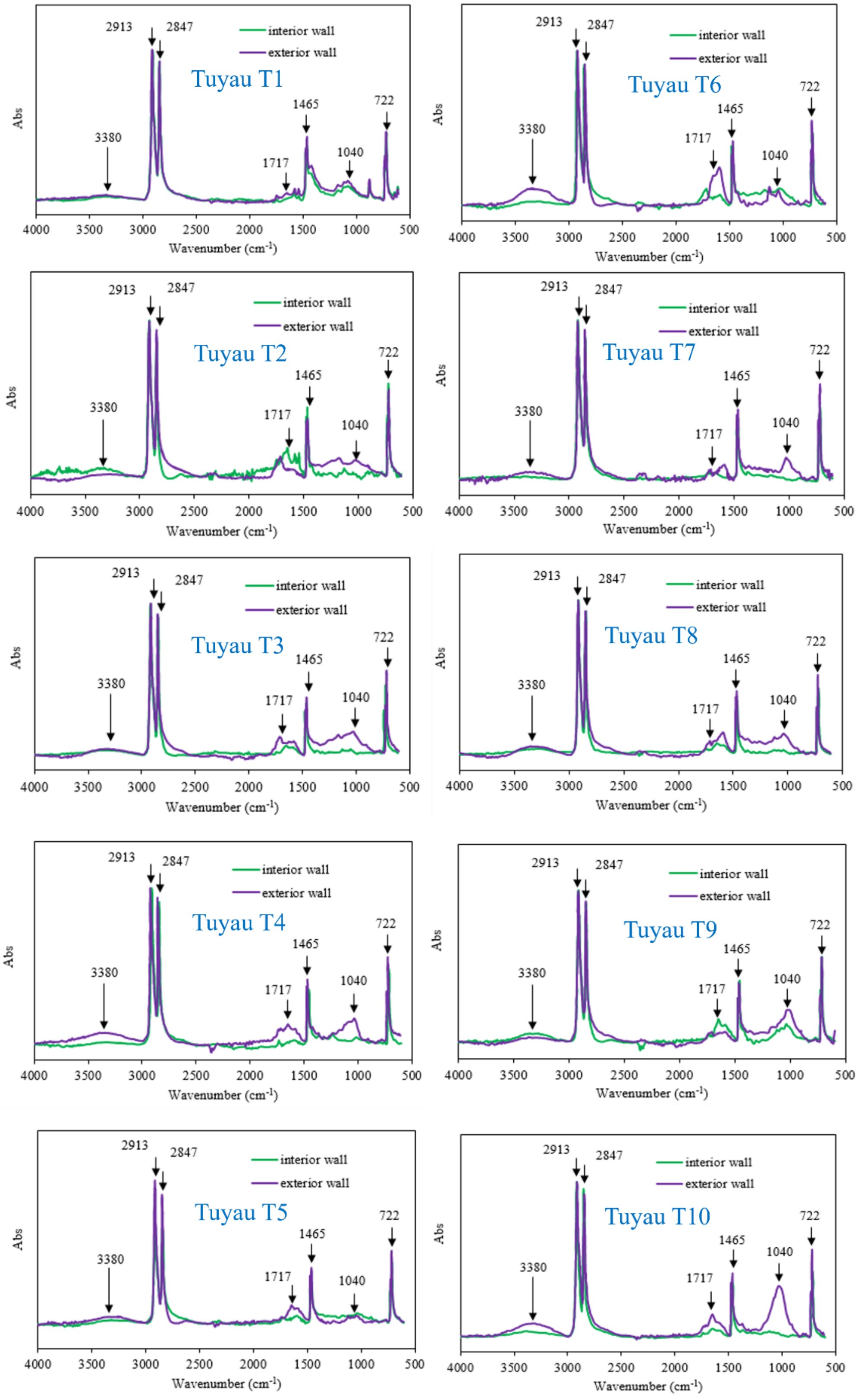


Figure 8.1 Spectres FTIR des tuyaux prélevés sur le terrain

8.2.5 Essai de résistance à la fissuration sous contrainte (SCR).

Les dimensions de l'échantillon d'essai et la procédure sont similaires à ceux de l'essai de résistance à la fissuration sous contrainte des 6 tuyaux neufs [206]. Les résultats des 10 tuyaux prélevés sur le terrain sont présentés au tableau 8.5. La durée de vie des tuyaux a été déterminée en calculant les paramètres A, B, C à partir de la méthode de RPM (Tableaux 8.6 et 8.7).

Tableau 8.5 Résultats des essais SCG (FM 5-572)

Spécimen	Condition d'essai	Temps moyen de rupture (heures)
T1	3,1 MPa/80^{0}C	187,3
	4,48 MPa/80^{0}C	76,5
	4,48 MPa/70^{0}C	412,8
T2	3,1 MPa/80^{0}C	65,5
	4,48 MPa/80^{0}C	2,1
	4,48 MPa/70^{0}C	6,1
T3	3,1 MPa/80^{0}C	194,8
	4,48 MPa/80^{0}C	11,7
	4,48 MPa/70^{0}C	36,4
T4	3,1 MPa/80^{0}C	313,6
	4,48 MPa/80^{0}C	0,1
	4,48 MPa/70^{0}C	206,8
T5	3,1 MPa/80^{0}C	-
	4,48 MPa/80^{0}C	> 1683
	4,48 MPa/70^{0}C	-
T6	3,1 MPa/80^{0}C	-
	4,48 MPa/80^{0}C	> 1683
	4,48 MPa/70^{0}C	-
T7	3,1 MPa/80^{0}C	-
	4,48 MPa/80^{0}C	> 1683
	4,48 MPa/70^{0}C	-
T8	3,1 MPa/80^{0}C	-
	4,48 MPa/80^{0}C	> 1684
	4,48 MPa/70^{0}C	-
T9	3,1 MPa/80^{0}C	-
	4,48 MPa/80^{0}C	> 1684
	4,48 MPa/70^{0}C	-
T10	3,1 MPa/80^{0}C	-
	4,48 MPa/80^{0}C	> 1638
	4,48 MPa/70^{0}C	-

Tableau 8.6 Paramètres A, B, C selon la méthode de RPM

Spécimen	A	B	C
T1	-23,227	11281,090	-859,309
T2	-15,932	15088,330	-3325,493
T3	-15,839	135962,390	-2699,768
T4	-	-	-
T5	-	-	-
T6	-	-	-
T7	-	-	-
T8	-	-	-
T9	-	-	-
T10	-	-	-

Tableau 8.7 Durée de vie des tuyaux prélevés sur le terrain à 23^0C et 10^0C

Spécimen	à 10^0C, 3,45 MPa (500psi) (Québec)	à 23^0C, 3,45 MPa (500psi) (Floride)
T1	31489 ans	1280 ans
T2	53 ans	6 ans
T3	248 ans	26 ans
T4	-	-
T5	-	-
T6	-	-
T7	-	-
T8	-	-
T9	-	-
T10	-	-

La durée de vie estimée n'a été déterminée que pour les tuyaux T1, T2 et T3 car il n’y a pas suffisamment de données pour les autres afin d’effectuer une prédiction fiable. Les températures de référence pour estimer la durée de vie des tuyaux sont 23^0C, température moyenne correspondant à un climat subtropical (Floride) et 10^0C, température moyenne correspondant à un climat nordique (Québec).Il est à noter que la durée de vie estimée des tuyaux prélevés sur le terrain varie considérablement, ce qui peut s'expliquer, comme nous l’avons déjà indiqué, par la durée en service au moment des essais, les propriétés de la résine, le procédé de fabrication et les conditions environnementales auxquelles les tuyaux sont soumis.

8.3 Conclusions

- Le module de traction à long terme a été évalué à l'aide des données de DMA. Les résultats obtenus sont similaires à ceux des tuyaux (A-R, A-V, B-R, B-V, C-V, D-R) qui ont été immergés dans des solutions d'acide, de base et de sel ou soumis à des cycles de gel/dégel.
- La résistance à la traction résiduelle des tuyaux prélevés sur le terrain a été déterminée et les résultats montrent une différence de valeur de résistance entre les différents tuyaux. Celle-ci dépend de la durée de service, des propriétés de la résine, du procédé de fabrication et des conditions environnementales auxquelles les tuyaux sont soumis.
- Tous les échantillons présentent des valeurs d’OIT très bonnes malgré une certaine oxydation sur la paroi extérieure des tuyaux sur le terrain. Les résultats montrent que ces valeurs répondent aux recommandations d’au moins 20 minutes.
- L’analyse chimique par FTIR des parois extérieure et intérieure montre que l’oxydation est faible et a essentiellement lieu sur la paroi externe.
- Les échantillons ont été découpés et envoyés au Texas pour les essais de SCR. La durée de vie des tuyaux prélevés sur le terrain varie considérablement, ce qui peut s'expliquer par les variations dans la durée de service, les propriétés de la résine, le procédé de fabrication et les conditions environnementales auxquelles les tuyaux sont soumis.

9 CHAPITRE 9: CONCLUSIONS ET TRAVAUX FUTURS

9.1 Conclusions générales

Cette vaste étude des propriétés physiques, mécaniques et fonctionnelles de tuyaux de PEHD fabriqués avec des résines vierges et recyclées a été entreprise à la demande du ministère des Transports du Québec dans le but de répondre à un certain nombre de questions portant sur la durabilité de ses tuyaux pour les utilisations dans les ponceaux et le drainage des eaux pluviales. Entre autres, le Ministère s'interrogeait sur: (1) le maintien des propriétés fonctionnelles des tuyaux à court et long terme; (2) leur durabilité selon l'environnement dans lequel ils seront placés (gel-dégel, humidité, UV, milieux corrosifs); (3) l'impact du type de résine employée, vierge ou recyclée et (4) la qualité des tuyaux selon le fabricant. Ainsi, quatre fabricants ont été sélectionnés (A à D) et leurs installations visitées. L'étude a porté sur six tuyaux de 900 mm de diamètre, dont quatre à profil ouvert et deux à profil fermé. Trois de ces tuyaux ont été fabriqués selon la norme BNQ 3624-120 (2016) [19] à partir de résine vierge (A-V, B-V, C-V), deux avec différentes proportions de résine recyclée post-consommation (A-R, D-R) et un avec de la résine recyclée post-production (B-R). Les propriétés de ces échantillons de référence ont été déterminées tandis qu'un certain nombre a été conditionné avec des cycles de gel-dégel et dans des solutions saline, acide et alcaline à 60^0C jusqu'à une durée de 12 mois. La résistance à la traction à court et à long terme, le module à long terme, la résistance au fluage, la résistance à la fissuration lente et l'oxydation ont ensuite été évalués.

Parallèlement, 10 échantillons de tuyaux en service ont été prélevés pour fins d'analyses sans qu'il soit possible de retracer leur origine ni la nature de leurs composants (type de résine, additifs, etc.). Seuls, leur durée en service et leur environnement étaient connues. Les différentes analyses et essais effectués sur tous ces échantillons ont permis de dresser un portrait précis de leurs propriétés à court et long terme ainsi que du maintien de ces propriétés dans le temps dans les conditions d'utilisation anticipées.

Pour les tuyaux neufs :

Pour ce qui est des <u>propriétés à court terme</u>, nous pouvons conclure que :

- Les résultats de caractérisation mécanique sont conformes aux recommandations de la norme ASTM D3350-14 [29] pour les tuyaux des fabricants A, C et D. Par contre, les données des essais de traction effectués selon la norme ASTM D638-14 [20] sur les tuyaux du manufacturier B sont légèrement inférieures aux recommandations de cette même norme.
- Il n'existe aucune incidence de l'origine de la résine, vierge ou recyclée, sur les propriétés mécaniques à court terme des tuyaux.
- Les mesures physiques (densité, indice de fusion, stabilité thermique, point de ramollissement et teneur en noir de carbone) sont conformes à la norme ASTM D3350-14 [29].

- L'exposition des échantillons aux UV n'affecte pas significativement les propriétés des tuyaux en raison de la protection offerte par le noir de carbone et les antioxydants.

En ce qui concerne les propriétés et essais à long terme qui sont de trois types (rupture ductile, rupture fragile par fissuration sous contrainte et dégradation chimique), les conclusions sont les suivantes :

Pour la rupture ductile :

- Les résultats des essais de résistance à la traction après exposition aux différents milieux indiquent que les solutions chimiques n'affectent pas de manière significative la résistance à la traction des tuyaux quel que soit le type de résine.
- Suite aux essais de SIM, l'extrapolation des courbes maîtresses montre que la limite élastique à 100 ans est d'au moins 8 MPa (1160 lb/po^2) à 23^{0}C, température moyenne correspondant à un climat subtropical (Floride) et 10 MPa (1450 lb/po^2) à 10^{0}C, température moyenne correspondant à un climat nordique (Québec).
- Le calcul du module de traction à 100 ans mesuré par DMA et calculé à partir des courbes maîtresses indique que la durée de vie estimée est similaire à celle obtenue avec la méthode SIM.

Pour la rupture fragile par fissuration sous contrainte (SCR)

- Les essais de SCR réalisés selon la norme FM 5-573 [25] sur des échantillons de référence et conditionnés en solutions acides, basiques, salines et avec des cycles de gel-dégel montrent que certains échantillons présentent une rupture ductile, ce qui a nécessité une réduction des contraintes pour la suite du projet afin d'évaluer adéquatement la SCR. Les résultats montrent que la durée de vie anticipée des tuyaux répond aux exigences pour une durée de service de 100 ans à 10^{0}C et une contrainte de 3,45 MPa.

Pour la dégradation chimique

- À l'origine, tous les échantillons présentent des valeurs d'OIT conformes. Après 12 mois de conditionnement en solutions, celles-ci sont plus ou moins affectées, mais demeurent supérieures à la valeur seuil de 20 minutes. On notera que le taux d'antioxydants des échantillons soumis aux cycles de gel-dégel diminue plus lentement que pour ceux conditionnés en solutions du fait de l'absence d'eau susceptible de les dissoudre.

Pour les tuyaux sur le terrain

Des échantillons de tuyaux prélevés sur le terrain en service ont été testés. Le module de traction à long terme a été évalué par DMA et les résultats obtenus sont similaires à ceux des tuyaux conditionnés précédemment. Les résistances à la traction résiduelle présentent des différences selon les tuyaux, qui peuvent dépendre de la durée en service, des propriétés de la résine, du procédé de fabrication et des conditions environnementales auxquelles les tuyaux sont soumis. Tous les échantillons présentent des valeurs d'OIT supérieures à la recommandation de 20 minutes, malgré une certaine perte sur les parois extérieures. L'analyse FTIR confirme cette conclusion puisqu'on observe une légère oxydation de ces dernières. Suite aux essais de SCR, une grande variation de la durée de vie a été enregistrée, qui peut s'expliquer par les mêmes facteurs que ceux indiqués ci-haut.

En résumé, ce projet de recherche confirme la durabilité des tuyaux en PEHD pour les applications envisagées (ponceaux, drains d'eaux pluviales) et dans les environnements prévus (climat nordique, eaux pluviales), qu'ils soient fabriqués à partir de résine vierge ou de résine recyclée. Les différents paramètres mécaniques ou chimiques (résistance à l'oxydation) à court et long terme essentiels à la bonne tenue des tuyaux en service durant de nombreuses décennies sont positifs, en sorte que ces matériaux peuvent être employés en toute sérénité par le Ministère. On peut conclure que les tuyaux en PEHD du projet répondent aux exigences du BNQ 3624-120 (2016) [19].

9.2 Recommandations pour des études futures

Ce projet de recherche présente des résultats expérimentaux inédits pour les tuyaux en PEHD recyclés utilisés dans les infrastructures de transport du Québec où l'environnement est rude. Cette étude était nécessaire étant donné l'impact que peuvent avoir les facteurs climatiques (basses températures et cycles de gel-dégel) sur la performance à long terme des tuyaux. Cependant, des études supplémentaires sur le comportement des tuyaux en PEHD recyclés sont recommandées en fonction des résultats de l'étude actuelle pour couvrir les points suivants :

- Identifier les types d'antioxydants dans les tuyaux et évaluer l'impact de l'environnement d'exposition sur ceux-ci.
- Utiliser la méthode SSM (Stepped Isotress Method) pour prédire la résistance à la traction à long terme des tuyaux. Cette méthode est basée sur la SIM bien qu'elle utilise la contrainte à l'énergie d'activation au lieu de la température. Une température élevée étant l'un des facteurs modifiant les propriétés du HDPE, la méthode SSM éviterait cet inconvénient.
- Augmenter le contenu en résine recyclée tout en garantissant la performance du tuyau.
- Étudier la possibilité de combiner un nanocomposite pour améliorer la rigidité et le module de conservation du tuyau.

ANNEXE A: LISTE DES PUBLICATIONS

Les différentes publications rédigées au cours de la thèse pour des conférences internationales ou des journaux sont listées.

Articles de journaux:

1. Nguyen, K. Q., Mwiseneza, C., Mohamed, K., Cousin, P., Robert, M., & Benmokrane, B. (2021). Long-term testing methods for HDPE pipe-advantages and disadvantages: A review. Engineering Fracture Mechanics, 107629. https://doi.org/10.1016/j.engfracmech.2021.107629.
2. K. Q. Nguyen, P. Cousin, K. Mohamed, M. Robert, and B. Benmokrane, "Comparing Short-Term Performance of Corrugated HDPE Pipe Made with or without Recycled Resins for Transportation Infrastructure Applications," J. Mater. Civ. Eng., vol. 34, no. 2, p. 04021427, Feb. 2022, https://doi.org/10.1061/(ASCE)MT.1943-5533.0004067.
3. Nguyen, K. Q., Cousin, P., Mohamed, K., Robert, M., A. El-Safty & Benmokrane, B. (2021). Effects of Ultraviolet Radiation on Recycled and Virgin HDPE Corrugated Pipes Used in Road Drainage Systems. (soumis), 12 Octobre 2021.
4. Nguyen, K. Q., Cousin, P., Mohamed, K., Robert, M., A. El-Safty & Benmokrane, B. Stress Crack Resistance and Life Prediction of Corrugated Recycled and Virgin HDPE Pipes Used in Road Drainage Systems: A Case Study in Quebec, Canada. (soumis), 16 décembre 2021.
5. Nguyen, K. Q., Cousin, P., Mohamed, K., Robert, M., & Benmokrane, B. Effects of Exposure Conditions on Antioxidant Depletion, Tensile Strength, and Long-term Modulus from Corrugated HDPE pipes made With and Without Recycled Resins. (soumis), 19 janvier 2022.

Articles de conferences:

6. Nguyen, K. Q., Cousin, P., Mohamed, K., Robert, M., & Benmokrane, B. (2021). Influence of Wall Thickness on the Thermo-mechanical properties of Aging HDPE pipes under Freeze-thaw cycles in Quebec province, Canada. CSCE Annual Conference. (accepté), 22 April 2021.
7. Nguyen, K. Q., Cousin, P., Mohamed, K., Robert, M., & Benmokrane, B. (2021). Stress Crack Resistance of Recycled and Virgin HDPE corrugated pipe for Transportation Infrastructure Applications. CSCE Annual Conference. (accepté), 22 April 2021.
8. Nguyen, K. Q., Mwiseneza, C., Mohamed, K., Cousin, P., Robert, M., L. Marc-Antoine & Benmokrane, B. (2021). The short-term performance of corrugated HDPE pipe made with and without recycled resins - physicochemical properties. ACMBS-VIII (8th International Conference on Advanced Composite Materials in Bridges and Structures). (accepté), 27 March, 2020.

Rapports de recherche:

9. Nguyen, K. Q., Cousin, P., Mohamed, K., Robert, M., & Benmokrane, B. Prédiction de la durée de vie des tuyaux en polyéthylène à haute densité (PEHD) avec et sans résines recyclées (Contrat de recherche R683.2).

LISTE DES RÉFÉRENCES

[1] H. PPI, “Second Edition Handbook of PE Pipe | HDPE Handbook,” 2008. https://plasticpipe.org/publications/pe-handbook.html (accessed Oct. 24, 2019).

[2] Reddy, D V, “Evaluation of plastic piping for culverts and storm sewers,” 1999.

[3] AASHTO-M 294, “Standard Specification for Corrugated Polyethylene Pipe, 300- to 1500-mm (12- to 60-in.) Diameter.” American Association of State Highway and Transportation Officials, 2018.

[4] M. A. Spalding and A. Chatterjee, “Handbook of Industrial Polyethylene and Technology: Definitive Guide to Manufacturing, Properties, Processing, Applications and Markets set.” John Wiley & Sons, 2017.

[5] J. M. Normann, R. J. Houghtalen, and W. J. Johnston, “Hydraulic Design of Highway Culverts,” 2001.

[6] FHWA, “Broadens Pipe Choices | Civil + Structural Engineer Magazine,” 2006. https://csengineermag.com/fhwa-broadens-pipe-choices/ (accessed Nov. 17, 2019).

[7] K. Haas, “Lifecycle Cost and Performance of Plastic Pipelines in Modern Water Infrastructure,” *University of California, Davis, USA*, 2012.

[8] Y. G. Hsuan and R. M. Koerner, “Antioxidant Depletion Lifetime in High Density Polyethylene Geomembranes,” *Journal of Geotechnical and Geoenvironmental Engineering*, vol. 124, no. 6, pp. 532–541, Jun. 1998, doi: 10.1061/(ASCE)1090-0241(1998)124:6(532).

[9] W. Mueller and I. Jakob, “Oxidative Resistance of High-density Polyethylene Geomembranes,” *Polymer Degradation and Stability*, vol. 79, no. 1, pp. 161–172, Jan. 2003, doi: 10.1016/S0141-3910(02)00269-0.

[10] Y. G. Hsuan and T. McGrath, “HDPE pipe: Recommended Material Specifications and Design Requirements. NCHRP report 429,” *Transportation Research Board, National Research Council, Washington, DC*, 1999.

[11] M. Pluimer, “Establishing 100-year Service Life for Corrugated HDPE Drainage Pipe,” in *Pipelines 2006: Service to the Owner*, 2006, pp. 1–8.

[12] Mulder J. Dreiling G. Meijering, T. and H. Vogt, “100 Year Service Life of Polypropylene And Polyethylene Gravity Sewer Pipes,” *Technical report, TEPPFA Project in cooperation with Borealis and Lyondell Basell*, 2014. https://plasticpipe.org/pdf/tepffa-european-plastic-pipe-fittings-association.pdf (accessed Nov. 13, 2019).

[13] P. Fleming, E. Faragher, and C. Rogers, “Laboratory and Field Testing of Large-diameter Plastic Pipe,” *Transportation research record*, vol. 1594, no. 1, pp. 208–216, 1997.

[14] E. Faragher, P. R. Fleming, and C. D. F. Rogers, “Analysis of Repeated-Load Field Testing of Buried Plastic Pipes,” *Journal of Transportation Engineering*, vol. 126, no. 3, pp. 271–277, May 2000, doi: 10.1061/(ASCE)0733-947X(2000)126:3(271).

[15] T. McGrath and B. Schafer, “Parallel Plate Testing and Simulation of Corrugated Plastic Pipe,” 2003.

[16] R. P. Burgon, S. L. Folkman, and A. P. Moser, “Comparison of Measured and Computed Stiffness of High-Density Polyethylene Pipe,” *Transportation Research Record*, vol. 1976, no. 1, pp. 162–171, Jan. 2006, doi: 10.1177/0361198106197600118.

[17] T. Masada, “Improved Solution for Pipe Stiffness as Measured by Parallel-Plate Load Test Method,” in *Pipelines 2006*, Chicago, Illinois, United States, Jul. 2006, pp. 1–9. doi: 10.1061/40854(211)79.

[18] Y. G. Hsuan and T. McGrath, “Protocol for Predicting Long-term Service of Corrugated High Density Polyethylene Pipes,” *Florida Department of Transportation*, vol. 92, 2005.

[19] BNQ 3624-120, “Tuyaux à Profil Ouvert et à Paroi Intérieure Lisse en Polyéthylène (PE) et Raccords en Polyéthylène (PE) pour les Égouts Pluviaux, les Ponceaux et le Drainage des Sols.” Bureau de Normalisation du Québec, 2016.

[20] ASTM D638, “Standard Test Methods for Tensile Properties of Plastics.” ASTM International, West Conshohocken, PA, USA, 2014.

[21] ASTM D6992, “Standard Test Method for Accelerated Tensile Creep and Creep-Rupture of Geosynthetic Materials Based on Time-Temperature Superposition Using the Stepped Isothermal Method.” ASTM International West Conshohock-en, PA, USA, 2016.

[22] ASTM F2136, “Standard Test Method for Notched, Constant Ligament-Stress (NCLS) Test to Determine Slow-Crack-Growth Resistance of HDPE Resins or HDPE Corrugated Pipe.” ASTM International West Conshohock-en, PA, USA, 2018.

[23] ASTM F1473, “Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins.” ASTM International West Conshohock-en, PA, USA, 2018.

[24] J. Zhang, “Experimental Study of Stress Cracking in High Density Polyethylene Pipes,” PhD Thesis, Drexel University, 2005.

[25] FM 5-573, “Florida Method of test for Predicting the Crack Free Service Life of HDPE corrugated pipes.” Florida department of transportation, 2008.

[26] ASTM D2240, “Standard Test Method for Rubber Property—Durometer Hardness.” ASTM International, West Conshohocken, PA, USA, 2015.

[27] ASTM D618, “Standard Practice for Conditioning Plastics for Testing.” ASTM International, West Conshohocken, PA, USA, 2013.

[28] ASTM D3895, “Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry.” ASTM International West Conshohock-en, PA, USA, 2019.

[29] ASTM D3350, “Standard Specification for Polyethylene Plastics Pipe and Fittings Materials.” ASTM International, West Conshohocken, PA, USA, 2014.

[30] R. W. Thomas and D. Cuttino, “Performance of Corrugated Pipe Manufactured with Recycled Polyethylene Content.” Washington, D.C: Transportation Research Board, 2011.

[31] J. B. Goddard, T. Walsh, and S. W. Dean, “A Brief History of the Development and Growth of the Corrugated Polyethylene Pipe Industry in North America,” *J. ASTM Int.*, vol. 8, no. 6, p. 102693, 2011, doi: 10.1520/JAI102693.

[32] P. pipe institute PPI Handbook, “Chapter 1 - Introduction,” p. 9, 2008.

[33] P. pipe institute PPI Handbook, “Chapter 3 - Material Properties,” p. 61, 2008.

[34] L. J. Petroff, “Occasional and Recurring Surge Design Considerations for HDPE Pipe,” in *Pipelines 2013*, Fort Worth, Texas, United States, Jun. 2013, pp. 161–170. doi: 10.1061/9780784413012.014.

[35] C. G. Rubeiz, “Case Studies on the Use of HDPE Pipe for Municipal and Industrial Projects in North America,” in *Pipeline Engineering and Construction*, San Diego, California, United States, Jul. 2004, pp. 1–10. doi: 10.1061/40745(146)22.

[36] R. Ortega, C. Klopfenstein, and A. Morris, “HDPE, An Alternative with Limitations; Houston’s Experience,” in *Pipeline Engineering and Construction*, San Diego, California, United States, Jul. 2004, pp. 1–10. doi: 10.1061/40745(146)39.

[37] S. Stuart, “Evaluation of HDPE and PVC Pipes Used for Cross-drains in Highway Construction,” PhD Thesis, Auburn University, 2011.

[38] R. K. Watkins, R. C. Reeve, and J. B. Goddard, “Effect of Heavy Loads on Buried Corrugated Polyethylene pipe,” no. 903. 1983.

[39] N. Hashash and E. T. Selig, “Analysis of the Performance of a Buried High density Polyethylene Pipe,” *Structural performance of flexible pipes*, pp. 95–103, 1990.

[40] A. P. Moser, “The Structural Performance of Buried 48-inch diameter N-12 HC Polyethylene Pipes,” *Buried Structural Laboratory, Utah State University, Logan, Utah*, 1994.

[41] J. B. Goddard, “Tech Report 4.103: Plastic Pipe Design,” 1992.

[42] S. Nazar, “Performance of Plastic Drainage Pipe,” *ORF Contract*, no. 68–10830, 1988.

[43] M. Alzerreca, M. Paris, O. Boyron, D. Orditz, G. Louarn, and O. Correc, “Mechanical Properties and Molecular Structures of Virgin and Recycled HDPE Polymers used in Gravity Sewer Systems,” *Polymer Testing*, vol. 46, pp. 1–8, Sep. 2015, doi: 10.1016/j.polymertesting.2015.06.012.

[44] M. Pluimer, L. McCarthy, A. Welker, and E. Musselman, “Evaluation of Corrugated HDPE Pipes Manufactured with Recycled Content underneath Railroads,” in *Pipelines 2015*, Baltimore, Maryland, Aug. 2015, pp. 553–563. doi: 10.1061/9780784479360.051.

[45] M. L. Pluimer, “Evaluation of Corrugated HDPE pipes Manufactured with Recycled content in Commuter Rail Applications.” Villanova University, 2016.

[46] R. W. Thomas, “Performance of Corrugated pipe Manufactured with Recycled Polyethylene Content,” vol. 696. *Transportation Research Board*, 2011.

[47] M. Pluimer*,* Joel Sprague, Richard Thomas, Leslie McCarthy, Andrea Welker, Shad Sargand, Ehab Shaheen, and Kevin White., “Field Performance of Corrugated Pipe Manufactured with Recycled Polyethylene Content.” Washington, D.C.: *Transportation Research Board*, 2018, p. 24934. doi: 10.17226/24934.

[48] MTO, “MTO Gravity Pipe Design Guidelines: Circular Culverts and Stormsewers,” *Ontario Ministry of Transportation Library*, 2014. https://www.library.mto.gov.on.ca/SydneyPLUS/Sydney/Portal/default.aspx?component=AAAAIY&record=84d90004-0ccc-4fdf-aad7-c8a01b2e8591 (accessed Nov. 19, 2020).

[49] ASTM F1675, “Standard Practice for Life-cycle Cost Analysis of Plastic Pipe used for Culverts, Storm Sewers, and other Buried Conduits.” ASTM International, West Conshohocken, PA, USA, 1996.

[50] J. O. Hurd, “Service life Model Verification for Concrete Pipe Culverts in Ohio,” *Transportation Research Record*, vol. 1191, pp. 118–131, 1988.

[51] F. C. Hadipriono, “Durability of Concrete Pipe Culverts: Service Life Assessment,” *The Ohio State University, Columbus, Ohio*, 1986.

[52] J. C. Potter, “Life Cycle Cost for Drainage Structures,” ARMY ENGINEER WATERWAYS EXPERIMENT STATION VICKSBURG MS GEOTECHNICAL LAB, 1988.

[53] R. K. Rowe and H. P. Sangam, “Durability of HDPE Geomembranes,” *Geotextiles and Geomembranes*, vol. 20, no. 2, pp. 77–95, 2002.

[54] T. J. McGrath and S. Gumpertz, “Protocol for 100-year Service Life of Corrugated HDPE,” *Project 030159. Rep. Prepared for the Florida Department of Transportation*, 2003.

[55] Y. G. Hsuan and A. Civil, “Protocol for 100 years Service Life of Corrugated High Density Polyethylene Pipes. Part II: Stress Crack Resistance, Oxidation Resistance and Viscoelastic Properties of Finished Corrugated Pipes,” *Rep. to the Florida Dept. of Transportation, Florida Dept. of Transportation, Tallahassee, FL*, 2003.

[56] ASTM F894, “Standard Specification for Polyethylene (PE) Large Diameter Profile Wall Sewer and Drain Pipe.” ASTM International, West Conshohocken, PA, USA, 2019.

[57] CSA B182.6, “Profile Polyethylene Sewer Pipe and Fittings for Leak-proof Sewer Applications.” Canadian Standards Association, 2011.

[58] CSA B182.8, “Profile Polyethylene Storm Sewer and Drainage Pipe and Fittings.” Canadian Standards Association, 2018.

[59] H. Blom, J. Teh, and A. Rudin, “PP/PE blends. IV. Characterization and Compatibilization of Blends of Postconsumer Resin with Virgin PP and HDPE,” *Journal of Applied Polymer Science*, vol. 70, no. 11, pp. 2081–2095, 1998.

[60] A. Dormer, D. P. Finn, P. Ward, and J. Cullen, “Carbon Footprint Analysis in Plastics Manufacturing,” *Journal of Cleaner Production*, vol. 51, pp. 133–141, Jul. 2013, doi: 10.1016/j.jclepro.2013.01.014.

[61] Franklin-Associates, “Life Cycle Impacts for Postconsumer Recycled Resins: PET, HDPE, and PP,” Association of Plastic Recyclers (APR), 2018.

[62] M. R. Korhonen and H. Dahlbo, “Reducing Greenhouse Gas Emissions by Recycling Plastics and Textiles into Products,” Finnish Environment Institute, Helsinki, Finland, 2007.

[63] F. Welle, “Post-consumer Contamination in High-density Polyethylene (HDPE) Milk Bottles and the Design of a bottle-to-bottle Recycling Process,” *Food Additives and Contaminants*, vol. 22, no. 10, pp. 999–1011, Oct. 2005, doi: 10.1080/02652030500157742.

[64] E. T. Shaheen, “Long Term Performance of Corrugated HDPE Pipes Produced with Post-consumer Recycled Materials under Constant Deflection,” Ohio University, 2018.

[65] ASTM F2306/F2306M, “Standard Specification for 12 to 60 in. [300 to 1500 mm] Annular Corrugated Profile-Wall Polyethylene (PE) Pipe and Fittings for Gravity-Flow Storm Sewer and Subsurface Drainage Applications.” ASTM International West Conshohock-en, PA, USA, 2019.

[66] G. W. Ring, “Culvert durability: where are we?” no. 1001. 1984.

[67] J. Q. Zhao *et al.*, “Durability and Performance of Gravity Pipes: State-of-the-art Literature Review.” Institute for Research in Construction, 1998. [Online]. Available: https://nrc-publications.canada.ca/eng/view/accepted/?id=3f56015a-909e-45fb-9b8a-c8e536b96d54

[68] L. Gabriel, “H., Eric T. Moran, 1998. NCHRP Synthesis 254. Service life of Drainage Pipe. A Synthesis of Highway Practice,” *Transportation Research Board. Notional Research Council. National Cooperative Highway Research Program. National Academy press, Washington, DC*, 1998.

[69] A. Chudnovsky, Z. Zhou, H. Zhang, and K. Sehanobish, “Lifetime Assessment of Engineering Thermoplastics,” *International Journal of Engineering Science*, vol. 59, pp. 108–139, Oct. 2012, doi: 10.1016/j.ijengsci.2012.03.016.

[70] J. A. Alvarado Contreras, “Micromechanical Modelling of Polyethylene,” PhD Thesis, University of Waterloo, 2007.

[71] J. J. Cheng, “Mechanical and Chemical Properties of HDPE: Effects of Microstructure on Creep Characteristics,” PhD Thesis, University of Waterloo, 2008.

[72] ISO 9080, “Plastics Piping and Ducting Systems — Determination of the Long-term Hydrostatic Strength of Thermoplastics Materials in Pipe form by Extrapolation.” International Organization for Standardization., 2012.

[73] ASTM D2837-13e1, “Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials or Pressure Design Basis for Thermoplastic Pipe Products.” ASTM International West Conshohock-en, PA, USA, 2013.

[74] A. Chudnovsky, “Slow Crack Growth, Its modeling and Crack-layer Approach: A review,” *International Journal of Engineering Science*, vol. 83, pp. 6–41, Oct. 2014, doi: 10.1016/j.ijengsci.2014.05.015.

[75] B.-H. Choi, Z. Zhou, A. Chudnovsky, S. S. Stivala, K. Sehanobish, and C. P. Bosnyak, “Fracture Initiation Associated with Chemical Degradation: Observation and Modeling,” *International Journal of Solids and Structures*, vol. 42, no. 2, pp. 681–695, Jan. 2005, doi: 10.1016/j.ijsolstr.2004.06.028.

[76] L. Kurelec, M. Teeuwen, H. Schoffeleers, and R. Deblieck, “Strain Hardening Modulus as a Measure of Environmental Stress Crack Resistance of High-Density Polyethylene,” *Polymer*, vol. 46, no. 17, pp. 6369–6379, Aug. 2005, doi: 10.1016/j.polymer.2005.05.061.

[77] A. Lustiger and R. L. Markham, “Importance of Tie Molecules in Preventing Polyethylene Fracture under Long-term Loading Conditions,” *Polymer*, vol. 24, no. 12, pp. 1647–1654, Dec. 1983, doi: 10.1016/0032-3861(83)90187-8.

[78] ASTM D1693-15e1, “Standard Test Method for Environmental Stress-Cracking of Ethylene Plastics.” ASTM International West Conshohock-en, PA, USA, 2015.

[79] W.-K. Wong and Y. G. Hsuan, “Interaction between Carbon Black and Antioxidants in High-density Polyethylene Pipe Resin,” *Transportation Research Record*, vol. 2310, no. 1, pp. 137–144, 2012.

[80] T. J. McGrath, I. D. Moore, and Y. G. Hsuan, “Updated Test and Design Methods for Thermoplastic Drainage pipe,” vol. 631. *Transportation Research Board*, 2009.

[81] K. Chaoui, R. Khelif, N. Zeghib, and A. Chateauneuf, “Failure Analysis of Polyethylene Gas Pipes,” in *Safety, reliability and risks associated with water, oil and gas pipelines*, Springer, 2008, pp. 131–163.

[82] B.-H. Choi, Z. Zhou, and A. Chudnovsky, “Modeling of Stress Corrosion Cracking in Plastic Pipes,” in *Pipelines 2008*, Atlanta, Georgia, United States, Jul. 2008, pp. 1–10. doi: 10.1061/40994(321)65.

[83] K. Oliphant, M. Conrad, and W. Bryce, “Fatigue of Plastic Water Pipe: A Technical Review with Recommendations for PE4710 Pipe Design Fatigue,” in *Pipelines 2012*, Miami Beach, Florida, Aug. 2012, pp. 38–60. doi: 10.1061/9780784412480.005.

[84] Y.-Q. Zhou and N. Brown, “The Mechanism of Fatigue Failure in a Polyethylene Copolymer,” *J. Polym. Sci. B Polym. Phys.*, vol. 30, no. 5, pp. 477–487, Apr. 1992, doi: 10.1002/polb.1992.090300507.

[85] Y. Zhou and N. Brown, “Evaluating the Fatigue Resistance of Notched Specimens of Polyethylene,” *Polym. Eng. Sci.*, vol. 33, no. 21, pp. 1421–1425, Nov. 1993, doi: 10.1002/pen.760332107.

[86] M. Parsons, E. V. Stepanov, A. Hiltner, and E. Baer, “Correlation of Stepwise Fatigue and Creep Slow Crack Growth in HDPE.,” *Journal of Materials Science*, vol. 34, no. 14, pp. 3315–3326, 1999.

[87] Divyashree, “An Investigation of Durability and Reliability of HDPE Pipe for Large Diameter Water Transmission Applications,” PhD Thesis, University of Texas, Arlington, TX, USA, 2014.

[88] S.-S. Yeo and Y. G. Hsuan, “Evaluation of Creep Behavior of High Density Polyethylene and Polyethylene-terephthalate Geogrids,” *Geotextiles and Geomembranes*, vol. 28, no. 5, pp. 409–421, Oct. 2010, doi: 10.1016/j.geotexmem.2009.12.003.

[89] S. S. Yeo, “Evaluation of Creep Behavior of Geosynthetics Using Accelerated and Conventional Methods,” PhD Thesis, Drexel University, Philadelphia, PA, USA, 2007.

[90] I. P. Giannopoulos and C. J. Burgoyne, “Prediction of the Long-term Behaviour of High Modulus Fibres using the Stepped Isostress Method (SSM),” *J Mater Sci*, vol. 46, no. 24, pp. 7660–7671, Dec. 2011, doi: 10.1007/s10853-011-5743-x.

[91] C. Domínguez, R. A. García, M. Aroca, and A. Carrero, “Study of the PENT Test Conditions for Reducing Failure Times in High-resistance Polyethylene Resins for Pipe Applications,” *Mech Time-Depend Mater*, vol. 16, no. 1, pp. 105–115, Feb. 2012, doi: 10.1007/s11043-011-9151-z.

[92] Y. G. Hsuan and J. Zhang, “Stress Crack Resistance of Corrugated High-Density Polyethylene Pipes in Different Test Environments and Temperatures,” *Transportation Research Record*, vol. 1928, no. 1, pp. 220–225, Jan. 2005, doi: 10.1177/0361198105192800123.

[93] M. Schmid, A. Ritter, and S. Affolter, “Determination of Oxidation Induction Time and Temperature bY DSC: Results of Round Robin Tests,” *J Therm Anal Calorim*, vol. 83, no. 2, pp. 367–371, Feb. 2006, doi: 10.1007/s10973-005-7142-5.

[94] J. Dealy and D. Plazek, “Time-temperature Superposition—A Users Guide,” *Rheol. Bull*, vol. 78, no. 2, pp. 16–31, 2009.

[95] K. Alwis and C. J. Burgoyne, “Time-temperature Superposition to determine the Stress-Rupture of Aramid Fibres,” *Applied Composite Materials*, vol. 13, no. 4, pp. 249–264, 2006.

[96] C. H. Popelar, V. H. Kenner, and J. P. Wooster, “An Accelerated Method for establishing the Long-term Performance of Polyethylene Gas Pipe Materials,” *Polym. Eng. Sci.*, vol. 31, no. 24, pp. 1693–1700, Dec. 1991, doi: 10.1002/pen.760312402.

[97] FM 5-577, “Florida Method of Test for predicting the Long-term Modulus of HDPE Corrugated Pipes.” Florida department of transportation, 2008.

[98] ASTM D2412-11, “Standard Test Method for Determination of External Loading Characteristics of Plastic Pipe by Parallel-Plate Loading.” ASTM International West Conshohock-en, PA, USA, 2018.

[99] J. S. Thornton, J. N. Paulson, and D. Sandri, “Conventional and Stepped Isothermal Methods for characterizing Long term Creep Strength of Polyester Geogrids,” in *Sixth International Conference on Geosynthetics, Atlanta, USA*, 1998, pp. 691–698.

[100] J. H. Greenwood and W. Voskamp, “Predicting the Long-term Strength of a Geogrid using the Stepped Isothermal Method,” in *Eurogeo 2000: proceedings of the 2nd european geosynthetics conference. volume 1: mercer lecture, keynote lectures, geotechnical applications*, 2000, vol. 1.

[101] K. G. N. C. Alwis and C. J. Burgoyne, “Accelerated Creep Testing for Aramid Fibres using the Stepped Isothermal Method,” *J Mater Sci*, vol. 43, no. 14, pp. 4789–4800, Jul. 2008, doi: 10.1007/s10853-008-2676-0.

[102] J. D. Ferry, “Viscoelastic properties of polymers.” NY: John Wiley and Sons, 1980.

[103] Y. G. Hsuan, “Phase II of Long-term Properties of Corrugated HDPE Pipes,” Florida Department of Transportation Materials Research Lab, Drexel University, Philadelphia, Pennsylvania, USA, Final Report, 2012.

[104] F. Achereiner, K. Engelsing, M. Bastian, and P. Heidemeyer, “Accelerated Creep testing of Polymers using the Stepped Isothermal Method,” *Polymer Testing*, vol. 32, no. 3, pp. 447–454, May 2013, doi: 10.1016/j.polymertesting.2013.01.014.

[105] ASTM D5262-07, “Standard Test Method for Evaluating the Unconfined Tension Creep and Creep Rupture Behavior of Geosynthetics.” ASTM International West Conshohock-en, PA, USA, 2016.

[106] FM 5-572, “Florida Method of test for determining Stress Crack Resistance of HDPE Corrugated Pipes.” Florida department of transportation, 2008.

[107] ASTM F3181, “Standard Test Method for The Un-notched, Constant Ligament Stress Crack Test (UCLS) for HDPE Materials Containing Post- Consumer Recycled HDPE.” ASTM International West Conshohock-en, PA, USA, 2016.

[108] M. Robert, P. Wang, P. Cousin, and B. Benmokrane, “Temperature as an Accelerating Factor for Long-Term Durability Testing of FRPs: Should There Be Any Limitations?,” *J. Compos. Constr.*, vol. 14, no. 4, pp. 361–367, Aug. 2010, doi: 10.1061/(ASCE)CC.1943-5614.0000102.

[109] M. Hadid, B. Guerira, M. Bahri, and A. Zouani, “Assessment of the Stepped Isostress Method in the prediction of Long-term Creep of Thermoplastics,” *Polymer Testing*, vol. 34, pp. 113–119, Apr. 2014, doi: 10.1016/j.polymertesting.2014.01.003.

[110] A. K. Sahu and K. Sudhakar, “Effect of UV exposure on Bimodal HDPE Floats for Floating Solar Application,” *Journal of Materials Research and Technology*, vol. 8, no. 1, pp. 147–156, Jan. 2019, doi: 10.1016/j.jmrt.2017.10.002.

[111] A. K. Sahu, K. Sudhakar, and R. M. Sarviya, “U.V light Effect on the Mechanical Behaviour of HDPE/Carbon black Composites,” *IOP Conf. Ser.: Mater. Sci. Eng.*, vol. 788, p. 012054, Jun. 2020, doi: 10.1088/1757-899X/788/1/012054.

[112] P. pipe institute PPI, “High-Density Polyethylene Pipe Systems,” 2015. https://plasticpipe.org/pdf/high_density_polyethylene_pipe_systems.pdf

[113] D. J. Lohse, “POLYOLEFINS,” in *Applied Polymer Science: 21st Century*, Elsevier, 2000, pp. 73–91. doi: 10.1016/B978-008043417-9/50008-8.

[114] J. Kurdziel and M. Pluimer, “Engineering and Testing Requirements for Infrastructure Pipeline Applications utilizing HDPE Recycled Materials. Plastic Pipes Conference association PPCA,” 2018. https://www.pe100plus.com/PPCA/Engineering-and-testing-requirements-for-infrastructure-pipeline-applications-utilizing-HDPE-recycled-materials-p1716.html (accessed Jun. 20, 2020).

[115] S. Rajendran, A. Hodzic, C. Soutis, and A. MariamAl-Maadeed, “Review of Life Cycle Assessment on Polyolefins and Related Materials,” *Plastics, Rubber and Composites*, vol. 41, no. 4–5, pp. 159–168, Jun. 2012, doi: 10.1179/1743289811Y.0000000051.

[116] K. Q. Nguyen, C. Mwiseneza, K. Mohamed, P. Cousin, M. Robert, and B. Benmokrane, “Long-term Testing Methods for HDPE Pipe - Advantages and Disadvantages: A review,” *Engineering Fracture Mechanics*, vol. 246, p. 107629, Apr. 2021, doi: 10.1016/j.engfracmech.2021.107629.

[117] ASTM D1248, “Standard Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable.” ASTM International, West Conshohocken, PA, USA, 2016.

[118] ASTM D1505, “Standard Test Method for Density of Plastics by the Density-Gradient Technique.” ASTM International, West Conshohocken, PA, USA, 2018.

[119] ASTM D792, “Standard test methods for density and specific gravity (relative density) of plastics by displacement.” ASTM International, West Conshohocken, PA, USA, 2013.

[120] ASTM D4883, “Standard Test Method for Density of Polyethylene by the Ultrasound Technique.” ASTM International, West Conshohocken, PA, USA, 2018.

[121] ASTM D1238, “Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer.” ASTM International, West Conshohocken, PA, USA, 2013.

[122] ASTM D5805-00, “Standard Test Methods for Rubber - Determination of Carbon Black in Masterbatches.” ASTM International, West Conshohocken, PA, USA, 2019.

[123] A. Pusz and K. Michalik, “Examining the Hardness of the High-density Polyethylene with method of the Cone,” *Archives of Materials Science and Engineering*, vol. 28, no. 8, pp. 467–470, 2007.

[124] X. Wen *et al.*, “Effect of Nanosized Carbon Black on Thermal Stability and Flame Retardancy of Polypropylene/carbon Nanotubes Nanocomposites: Polypropylene/carbon nanotubes/carbon black Nanocomposites,” *Polym. Adv. Technol.*, vol. 24, no. 11, pp. 971–977, Nov. 2013, doi: 10.1002/pat.3172.

[125] H. Yang *et al.*, “Effect of Carbon Black on improving Thermal Stability, Flame Retardancy and Electrical Conductivity of Polypropylene/carbon Fiber Composites,” *Composites Science and Technology*, vol. 113, pp. 31–37, Jun. 2015, doi: 10.1016/j.compscitech.2015.03.013.

[126] ASTM E2550, “Standard Test Methods for Thermal Stability by Thermogravimetry.” ASTM International, West Conshohocken, PA, USA, 2017.

[127] N. Dusunceli and O. U. Colak, “Modelling Effects of Degree of Crystallinity on Mechanical behavior of Semicrystalline Polymers,” *International Journal of Plasticity*, vol. 24, no. 7, pp. 1224–1242, Jul. 2008, doi: 10.1016/j.ijplas.2007.09.003.

[128] K. Balani, V. Verma, A. Agarwal, and R. Narayan, “Physical, Thermal, and Mechanical Properties of Polymers,” in *Biosurfaces*, John Wiley & Sons, Ltd, 2014, pp. 329–344. doi: 10.1002/9781118950623.app1.

[129] C. Aumnate, N. Rudolph, and M. Sarmadi, “Recycling of Polypropylene/Polyethylene Blends: Effect of Chain Structure on the Crystallization Behaviors,” *Polymers*, vol. 11, no. 9, p. 1456, Sep. 2019, doi: 10.3390/polym11091456.

[130] P. L. Ramkumar, D. M. Kulkarni, V. V. R. Abhijit, and A. Cherukumudi, “Investigation of Melt Flow Index and Impact Strength of Foamed LLDPE for Rotational Moulding Process,” *Procedia Materials Science*, vol. 6, pp. 361–367, 2014, doi: 10.1016/j.mspro.2014.07.046.

[131] M. S. Abbas-Abadi, M. N. Haghighi, and H. Yeganeh, “Effect of the Melt Flow Index and Melt Flow Rate on the Thermal Degradation Kinetics of Commercial Polyolefins,” *J. Appl. Polym. Sci.*, vol. 126, no. 5, pp. 1739–1745, Dec. 2012, doi: 10.1002/app.36775.

[132] E. Jakab and M. Omastová, “Thermal Decomposition of Polyolefin/carbon black Composites,” *Journal of Analytical and Applied Pyrolysis*, vol. 74, no. 1–2, pp. 204–214, Aug. 2005, doi: 10.1016/j.jaap.2005.02.001.

[133] S. Deveci, N. Antony, and B. Eryigit, “Effect of Carbon Black Distribution on the Properties of Polyethylene Pipes - Part 1: Degradation of Post Yield Mechanical Properties and Fracture Surface Analyses,” *Polymer Degradation and Stability*, vol. 148, pp. 75–85, Feb. 2018, doi: 10.1016/j.polymdegradstab.2018.01.011.

[134] ASTM D4218, “Standard Test Method for Determination of Carbon Black Content in Polyethylene Compounds by the Muffle-Furnace Technique.” ASTM International, West Conshohocken, PA, USA, 2015.

[135] I. Turku, T. Kärki, and A. Puurtinen, “Durability of Wood Plastic Composites Manufactured from Recycled Plastic,” *Heliyon*, vol. 4, no. 3, p. e00559, Mar. 2018, doi: 10.1016/j.heliyon.2018.e00559.

[136] J.-C. Huang, “Carbon Black Filled Conducting Polymers and Polymer Blends,” *Adv. Polym. Technol.*, vol. 21, no. 4, pp. 299–313, 2002, doi: 10.1002/adv.10025.

[137] G. Faraca and T. Astrup, “Plastic Waste from Recycling Centres: Characterisation and Evaluation of Plastic Recyclability,” *Waste Management*, vol. 95, pp. 388–398, Jul. 2019, doi: 10.1016/j.wasman.2019.06.038.

[138] J. Murphy, “Additives for Plastics Handbook.” Elsevier, 2001.

[139] L. F. Kadhim, “Mechanical Properties of High-Density Polyethylene/Chromium Trioxide under Ultraviolet Rays,” *International Journal of Applied Engineering Research*, vol. 12, no. 10, pp. 2517–2526, 2017.

[140] A. Zee, “Quantum field theory in a nutshell,” 2nd ed. Princeton, N.J: Princeton University Press, 2010.

[141] G. Kalay, R. A. Sousa, R. L. Reis, A. M. Cunha, and M. J. Bevis, “The Enhancement of the Mechanical Properties of a High-density Polyethylene,” *J. Appl. Polym. Sci.*, vol. 73, no. 12, pp. 2473–2483, Sep. 1999, doi: 10.1002/(SICI)1097-4628(19990919)73:12<2473::AID-APP16>3.0.CO;2-O.

[142] N. Dusunceli and B. Aydemir, “The Effects of Loading History and Manufacturing Methods on the Mechanical Behavior of High-Density Polyethylene,” *Journal of Elastomers & Plastics*, vol. 43, no. 5, pp. 451–468, Sep. 2011, doi: 10.1177/0095244311404181.

[143] S. L. Gassman, A. J. Schroeder, and R. P. Ray, “Field Performance of High Density Polyethylene Culvert Pipe,” *J. Transp. Eng.*, vol. 131, no. 2, pp. 160–167, Feb. 2005, doi: 10.1061/(ASCE)0733-947X(2005)131:2(160).

[144] F. S. Qureshi, S. H. Hamid, A. G. Maadhah, and M. B. Amin, “Weather-Induced Degradation of Plastic Pipes,” *Polymer-Plastics Technology and Engineering*, vol. 28, no. 7–8, pp. 663–670, Sep. 1989, doi: 10.1080/03602558908049821.

[145] Jan. F. Rabek, “Physical Aspects of the Photodegradation of Polymers,” in *Polymer Photodegradation*, Dordrecht: Springer Netherlands, 1995, pp. 1–23. doi: 10.1007/978-94-011-1274-1_1.

[146] A. F. C. Becerra and J. R. M. d’Almeida, “UV Effects on the Tensile and Creep Behaviour of HDPE,” *Polymers and Polymer Composites*, vol. 25, no. 5, pp. 327–332, Jun. 2017, doi: 10.1177/096739111702500502.

[147] P. K. Roy, P. Surekha, R. Raman, and C. Rajagopal, “Investigating the Role of Metal Oxidation State on the Degradation behaviour of LDPE,” *Polymer Degradation and Stability*, vol. 94, no. 7, pp. 1033–1039, Jul. 2009, doi: 10.1016/j.polymdegradstab.2009.04.025.

[148] M. Koutny, J. Lemaire, and A.-M. Delort, “Biodegradation of Polyethylene Films with Prooxidant Additives,” *Chemosphere*, vol. 64, no. 8, pp. 1243–1252, Aug. 2006, doi: 10.1016/j.chemosphere.2005.12.060.

[149] J. Attwood, M. Philip, A. Hulme, G. Williams, and P. Shipton, “The Effects of Ageing by Ultraviolet Degradation of Recycled Polyolefin Blends,” *Polymer Degradation and Stability*, vol. 91, no. 12, pp. 3407–3415, Dec. 2006, doi: 10.1016/j.polymdegradstab.2006.04.025.

[150] R. Maria *et al.*, “Monitoring the Influence of Different Weathering Conditions on Polyethylene Pipes by IR-microscopy,” *Polymer Degradation and Stability*, vol. 96, no. 10, pp. 1901–1910, Oct. 2011, doi: 10.1016/j.polymdegradstab.2011.07.004.

[151] K. A. Jassim, W. H. Jassim, and S. H. Mahdi, “The Effect of Sunlight on Medium Density Polyethylene Water Pipes,” *Energy Procedia*, vol. 119, pp. 650–655, 2017.

[152] T. Jiang, Y. Qi, Y. Wu, and J. Zhang, “Application of Antioxidant and Ultraviolet Absorber into HDPE: Enhanced Resistance to UV Irradiation,” *e-Polymers*, vol. 19, no. 1, pp. 499–510, Oct. 2019, doi: 10.1515/epoly-2019-0053.

[153] ASTM D4329, “Standard Practice for Fluorescent Ultraviolet (UV) Lamp Apparatus Exposure of Plastics.” ASTM International, West Conshohocken, PA, USA, 2013.

[154] Hitachi High-Tech, “Application Note: Thermal Analysis- DSC Measurement of Polyethylene_No_26.” Hitachi High-Tech Science Corporation, 1986. [Online]. Available: https://www.hitachi-hightech.com/file/global/pdf/products/science/appli/ana/thermal/application_TA_026e.pdf

[155] S. M. Khan *et al.*, “2D Carbon Fiber Reinforced High Density Polyethylene Multi-Layered Laminated Composite Panels: Structural, Mechanical, Thermal, and Morphological Profile,” *Journal of Materials Science & Technology*, vol. 32, no. 10, pp. 1077–1082, Oct. 2016, doi: 10.1016/j.jmst.2016.06.011.

[156] J. P. Luongo, “Infrared Study of Oxygenated Groups formed in Polyethylene during Oxidation,” *J. Polym. Sci.*, vol. 42, no. 139, pp. 139–150, Jan. 1960, doi: 10.1002/pol.1960.1204213916.

[157] S. Wang, J. Zhang, L. Liu, F. Yang, and Y. Zhang, “Evaluation of Cooling Property of High-density Polyethylene (HDPE)/titanium Dioxide (TiO2) Composites after Accelerated Ultraviolet (UV) Irradiation,” *Solar Energy Materials and Solar Cells*, vol. 143, pp. 120–127, Dec. 2015, doi: 10.1016/j.solmat.2015.06.032.

[158] E. M. Hoàng and D. Lowe, “Lifetime Prediction of a Blue PE100 Water Pipe,” *Polymer Degradation and Stability*, vol. 93, no. 8, pp. 1496–1503, 2008.

[159] J. Hassinen, “Deterioration of Polyethylene Pipes exposed to Chlorinated Water,” *Polymer Degradation and Stability*, vol. 84, no. 2, pp. 261–267, May 2004, doi: 10.1016/j.polymdegradstab.2003.10.019.

[160] J.-I. Weon, “Effects of Thermal Ageing on Mechanical and Thermal Behaviors of Linear Low Density Polyethylene pipe,” *Polymer Degradation and Stability*, vol. 95, no. 1, pp. 14–20, Jan. 2010, doi: 10.1016/j.polymdegradstab.2009.10.016.

[161] Y. Javadi, M. S. Hosseini, and M. K. R. Aghjeh, “The Effect of Carbon Black and HALS Hybrid Systems on the UV Stability of High-density Polyethylene (HDPE),” *Iranian Polymer Journal*, vol. 23, no. 10, pp. 793–799, 2014.

[162] R. K. Krishnaswamy, “Analysis of Ductile and Brittle Failures from Creep Rupture Testing of High-density Polyethylene (HDPE) Pipes,” *Polymer*, vol. 46, no. 25, pp. 11664–11672, Nov. 2005, doi: 10.1016/j.polymer.2005.09.084.

[163] Y. G. Hsuan, P. Gurian, and J. Zhang, “Factors Affecting Stress Crack Resistance of Corrugated High-Density Polyethylene Pipe,” *Transportation Research Record*, vol. 2028, no. 1, pp. 183–191, Jan. 2007, doi: 10.3141/2028-20.

[164] A. Frank, F. J. Arbeiter, I. J. Berger, P. Hutař, L. Náhlík, and G. Pinter, “Fracture Mechanics Lifetime Prediction of Polyethylene Pipes,” *J. Pipeline Syst. Eng. Pract.*, vol. 10, no. 1, p. 04018030, Feb. 2019, doi: 10.1061/(ASCE)PS.1949-1204.0000356.

[165] E. T. Shaheen, S. M. Sargand, T. Masada, and I. Khoury, “Laboratory Evaluations of Corrugated HDPE Pipes Containing Postconsumer Recycled Material under Constant Deflection Conditions,” *J. Pipeline Syst. Eng. Pract.*, vol. 11, no. 3, p. 04020032, Aug. 2020, doi: 10.1061/(ASCE)PS.1949-1204.0000476.

[166] S. Na, L. Nguyen, S. Spatari, and Y. G. Hsuan, “Effects of Recycled HDPE and Nanoclay on Stress Cracking of HDPE by Correlating J_c with Slow Crack Growth,” *Polym Eng Sci*, vol. 58, no. 9, pp. 1471–1478, Sep. 2018, doi: 10.1002/pen.24691.

[167] A. Frank, G. Pinter, and R. W. Lang, "Prediction of the Remaining Lifetime of Polyethylene Pipes After up to 30 years in Use," *Polymer Testing*, vol. 28, no. 7, pp. 737–745, Oct. 2009, doi: 10.1016/j.polymertesting.2009.06.004.

[168] J. Hessel, "100-years Service-live for Polyethylene Pipes," *3 R INTERNATIONAL*, vol. 46, no. 4, p. 242, 2007.

[169] X. Lu and N. Brown, "A test for Slow Crack Growth Failure in Polyethylene under a Constant Load," *Polymer Testing*, vol. 11, no. 4, pp. 309–319, 1992, doi: 10.1016/0142-9418(92)90025-7.

[170] E. Nezbedová, P. Hutař, M. Zouhar, Z. Knésl, J. Sadílek, and L. Náhlík, "The Applicability of the Pennsylvania Notch Test for a New generation of PE Pipe Grades," *Polymer Testing*, vol. 32, no. 1, pp. 106–114, Feb. 2013, doi: 10.1016/j.polymertesting.2012.09.009.

[171] R. Thomas and D. Cuttino, "The BFF Test: An Un-Notched Stress Crack Test for Evaluating Resin Blends Containing Recycled HDPE for Use in Corrugated Drainage Pipe." 2010.

[172] Y. G. Hsuan, J.-Y. Zhang, and W.-K. Wong, "Evaluation of Long-term Stress Crack Resistance of Corrugated High Density Polyethylene pipes," *Plastics, Rubber and Composites*, vol. 36, no. 5, pp. 201–206, Jun. 2007, doi: 10.1179/174328907X191288.

[173] K. Q. Nguyen, P. Cousin, K. Mohamed, M. Robert, and B. Benmokrane, "Comparing Short-Term Performance of Corrugated HDPE Pipe Made with or without Recycled Resins for Transportation Infrastructure Applications," *J. Mater. Civ. Eng.*, vol. 34, no. 2, p. 04021427, Feb. 2022, doi: 10.1061/(ASCE)MT.1943-5533.0004067.

[174] H. P. Sangam and R. K. Rowe, "Effects of Exposure Conditions on the Depletion of Antioxidants from High-density Polyethylene (HDPE) Geomembranes," *Canadian Geotechnical Journal*, vol. 39, no. 6, pp. 1221–1230, 2002.

[175] R. K. Rowe, S. Rimal, and H. Sangam, "Ageing of HDPE Geomembrane Exposed to Air, Water and Leachate at Different Temperatures," *Geotextiles and Geomembranes*, vol. 27, no. 2, pp. 137–151, 2009.

[176] E. and C. C. Canada, "Canadian Climate Normals - Climate - Environment and Climate Change Canada," Jul. 09, 2021. https://climate.weather.gc.ca/climate_normals/index_e.html (accessed Sep. 01, 2021).

[177] Y. G. Hsuan and R. M. Koerner, "Effects of Outdoor Exposure on a High Density Polyethylene Geomembrane," 1991.

[178] A. L. ROLLIN, J. MLYNAREK, and J. LAFLEUR, "4.7 Performance Changes in Aged In-Situ HDPE Geomembrane," *Landfilling of waste: Barriers*, vol. 2, p. 431, 1993.

[179] R. K. Rowe, Y. G. Hsuan, C. B. Lake, P. Sangam, and S. Usher, "Evaluation of a Composite (geomembrane/clay) Liner for a Lagoon after 14 years of Use," in *Proc. 6th Int. Conf. on Geosynthetics, Atlanta*, 1998, pp. 191–196.

[180] R. K. Rowe, "Long-term Performance of Contaminant Barrier Systems," *Géotechnique*, vol. 55, no. 9, pp. 631–678, Nov. 2005, doi: 10.1680/geot.2005.55.9.631.

[181] Y. G. Hsuan and R. M. Koerner, "Long term Durability of HDPE Geomembrane: Part I—Depletion of Antioxidant," *GRI Report*, vol. 16, p. 35, 1995.

[182] U. Andersson, "Which Factors Control the Lifetime of Plastic Pipes and how the Lifetime can be Extrapolated," *Book-institute of materials*, vol. 759, pp. 311–320, 2001.

[183] R. P. Krushelnitzky and R. W. I. Brachman, "Antioxidant Depletion in High-density Polyethylene Pipes exposed to Synthetic Leachate and Air," *Geosynthetics International*, vol. 18, no. 2, pp. 63–73, Apr. 2011, doi: 10.1680/gein.2011.18.2.63.

[184] L. G. Tisinger and J. P. Giroud, “the Durability of HDPE Membranes,” *Geotechnical Fabrics Report*, vol. 11, no. 6, 1993.

[185] J. J. Fay and R. E. King, “Antioxidants for Geosynthetic Resins and Applications,” in *Geosynthetic resins, formulations and manufacturing. Edited by YG Hsuan and RM Koerner. GRI Conference Series, Industrial Fabrics Association International (IFAI), St Paul, Minn*, 1994, pp. 77–96.

[186] N. Grassie and G. Scott, “Polymer degradation and stabilisation.” CUP Archive, 1988.

[187] Y. G. Hsuan and W.-K. Wong, “Methodology to Evaluate Oxidation Degradation of High-Density Polyethylene Corrugated Pipe Resin,” *Transportation Research Record*, vol. 2172, no. 1, pp. 192–198, 2010.

[188] U. Gedde, J. Viebke, H. Leijström, and M. Ifwarson, “Long-term Properties of Hot-water Polyolefin Pipes—a review,” *Polymer Engineering & Science*, vol. 34, no. 24, pp. 1773–1787, 1994.

[189] A. J. Whelton, A. M. Dietrich, and D. L. Gallagher, “Impact of Chlorinated Water Exposure on Contaminant Transport and Surface and Bulk Properties of High-density Polyethylene and Cross-linked Polyethylene Potable Water Pipes,” *Journal of Environmental Engineering*, vol. 137, no. 7, pp. 559–568, 2011.

[190] H.-Y. Jeon, A. Bouazza, and K.-Y. Lee, “Depletion of Antioxidants from an HDPE Geomembrane upon Exposure to Acidic and Alkaline solutions,” *Polymer Testing*, vol. 27, no. 4, pp. 434–440, Jun. 2008, doi: 10.1016/j.polymertesting.2008.01.003.

[191] R. K. Rowe and S. Rimal, “Depletion of Antioxidants from a HDPE Geomembrane in a Composite Liner,” *J. Geotech. Geoenviron. Eng.*, vol. 134, no. 1, pp. 68–78, Jan. 2008, doi: 10.1061/(ASCE)1090-0241(2008)134:1(68).

[192] R. K. Rowe, M. Z. Islam, R. W. I. Brachman, D. N. Arnepalli, and A. R. Ewais, “Antioxidant Depletion from a High-Density Polyethylene Geomembrane under Simulated Landfill Conditions,” *J. Geotech. Geoenviron. Eng.*, vol. 136, no. 7, pp. 930–939, Jul. 2010, doi: 10.1061/(ASCE)GT.1943-5606.0000302.

[193] R. K. Rowe, M. Z. Islam, and Y. G. Hsuan, “Leachate Chemical Composition Effects on OIT Depletion in an HDPE Geomembrane,” *Geosynthetics International*, vol. 15, no. 2, pp. 136–151, Apr. 2008, doi: 10.1680/gein.2008.15.2.136.

[194] C. Vasile and M. Pascu, “Practical guide to polyethylene.” iSmithers Rapra Publishing, 2005.

[195] W. F. Guthrie, “NIST/SEMATECH e-Handbook of Statistical Methods (NIST Handbook 151).” National Institute of Standards and Technology, 2020. doi: 10.18434/M32189.

[196] H. L. Harter, “Tables of Range and Studentized Range,” *Ann. Math. Statist.*, vol. 31, no. 4, pp. 1122–1147, Dec. 1960, doi: 10.1214/aoms/1177705684.

[197] J. B. Howard, “DTA for Control of Stability in Polyolefin Wire and Cable Compounds,” *Polym. Eng. Sci.*, vol. 13, no. 6, pp. 429–434, Nov. 1973, doi: 10.1002/pen.760130606.

[198] J. Luston, “Physical loss of stabilizers from polymers.” *G. Scott Ed., Development in Polymer Stabilization-2*. Applied Science Publishers, 1986.

[199] M. L. Williams, R. F. Landel, and J. D. Ferry, “The Temperature Dependence of Relaxation Mechanisms in Amorphous Polymers and Other Glass-forming Liquids,” *J. Am. Chem. Soc.*, vol. 77, no. 14, pp. 3701–3707, Jul. 1955, doi: 10.1021/ja01619a008.

[200] R. F. Landel and L. E. Nielsen, “Mechanical properties of polymers and composites.” CRC press, 1993.

[201] N. Zeggai *et al.*, “Analysis of Dynamic Mechanical Properties of Photochemically Crosslinked Poly(isobornylacrylate-co-isobutylacrylate) applying WLF and Havriliak-

Negami Models," *Polymer Testing*, vol. 72, pp. 432–438, Dec. 2018, doi: 10.1016/j.polymertesting.2018.10.038.

[202] D. M. Price, "Thermomechanical, Dynamic Mechanical and Ielectric Methods," in *RSC Paperbacks*, P. Haines, Ed. Cambridge: Royal Society of Chemistry, 2002, pp. 94–128. doi: 10.1039/9781847551764-00094.

[203] K. Q. Nguyen, P. Cousin, K. Mohamed, M. Robert, and B. Benmokrane, "Influence of Wall Thickness on the Thermo-Mechanical Properties of Aging HDPE pipes under Freeze-thaw cycles in Quebec Province, Canada," Art. no. 7000, Nov. 2021, Accessed: Dec. 07, 2021. [Online]. Available: https://yahootechpulse.easychair.org/publications/preprint/cRXH

[204] J. V. Gulmine, P. R. Janissek, H. M. Heise, and L. Akcelrud, "Polyethylene Characterization by FTIR," *Polymer Testing*, vol. 21, no. 5, pp. 557–563, 2002, doi: 10.1016/S0142-9418(01)00124-6.

[205] K. Q. Nguyen, P. Cousin, K. Mohamed, M. Robert, A. El-Safty, and B. Benmokrane, "Effects of Ultraviolet Radiation on Recycled and Virgin HDPE Corrugated Pipes Used in Road Drainage Systems." Dec. 07, 2021. doi: 10.21203/rs.3.rs-978155/v1.

[206] K. Q. Nguyen, K. Mohamed, P. Cousin, M. Robert, and B. Benmokrane, "Stress Crack Resistance of Recycled and Virgin HDPE Corrugated Pipe for Transportation Infrastructure Applications," Art. no. 5488, May 2021, Accessed: Dec. 07, 2021. [Online]. Available: https://login.easychair.org/publications/preprint/BK26