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Study of the Thermal, Mechanical Properties and Lifetime of PVC Polymer

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Master of Science in Physics

By

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بسم الله الرحمن الرحيم

﴿ إِنَّا كُلَّ شَيْءٍ خَلَقْنَٰهُ

بِقَدَرٍ ﴿٤٩﴾ ﴾ القمر: ٤٩

Dedication

This thesis is dedicated

To Prophet
MOHAMMED
Peace Be Upon Him

And
My family

Acknowledgement

Praise be to **ALLAH** for all things

I am so grateful to everybody who has given me the support I needed to complete this work. My deep appreciation is extended to my supervisors Dr. Widad Hamdi Jassim and Dr. Mohammed Abdul-Nebi for their keen involvement and competent guidance in every phase of this work, and to Dr. Awham M. Hamid for her encouragement and support.

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Abstract

In this work, mechanical behavior of PVC product like pipes with different origins (K.S.A and Egypt) and Turkey doors with different manufacturing (Nova and Firat) which are used in Iraqi markets were studied.

The objective of this work was to study the mechanical properties like ultimate tensile strength, Young's modulus, elongation at Max., elongation at break, impact strength, flexural strength and hardness of the pipes and doors.

The experimental results show that K.S.A pipes exhibited maximum ultimate tensile strength and Young's modulus than that the Egypt pipes. However, the Egypt pipes exhibited maximum elongation at Max. and elongation at break when compared with K.S.A pipes.

All the tensile properties of Firat doors are greater than that of Nova doors.

Comparison of the flexural strength of pipes and doors. The pipes have the same flexural strength but the Firat doors have maximum value than Nova doors.

Egypt pipes have a higher impact strength than K.S.A pipes. While the Firat doors have a higher impact strength than Nova doors.

The experimental results of hardness Shore D indicated that K.S.A pipes exhibited maximum hardness number (83.7) than Egypt one, while the Firat and Nova doors have the same values.

In this study, thermal properties of Firat and Nova have been evaluated, firstly by thermal conductivity and second by thermal stability and degradation. The Firat doors have better thermal conductivity than that of Nova doors. From the thermogravimetric analysis at different heating rates (2, 5, 10, 20 and 40 ˚C/min). The Firat doors have a higher thermal stability than Nova one.

The Egypt pipes exhibited more thermal stability than the K.S.A pipes.

From the TGA curves the activation energy of pipes and doors are determined at different conversion levels. At 30% conversion level when the mechanism of degradation would be stable and the value of activation energy of Egypt pipes and Firat doors is (108.35 KJ/mole) when it is (101.39 KJ/mole) for K.S.A pipes and (98.35 KJ/mole) for Nova doors.

Thermogravimetric analysis (TGA) provides a method for accelerating the lifetime testing of polymers so that short-term experiments can be used to predict in use lifetime.

The lifetime is decreased with increase of the temperature, at fixed temperature like 70 ˚C the lifetime of Egypt pipes is (≈100 years) while lifetime of K.S.A pipes is (≈21 years). At the same temperature (70 ˚C) the lifetime of Firat doors is (≈72 years) while the lifetime of Nova doors is (≈21 years).

Thermomechanical analysis (TMA) test showed the glass transition temperature (T_g) and softening point (T_s) for pipes and doors. The K.S.A pipes have value for T_g and T_s higher than that of Egypt pipes, while the Firat and Nova doors have the same values.

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List of abbreviations and Latin symbols

Abbreviation/ Symbol	**Meaning**
PVC	Polyvinyl chloride
UPVC	Unplasticised polyvinyl chloride
VCM	Vinyl chlorine monomer
EDC	Ethylene dichloride
E	Young's modulus
Pa	Pascals
σ_{bend}	Flexural strength
δ	Deflection of the beam
w	Width
L	Length
h	Height
I.S.	Impact strength
U_c	Fracture energy
A	Cross – sectional area
k	Thermal conductivity
Q	Heat flux
UV	Ultra violet
E_a	Activation energy
R	Universal gas constant
γ	Weight of active material
$M_\circ$	Initial mass
M_f	Final mass
β	Rate of heating
TMA	Thermomechanical analysis
TGA	Thermogravimetric analysis

Chapter One

Introduction

1-1 Introduction:

Today designers and engineers readily turn to plastics because they offer combinations of properties not available in any other materials. Plastics offer advantages such as lightness, resilience, resistance to corrosion, colour fastness, transparency, ease of processing, etc. [1].

PVC is one of the three most abundantly produced synthetic polymers. PVC is one of the earliest produced polymers. In 1835, Justus von Liebig and his research student, Victor Regnault, reacted ethylene dichloride with alcoholic potash forming the monomer vinyl chloride. In Germany, Klasse, found that vinyl chloride could be made by the addition of hydrogen chloride to acetylene in a system that could be scaled up for commercial production. PVC was used during First World War as a replacement for corrosion-prone metals, in Second World War; PVC became the material of choice to protest electrical wires for the air force and navy [2, 3].

Over the past 60 years PVC has grown to be one of the most commonly used manmade polymers because of the unrivalled combination of properties that it offers. Its ratio of economic cost to performance makes it immediately more accessible than most other materials and the diversity of ways in which it can be used challenges the imagination, from roofing membranes to credit cards, from children’s toys to pipes water [4].

Crude oil/natural gas and rock salt are the starting products for PVC manufacturing. Ethylene is the result of crude oil in the intermediate stage of naphtha through thermal “cracking”. Chlorine, on the other hand, is produced from rock salt through chloralkali electrolysis [5].

There are two important kinds of PVC [6, 7]:

1− Rigid PVC which is used in fabrication of pipes and plastic plates (sheets), Rigid PVC, also known as U-PVC (the U stands for "unplasticised") is used extensively in building applications such as door frames and pipes.

2- Flexible PVC, which is composed from polymer with the addition of plasticizers. This type of PVC is used in fabrication of films, coating purposes and production of industrial leathers.

The PVC competes in many applications because of its excellent properties like [8]:

1- High electrical insulation.

2- High resistance for abrasion.

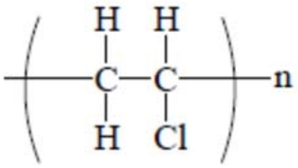
3- Low diffusion for humidity.

4- Good flexibility within range of temperatures.

5- Resistance to water, bases, acids, alcohols, oils, and aliphatic hydrocarbon components (compounds).

The vinyl chloride monomer consists of a carbon-carbon double bond, a pendent chloride atom, and three hydrogen atoms.

Where n is the degree of polymerization, i.e. the number of repeat units in the molecular chain. This monomer polymerizes by the addition (free radical) polymerization method [9].



$$\left(-\overset{H}{\underset{H}{C}}-\overset{H}{\underset{Cl}{C}}- \right)_n$$

1-2 Literature Review:

Roux and Eurin studied the evaluation by mechanical testing of the durability of PVC in (1977). Which were studied by tensile and indentation methods. In order to define the brittle transition in time-temperature space and the change in the brittle transition by indentation methods after artificial weathering of the PVC [10].

In (1979) Soiway, et al., studied the mechanical properties of rigid PVC and the effect of mineral fillers, to improve the resistance of PVC to brittle fracture. On the other hand, they can make crack initiation easier [11].

In (1981) Darwish et al., studied the applicability of linear elastic fracture mechanics (LEFM) to rigid PVC pipe materials. They found the LEFM-controlled as temperature decreases and displacement rate increases and the fracture toughness is sufficient to resist most anticipated flaws such as scratches [12].

In (1982) the fracture toughness of rigid PVC pipe materials over a range of test temperatures as a function of extrusion temperature studied by Mandell et al. The fracture toughness was showed to be reduced as the extrusion temperature is reduced below standard conditions [13].

The effect of processing on the fracture toughness in unplasticized PVC pipes was studied by Mai and Kerr in (1985). This work showed the UPVC under impact conditions processing has no significant effects on notched Charpy fracture toughness [14].

In (1992) Anandakumaran and Stonkus was determined degradation of PVC cable insulation. The found that the shift in the T_g or the characteristic melting peak predicted the maximum exposure temperature of the PVC [15].

In (1994) Hussain and Mahmood investigated the effect of rare earth metal on the thermal degradation of PVC. It was studied using thermogravimetric technique. They concluded that these metals increase the activation energy of PVC [16].

Polyvinyl chloride pipe degradation was studied in natural environments at Dhahran (K.S.A) and Florida by Ikram et al. in (1995). The data showed that the PVC pipe lost 50 percent of its elongation at break within 12 to 16 months at Dhahran and more than 24 months in Florida during outdoor exposure [17].

In (2000), Raghi, et al., investigated the effect of weathering on mechanical properties of blends consisting of UPVC and Indulin Lignin (IL), that are prepared by compression molding. Results showed that Lignin addition to PVC leads to an increase in tensile strength and a slight decrease in elongation at yield and at break [18].

The thermomechanical and thermogravimetric analysis of blends of (PVC) with maleic anhydride–allyl propionate (MA–AP) copolymer was studied by Boztu, et al., in (2004). They found that the mixture has more plasticity when increased in ratio of the (MA–AP) on the PVC [19].

In (2005), Yu and Selvadurai, discussed the mechanical behavior of PVC subjected to ethanol. The results exposure to pure ethanol resulted in a reduction in flexibility, and embrittlement [20].

In (2005) Benes et al. studied the Thermal behavior of PVC cable insulation were tested by TG/DTG and DSC during heating in the range 20-800˚C. which showed the kinetics of the PVC degradation in the range 200-340˚C and These kinetic parameters were used for the lifetime simulation of the PVC [21].

In (2006) Thongpin et al. studied the effect of addition of polyethylene to PVC on Thermal degradation mechanism. The degradation behavior was investigated in terms of decomposition temperature and glass transition temperature. The results suggested that adding small amounts of PE with PVC could thermally stabilize the PVC, as noted by an increase in its decomposition temperature and increase of the glass transition temperature [22].

In (2006) Chakrabarti and Chakraborty was investigated Mechanical and thermomechanical characterization of PVC/polyalkylacrylate blends. The results showed the tensile modulus, ultimate tensile strength and thermal stability of PVC was increased when adding the polyalkylacrylate blends [23].

The thermal behavior of rigid (PVC) was studied using thermogravimetric analysis by Cailean, et al. In (2007). They found that from thermal analysis it could be prediction the thermal lifetime of (PVC) products [24].

In (2008), Haddadi and Famili, studied the mechanical properties of (PVC)/polyurethane (PU) blends. The results showed that elongation at break and toughness increased when adding (PU) to PVC [25].

In (2010), Awham and Zaid investigated the diffusivity mechanism of different kinds of water into (UPVC). The results indicated that the behavior of water diffusion through the polymer was found to be affected by the type of water depending on the (pH) value and the percentage of total dissolved salts (TDS) in each type of water [26].

At the same year, Unar, et al. studied the effect of various additives on the mechanical properties of (PVC). It was observed that the tensile strength increases in the absence of plasticizing agents, with presence of fillers and elongation at break decreases with the increase of filler/stabilizer content for sample free of plasticizers (Rigid PVC compounds) [27].

In (2011) Naydenova and Velev studied the optimizing composition for the production of (PVC) profiles for doors and windows. The whiteness and impact resistance of PVC profiles were defined, subjected to two types of accelerated aging for 300 hours - with xenon and UV - B light sources. It was found that the studied parameters decreased over time. Xenon source leads to more intensive aging of PVC composite made of (U-PVC) [28].

In the same year Haider, et al. investigated the effect of glass addition on the physical properties of (PVC). This work carried out the thermomechanical analyzer (TMA) and differential scanning calometry (DSC) for tested Tg. It was observed that the increase of glass temperature (Tg) with high decrease of Young modulus (E) and transform its mechanical properties from elastic to plastic [29].

In (2012), Onyeaju, et. al. compared the thermal properties of asbestos and polyvinylchloride (PVC) ceiling sheets. The result showed that thermal conductivity, thermal resistivity, thermal absorptivity, thermal diffusivity and specific heat capacity values of PVC and asbestos ceiling sheets fall within the range of good insulating materials [30].

In (2013), Manjunath, et al. studied the cenospheres as fillers for PVC compounds for applications in electrical cables. The results, on the whole, indicate that cenospheres hold out promise as possible fillers for PVC compounds for use in electrical cable sheathing applications [31].

1-3 The Aim of Study:

Because of the large number of products imported from the PVC pipes and doors, it became necessary to find out what the best companies in practice. This study focused on a comparison between four different companies (The most popular in Iraq), In terms of:

- Mechanical properties: which include (Young's modulus, tensile strength, flexural strength, toughness and hardness).
- Thermal properties: which include (thermal conductivity, thermal stability, glass transition temperature and softening point).
- Determination of default lifetime of these products via thermal degradation profile.

1-4 Methodology:

1- Sample preparation for mechanical tests:

   a- Tensile test, according to ASTM-D 638.

   b- Bending test, according to ASTM-D 790.

   c- Impact test, according to ISO-179.

   d- Hardness Shore D ASTM- D2240.

2- Using the mechanical testing machine to determine the different mechanical properties (tensile, bending, impact and hardness shore D).

3- Using Lee's disc instrument, the thermal conductivity of doors are determined.

4- The thermogravimetric analysis instrument (TGA) was used to identify the kind of polymers and determine the activation energy of pipes and doors, the lifetime is calculated by using the extrapolation of relationship between the time and temperature.

5- The thermomechanical analysis instrument (TMA) was used to determine the glass transition temperature (T_g) and softening point (T_s).

Chapter Two

Theoretical Part

2-1 General Concepts

The word ‘‘polymer’’ is derived from the Greek poly and meros, meaning many and parts, respectively. Some scientists prefer to use the word ‘‘macromolecule,’’ or large molecule, instead of polymer [2].

The repeating structural units are derived from some simple and reactive molecules known as monomers and are linked to each other by covalent bonds. This process of formation of polymers from respective monomers is called polymerization [32, 33].

If -A- is the base unit, then a polymer molecule or macromolecule is represented by:

——-A-A-A-A-A-A———— or---(A)*n*---

Where *n* is an integer, called degree of polymerization of this macromolecule, DP (the number of repeat units in the molecule) [34-36].

2-2 Classification of Polymers: [33], [37-41]

There are several ways of classification of polymers based on some special considerations. The following are some of the common classifications of polymers:

2-2-1 Classification Based on Source:

Under this type of classification, there are three sub categories.

a- ***Natural polymers:***

These polymers are found in plants and animals. Examples are proteins, cellulose, starch, some resins and rubber.

b- Semi-synthetic polymers:

Cellulose derivatives as cellulose acetate (rayon) and cellulose nitrate, etc. are the usual examples of this sub category.

c- Synthetic polymers:

A variety of synthetic polymers as plastic (polyethylene), synthetic fibres (nylon 6,6) and synthetic rubbers (Buna - S) are examples of manmade polymers extensively used in daily life as well as in industry.

2-2-2 Classification Based on Structure of Polymers:

There are three different types based on the structure of the polymers.

a- Linear polymers

These polymers consist of long and straight chains. The examples are high-density polythene (HDPE), polyvinyl chloride (PVC), etc.

b- Branched chain polymers

These polymers contain linear chains having some branches, *e.g.*, low-density polythene (LDPE).

c- Cross linked or Network polymers

These are usually formed from bi-functional and tri-functional monomers and contain strong covalent bonds between various linear polymer chains, e.g. bakelite, melamine, etc.

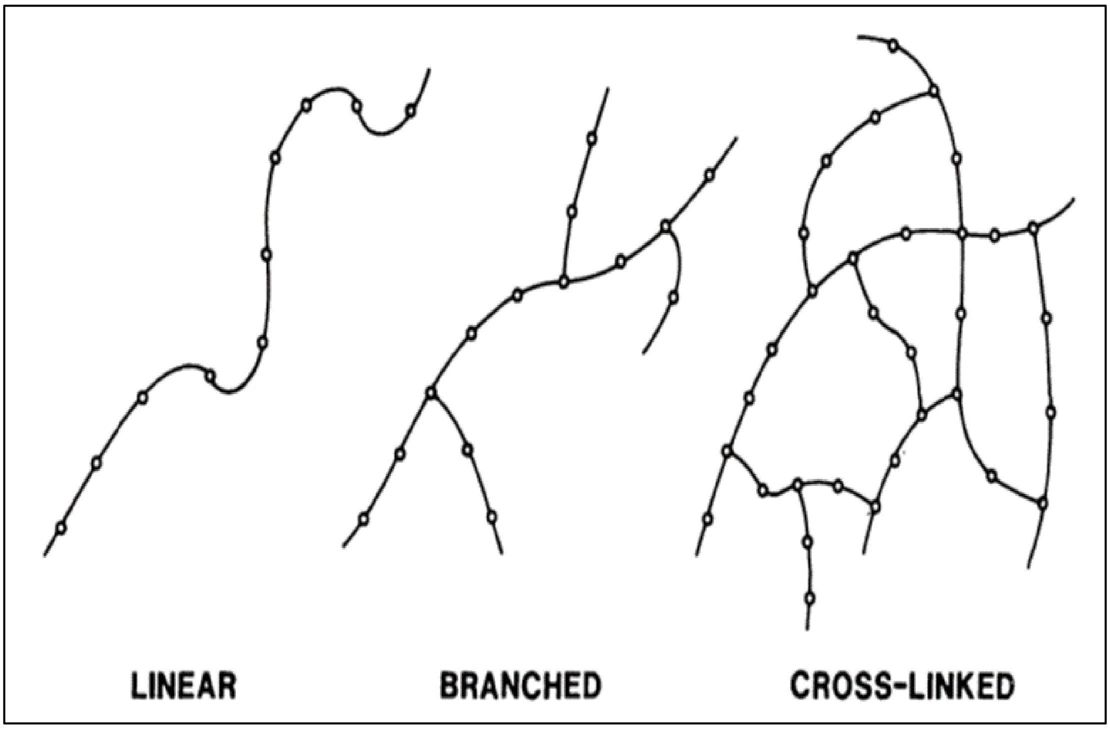


Fig. (2-1) Some types of polymers depending on the form of chains

2-2-3 Classification Based on Mode of Polymerization:

Polymers can also be classified on the basis of mode of polymerization into two sub groups.

a- Addition Polymers

The addition polymers are formed by the repeated addition of monomer molecules possessing double or triple bonds, *e.g.*, the formation of polythene from ethene and polypropene from propene. However, the addition polymers formed by the polymerisation of a single monomeric species are known as **homopolymers**, *e.g.*, polythene.

The polymers made by the addition of polymerisation from two different monomers are termed as **copolymers**, *e.g.*, Buna-S, Buna-N, etc.

b- Condensation Polymers

The condensation polymers are formed by repeated condensation reaction between two different bi-functional or tri-functional monomeric units. In these polymerisation reactions, the elimination of small molecules such as water, alcohol, hydrogen chloride, etc. take place. The examples are terylene (dacron), nylon 6, 6, nylon 6, etc. For example, nylon 6, 6 is formed by the condensation of hexamethylene diamine with adipic acid.

2-2-4 Classification Based on Thermal Behavior:

The most useful classification of polymers is based on their thermal (thermomechanical) response.

a- Thermoplastic Polymers

Thermoplastic polymers soften and flow under the action of heat and pressure. Upon cooling, the polymer hardens and assumes the shape of the mold (container). Thermoplastics, when compounded with appropriate ingredients, can usually withstand several of these heating and cooling cycles without suffering any structural breakdown. Examples of thermoplastic polymers are polyethylene, polystyrene, and nylon.

b- Thermosetting polymers

A thermoset is a polymer that, when heated, undergoes a chemical change to produce a cross-linked, solid polymer. Thermosets usually exist initially as liquids called prepolymers; they can be shaped into desired forms by the application of heat and pressure, but are incapable of undergoing repeated cycles of softening and hardening. Examples of thermosetting polymers include urea–formaldehyde, phenol–formaldehyde, and epoxies [33], [37-41].

2-3 Polymers and Plastics:

The words polymers and plastics are often taken as synonymous but in fact, there is a distinction. The polymer is the pure material, which results from the process of polymerization and it is usually taken as the family name for materials which have long chain-like molecules (and this includes rubbers). Pure polymers are seldom used on their own and it is when additives are present that the term plastic is applied. Polymers contain additives for a number of reasons. The following list outlines the purpose of the main additives used in plastics.

Fillers. some fillers, such as short fibres or flakes of inorganic materials, improve the mechanical properties of a plastic. Others, called extenders, permit a large volume of a plastic to be produced with relatively little actual resin. Calcium carbonate, silica and clay are frequently used extenders.

Lubricants. Lubricants such as wax or calcium stearate reduce the viscosity of the molten plastic and improve forming characteristics.

Pigments. Pigments are used to produce colours in plastics.

Stabilisers. Stabilisers prevent deterioration of the polymer due to environmental factors. Heat stabilisers are required in processing polyvinyl chloride. Stabilisers also prevent deterioration due to ultra-violet radiation[1].

2-4 PVC (Polyvinyl Chloride):

Polyvinyl chloride is a thermoplastic (CH_2=CHCl), also known as chloroethylene. Unmodified PVC is stronger and stiffer than PE and PP. Which is most often obtained by reacting ethylene with oxygen and hydrogen chloride, see figure (2-2). Vinyl chloride (VC) is produced from ethylene and chlorine at a ratio of 43% to 57%. VC is the monomeric building block of PVC [42-45].

Salt, which is abundant in supply, is actually the primary ingredient of vinyl. Chlorine derived from the electrolysis of salt water is combined with ethylene (a petroleum derivative) in a clean, automated closed-loop process that yields ethylene dichloride (EDC). EDC is then “cracked” under heat to produce vinyl chlorine monomer (VCM). Vinyl resin, typically in powder or granular form, is produced when the VCM is subjected to polymerization to produce polyvinyl chloride, or PVC. Polymerization is the process by which smaller organic molecules (monomers) are bonded together chemically into a much larger, chainlike repeating molecule called a polymer [42].

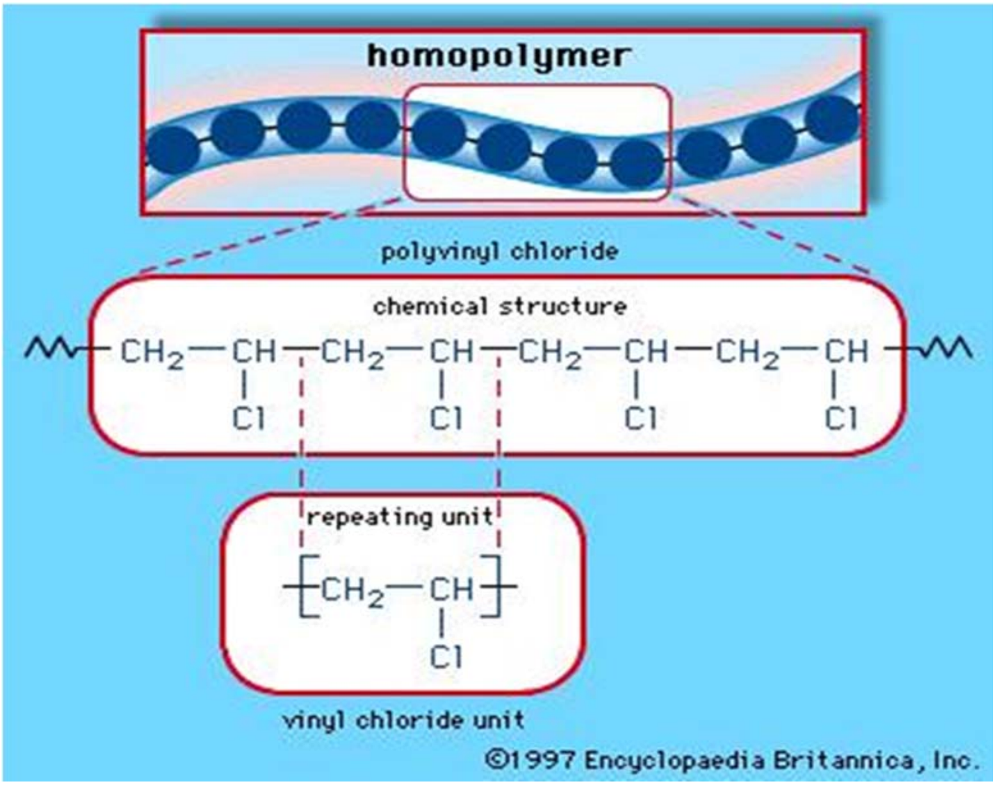


Fig. (2-2) structure of PVC

Uncompounded PVC is colourless and rigid, and possesses poor stability towards heat and light. Indeed, PVC is certainly the least stable of the high tonnage commercial polymers [32].

PVC is intrinsically fire retardant because of the presence of chlorine in the polymer matrix. PVC has excellent electrical insulation properties, making it ideal for cabling applications. Its good impact strength and weatherproof attributes make it ideal for construction products [36].

PVC can be used in numerous products due to its outstanding properties and therefore is an integral part of our lives.

Typical applications of PVC include; window frames, drainage pipe, water service pipe, medical devices, blood storage bags, cable and wire insulation, resilient flooring, roofing membranes, stationary, automotive interiors and seat coverings, fashion and footwear, packaging, cling film, credit cards, vinyl records, synthetic leather and other coated fabrics[42, 46].

PVC has been used extensively in a wide range of construction products for over half a century. PVC's strong, lightweight, durable and versatile characteristics make it ideal for window profiles. Unplasticised PVC is one of the stiffest polymers at normal ambient temperature and shows little deterioration after many years in service [36].

PVC is versitile and can be used for different colours and effects, often being used as an alternative to traditional wood frames.

The Building Research Establishment (BRE), the UK's leading authority on sustainable construction, has granted U-PVC windows a life span of more than 35 years however; there are many examples of products lasting much longer than this [36].

2-5 Mechanical Properties:

2-5-1 Tensile Test:

The tensile test is popular since the properties obtained can be applied to design different components. The tensile test measures the resistance of a material to a static or slowly applied force. The strain rates in a tensile test are typically small [47].

In general, the stress as the force acting per unit area over which the force is applied. Strain is defined as the change in dimension per unit length. Stress is typically expressed in Pa (Pascals). Strain has no dimensions [48].

In polymeric materials, this stress will correspond to disentanglement of polymer molecule chains or sliding of chains past each other.

Tensile Strength the stress obtained at the highest applied force is the tensile strength, which is the maximum stress on the engineering stress-strain curve. This value is also commonly known as the ultimate tensile strength (UTS) [47].

Elastic Properties The modulus of elasticity, or Young's modulus (E), is the slope of the stress-strain curve in the elastic region. This relationship between stress and strain in the elastic region is known as Hooke's Law [49, 50]:

$$E = \frac{stress}{strain} \ldots\ldots\ldots\ldots (2\text{-}1)$$

Tensile Toughness The energy absorbed by a material prior to fracture is known as tensile toughness and is sometimes measured as the area under the true stress–strain curve (also known as the work of fracture). We will define true stress and true strain since it is easier to measure engineering stress–strain, engineers often equate tensile toughness to the area under the engineering stress–strain curve [47, 51].

Elastic strain is defined as fully recoverable strain resulting from an applied stress. The strain is "elastic" if it develops instantaneously (i.e., the strain occurs as soon as the force is applied), remains as long as the stress is applied, and is recovered when the force is withdrawn. A material subjected to an elastic strain does not show any permanent deformation (i.e., it returns to its original shape after the force or stress is removed) [47].

The slope of a tensile stress-strain curve in the elastic portion defines the Young's modulus or modulus of elasticity (*E*) of a material see figure (2-5) The units of *E* are measured in pounds per square inch (psi) or Pascals (Pa) (same as those of stress). application of the load corresponds to moving from the origin up and along the straight line. Upon release of the load, the line is traversed in the opposite direction, back to the origin [52, 53].

Plastic deformation in a material is known as the plastic strain. In this case, when the stress is removed, the material does not go back to its original shape. A dent in a car is plastic deformation! Note that the word "plastic" here does not refer to strain in a plastic (polymeric) material, but rather to permanent strain in any material (figure (2-6)) [54].

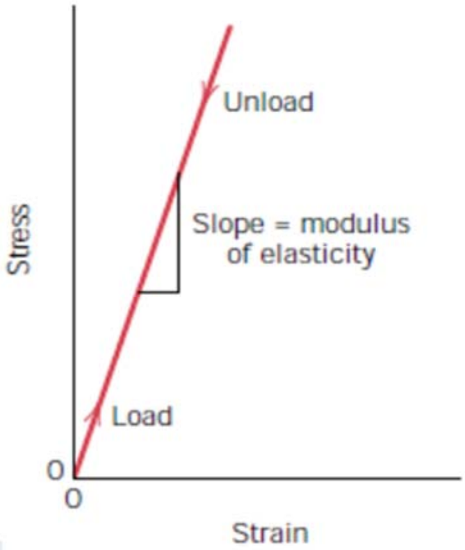


Fig. (2-5) schematic stress-strain diagram showing linear elastic deformation for loading and unloading cycles [55]

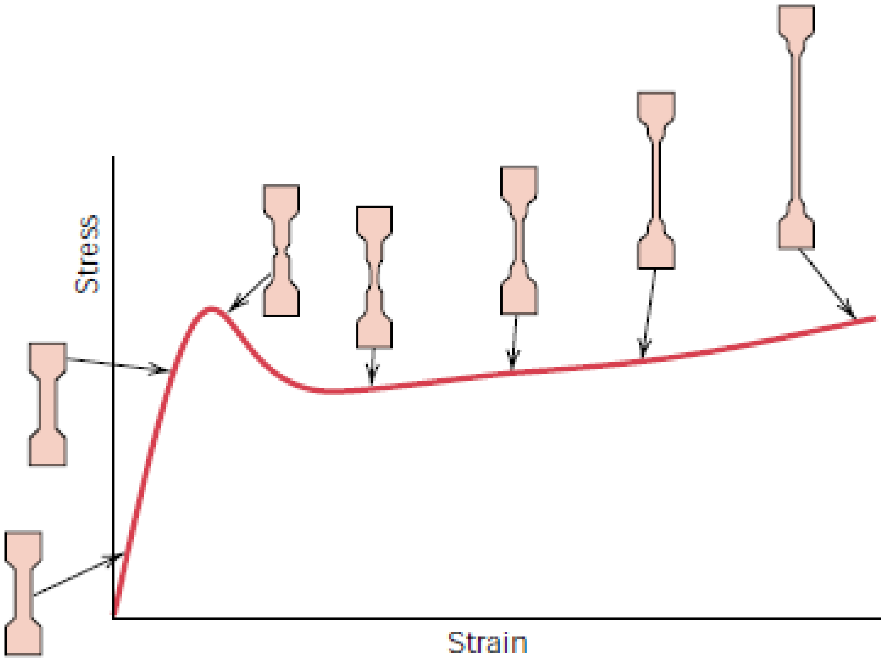


Fig. (2-6) Schematic tensile stress–strain curve for a semicrystalline polymer. Specimen contours at several stages of deformation are included [55]

2-5-2 Bending Test:

Showing the applying of the load at three points and causing bending, a tensile force acts on the material opposite the midpoint. Fracture begins at this location. The flexural strength, or modulus of rupture, describes the material's strength: [47, 55]

Flexural strength for three - point bend test see figure (2-7) [56]:

$$\sigma_{bend} = \frac{3FL}{2wh^2} \quad \text{.........} \quad (2\text{-}1)$$

Where F is the fracture load, L is the distance between the two outer points, w is the width of the specimen, and h is the height of the specimen. The flexural strength has units of stress. The results of the bend test are similar to the stress-strain curves; however, the stress is plotted versus deflection rather than versus strain.

The modulus of elasticity in bending, or the flexural modulus (E_{bend}), is calculated as [56]:

Flexural modulus $E_{bend} = \frac{FL^3}{4w\delta^3}$

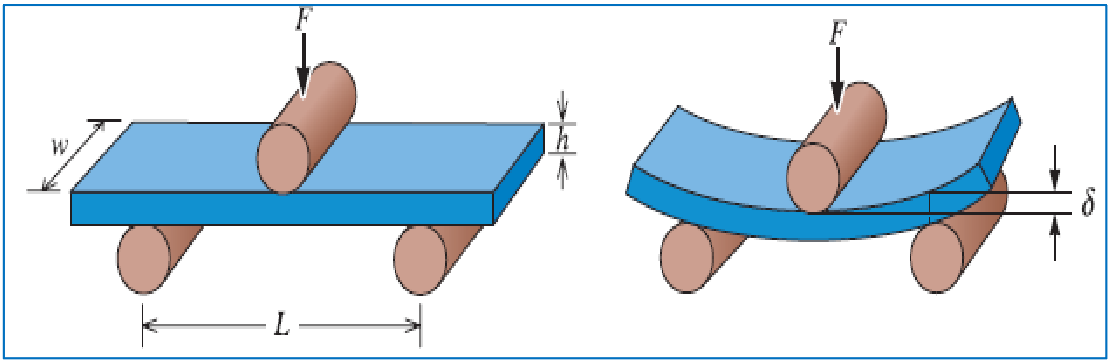


Fig. (2-7) The bending test.

2-5-3 Impact test:

When a material is subjected to a sudden, intense blow, in which the strain rate (or) is extremely rapid, it may behave in much more brittle a manner than is observed in the tensile test.

In the tensile or bending tests, the polymer molecules have time to disentangle or the chains to slide past each other and cause large plastic deformations. If, however, we apply an impact loading, there is insufficient time for these mechanisms to play a role and the materials break in a brittle manner. An impact test is often used to evaluate the brittleness of a material under these conditions. In contrast to the tensile test, in this test, the strain rates are much higher.

Many test procedures have been devised, including the Charpy test and the Izod test fig. (2-8). The Izod test is often used for plastic materials. The test specimen may be either notched or unnotched; V-notched specimens better measure the resistance of the material to crack propagation [47].

In the test, a heavy pendulum, starting at an elevation h_0, swings through its arc, strikes and breaks the specimen, and reaches a lower final elevation hf. If we know the initial and final elevations of the pendulum, we can calculate the difference in potential energy. This difference is the impact energy absorbed by the specimen during failure. For the Charpy test, the energy is usually expressed in joules (J). The ability of a material to withstand an impact blow is often referred to as the impact toughness of the material [47].

The specimen is fixed in its pertaining place, and then the energy gauge is initialized (on zero position) after that, the pendulum is freed where its potential energy would be changed to kinetic energy. Some of this kinetic energy is utilized to fracture the specimen, while the energy gauge reads the value of fracture energy (U_c) for the sample under test. Impact strength (I.S.) is calculated by applying the relationship: [57-59]

$$I.S. = \frac{U_C}{A} \qquad \text{………… (2-2)}$$

$$A = b \times t \qquad \text{…………. (2-3)}$$

Where:

U_c: is the fracture energy (KJoule) which is determined from Charpy impact test instrument.

A: is the cross – sectional area of the specimen.

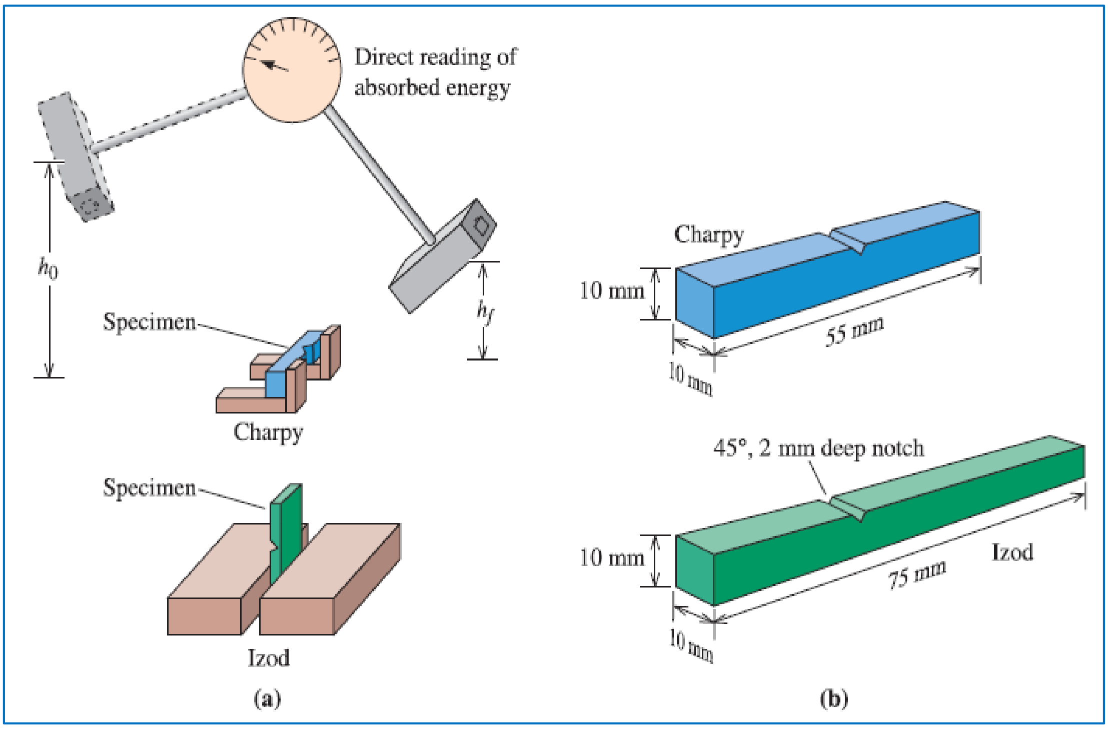


Fig. (2-8) The impact test: (a) the Charpy and Izod tests, and (b) dimensions of typical specimens [47].

2-5-4 Hardness test:

The hardness test measures the resistance to penetration of the surface of a material by a hard object. It is a qualitative measure of the strength of the material [47, 60].

The depth or size of the resulting indentation is measured, which in turn is related to a hardness number; the softer the material, the larger and deeper the indentation, and the lower the hardness index number [55].

2-6 Thermal Properties:

2-6-1 Thermal Conductivity:

The thermal conductivity *(k)* is a measure of the rate at which heat is transferred through a material. Thermal energy is transferred by two important mechanisms: transfer of free electrons and lattice vibrations (or phonons). Valence electrons gain energy, move toward the colder areas of the material, and transfer their energy to other atoms. The amount of energy transferred depends on the number of excited electrons and their mobility; these, in turn, depend on the type of material, lattice imperfections, and temperature. In addition, thermally induced vibrations of the atoms transfer energy through the material see figure (2-9) [47].

$$q = - k\frac{dT}{dX} \ldots\ldots\ldots\ldots \text{(2-4)}$$

Where q denotes the *heat flux,* or heat flow, per unit time per unit area (area being taken as that perpendicular to the flow direction), k is the thermal conductivity, and dT/dx is the *temperature gradient* through the conducting medium.

The thermal conductivity of polymers is very low—even in comparison with silicate glasses. Vibration and movement of the molecular polymer chains transfer energy [61].

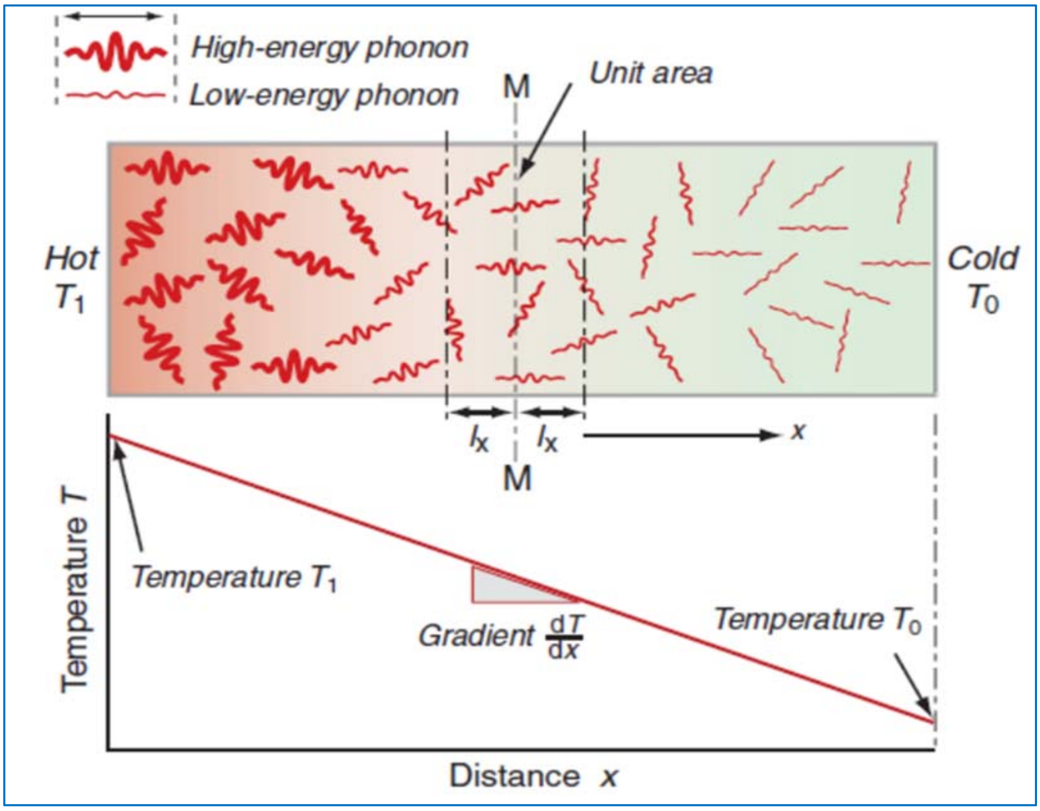


Fig. (2-9) the transmission of heat by the motion of phonons

2-6-2 Thermal Degradation:

Thermal degradation of polymers is ‘molecular deterioration as a result of overheating’. At high temperatures the components of the long chain backbone of the polymer can begin to separate (molecular scission) and react with one another to change the properties of the polymer. It is part of a larger group of degradation mechanisms for polymers that can occur from a variety of causes such as [62-64]:

- Heat (thermal degradation and thermal oxidative degradation when in the presence of oxygen).
- Light (photodegradation).
- Oxygen (oxidative degradation).
- Weathering (generally UV degradation).

In general, the ability of a plastic to resist these degradation causes is called the ‘stability’ (resistance to permanent changes in property caused solely by heat) of the plastic and in this article; we will concentrate on the process of thermal degradation with particular emphasis on thermal degradation in service as opposed to thermal degradation during processing [65].

The initiation of thermal degradation involves the loss of a hydrogen atom from the polymer chain (shown below as R.H) as a result of energy input from heat or light. A group of stabilizers work by reacting with the hydroperoxide (ROOH) to produce inactive and stable products such as ROH and break the cycle at the hydroperoxide propagation step.

In most cases a given plastic will incorporate a mix of stabilizers that are designed to work as a system to give the desired properties for the application. This mix will be designed specifically for the polymer being used and the requirements of the application. The mixture will also be designed to be applied at a specific concentration – over dosing stabilizers can in fact be detrimental to the plastic and the effect of the stabilizer.

Testing of plastics for thermal stability is subject to the same concerns as any accelerated test method. In general, accelerated testing involves high temperatures over short times and the results are then extrapolated back to the lower service temperature and longer time of the real application. In this situation, there is always a need to balance the time taken for the test against the error that is naturally involved in extrapolating back to the service temperature.

The assumed mechanism is generally an Arrhenius model (first proposed by the Swedish chemist Svante Arrhenius) [62, 66].

$k = A \exp(-E_a/RT)$ .... [67] (2-5)

Values of the Arrhenius parameters (E_a and A) where E_a the minimum energy required for the reaction (*activation energy*), and A the frequency of occurrence of the situation that may lead to product formation (the *frequency factor).* Where R is the universal gas constant.

In thermogravimetric analysis the conversion rate of reaction may be defined as the ratio of actual mass loss to the total mass loss corresponding to the degradation process [68]

$$\gamma = \frac{M_\circ - M}{M_\circ - M_f} \text{ ………….. (2-6)}$$

Where γ: weight of active material.

M, $M_\circ$ and M_f are the actual, initial and final masses or weights of the sample respectively.

The thermal degradation occurs in the reaction rate by the relationship:

$$R_T = -\frac{d\gamma}{dt} \text{ ………… (2-7)}$$

t: reaction time.

The reaction rate is relevant with rate constant (k) by equation (2-8).

$$-\frac{d\gamma}{dt} = k\,\gamma^n \text{ ………… (2-8)}$$

Where n: is the reaction order.

If we consider for approximation interactions degradation of the polymer as a first order (n=1)

From equations (2-7) and (2-8):

$$-\frac{d\gamma}{dt} = A \exp(-E_a/RT) \text{…….. (2-9)}$$

Since the

$$\beta = \frac{dT}{dt} \text{ ……… (2-10)}$$

Where β: is the rate of heating.

$$-\frac{d\gamma}{dt} = \frac{A}{\beta} \exp(-E_a/RT)\,\gamma \text{ …..... (2-11)}$$

By integration of both sides of the equation: [69]

$$-\frac{d\log(\beta)}{d\left(\frac{1}{T}\right)} \approx 0.457\,\frac{E_a}{R} \text{ ….…..... (2-12)}$$

2-6-3 Thermomechanical analysis (TMA)

Techniques in which the change of a dimension or a mechanical property of the sample is analysed while the sample is subjected to a temperature alteration. [70]

Traditionally, TMA is used to characterize linear expansion, glass transitions, and softening points of materials by applying a constant force to a specimen while varying temperature. For expansion measurements, a probe rests on a sample on a stage with minimal downward pressure. Other constant force experiments include measurement of penetration see figure (2-10) [71, 72].

There are two cases of TMA:

- *Thermodilatometry :* Techniques in which a negligible force acts on the sample. The instrument is a dilatometer. The classical instrument for the determination of the coefficient of thermal expansion (α) is the push-rod dilatometer. Via the push rod, the variation of the length of the rod-shaped sample is transmitted to a displacement transducer (electromagnetic, capacitive, optical, and mechanical) which gives an electrical signal proportional to the change in length. The arrangement of the sample in the furnace must be such that there is only low friction [27].
- *Static force thermomechanical analysis (sf-TMA):* Techniques in which a static force acts on the sample. To investigate the behaviour of substances under stresses similar to those occurring in practice, samples of suitable shape are subjected to static stress. Compressive, tensile, flexural or torsional stress may be concerned, even complex states of stress are simulated. Depending on the kind of stress to which the sample is to be exposed, suitable sample holding devices and force applying mechanisms are used [70]. It has also been used to determine changes in modulus with temperature [73].

The choice of probe type determines the actual modulus/expansion property being measured. Figure (2-11) illustrates the most useful attachments.

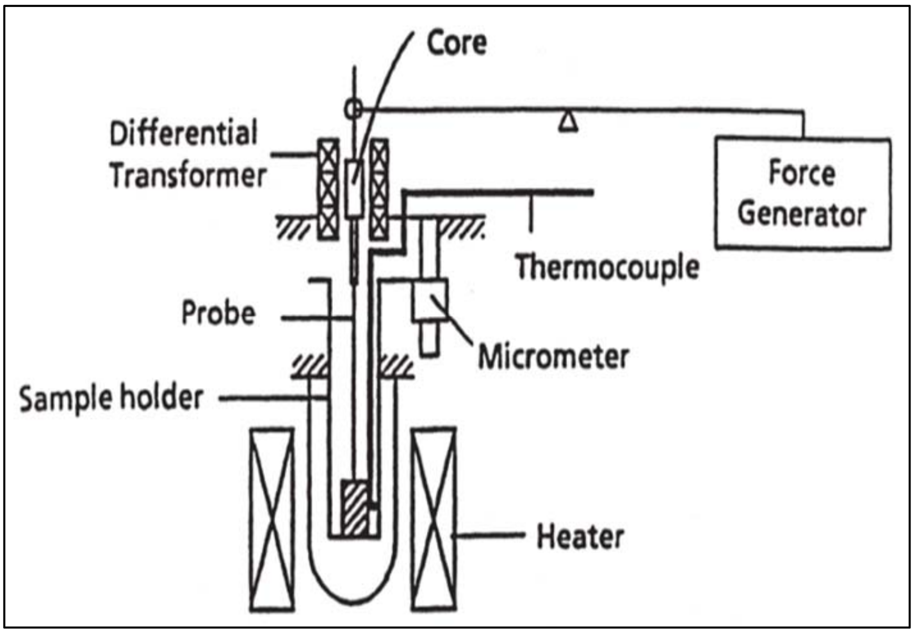


fig. (2-10) TMA measurement principle

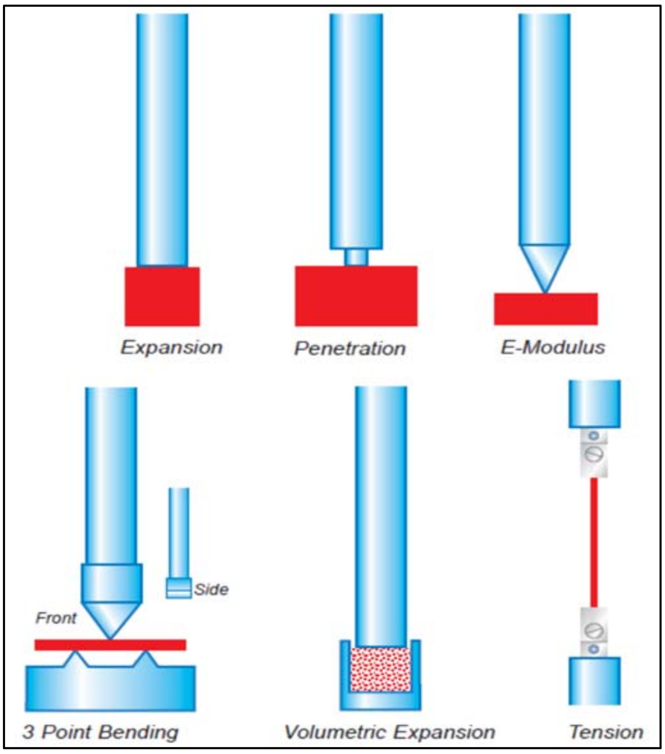


Fig. (2-11) prop types of TMA instrument

Chapter Three

Experimental Part

3-1 Sample Preparation:

We used samples of PVC pipes with different origins and another two samples of PVC doors with different manufactures as shown in table (3-1).

The specimens were prepared for different mechanical tests according to ASTM and ISO standards. The test specimens were cut by using different tools in workshop.

Table (3-1) PVC pipes and doors with different origins or manufacturing

NO.	Origin	Manufacturer	Application
1	K.S.A.	AL-JAWDAH	Pipes
2	Egypt	AL-SHARIF	Pipes
3	Turkey	NOVA	Door
4	Turkey	FIRAT	Door

3-2 Mechanical Property Test

Tensile, bending, impact and hardness tests, were carried out using tensile/bending testing machine, impact machine and hardness testing machine respectively. Three identical samples were tested for tensile strength, bending, impact strength and hardness.

3-2-1 Tensile strength:

The tensile behavior of samples was determined at room temperature using tensile testing machine model H50KT manufactured by (Tinius Olsen/UK). The samples were prepared in accordance with ASTM-D638. As in figure (3-1). The specimen was loaded between two manually adjustable grips of a (2000 N) computerized tensile testing machine, with an electronic extensometer. The system uses add-on cards for data analysis as in figure (3-2).

Each test was repeated thrice and the average value was taken to calculate the tensile properties of the samples.

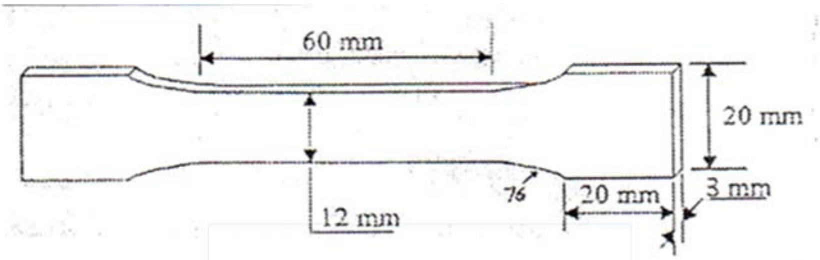


Fig.(3-1) tensile test specimen

Fig. (3-2) Tensile test machine

3-2-2 Flexural Strength:

lexural strength is determined by 3-point bend test. The test specimen as in fig. (3-3) in accordance with ASTM-D790 were used for test. Using bending instrument model H50KT manufactured by (Tinius Olsen/UK) as in fig. (3-4).

Fig.(3-3) Bending test specimen

Fig. (3-4) Flexural test machine

3-2-3 Impact Test Instrument

The Charpy impact strength of samples was tested using the standard test specimen in accordance with ISO-179, as in figure(3-5) having 45˚ V-notch with 2mm deep.

(Charpy test) model IMI manufacturer by Amityville/ New York this instrument consists mainly of pendulum and energy gauge. Charpy impact test consists of standard test piece that would be broken with one blow of a swinging hammer. The test piece is supported at both its ends in a way that the hammer strikes it at the middle. The testing method of this instrument includes lifting of the pendulum to its maximum height and fixing it firmly as shown in Fig (3-6).

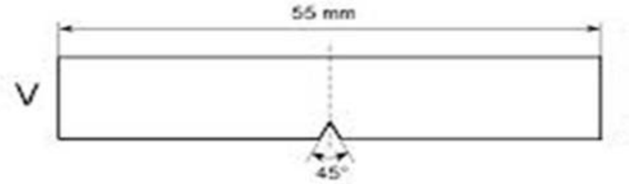


Fig.(3-5) Impact test specimen

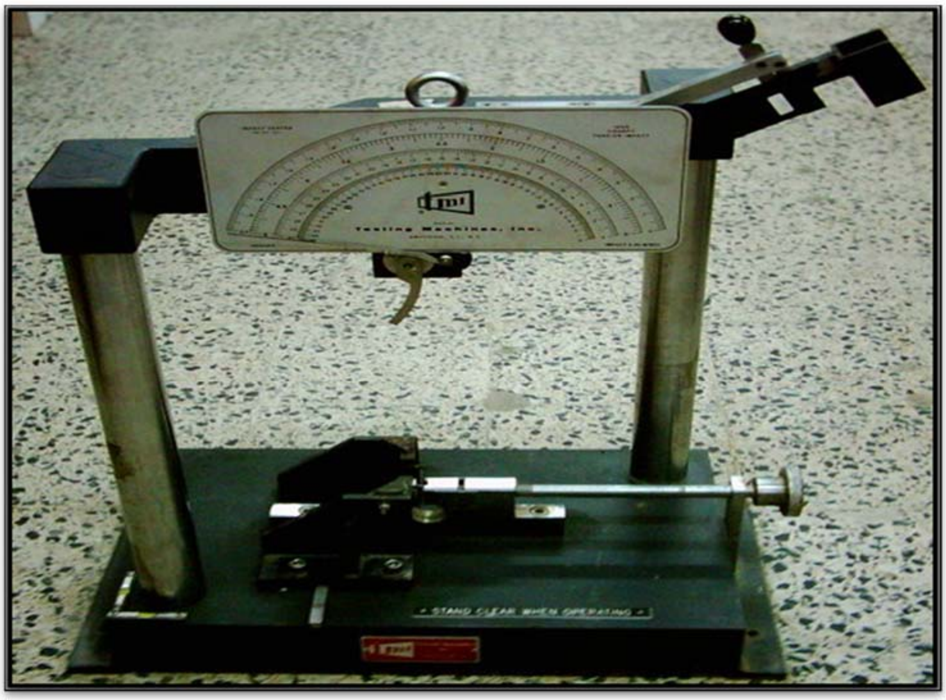


Fig.(3-6): Charpy impact test instrument

3-2-4 Hardness Test Instrument:

Shore D Durometer Hardness instrument showed in figure (3-7), fabricated by TIME GROUP INC Company, was used to carry out the hardness test by using the steel indenter of certain is pushed into the sample surface vertically with testing force, and when surface of pressure foot and the sample touched completely (as in figure (3-8)), the indenter extends out for a certain length "L" from the pressure foot plane. Greater length "L" means lower shore hardness value and smaller length "L" means bigger shore hardness value.

Fig. (3-7): Durometer Hardness instrument from type (Shore D)

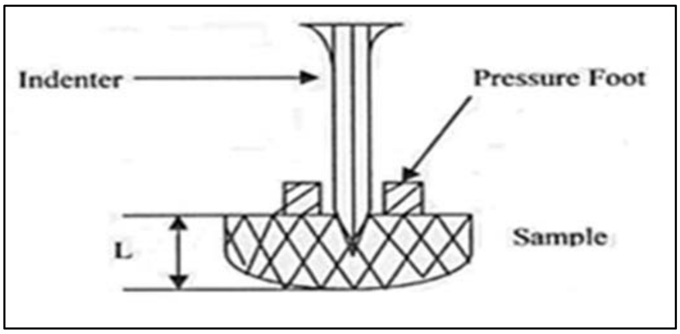


Fig. (3-8) scheme of hardness Shore D

3-3 Thermal Properties:

3-3-1 Thermal Conductivity Test Instrument:

Lee's disc instrument showed in figure (3-8), manufactured by the Griffen and George Company, was used to calculate the thermal conductivity of the samples under test. Instrument consists of three discs of brass (40 mm diameter by 12.25 mm thickness) and a heater. The sample (S) is placed between the discs A and B, while the heater is placed between B and C. Heater was supplied with voltage (6 volt) and the current value through the apparatus was about (0.25A). The heat transfers from the heater to the near two discs then to the third disc across the sample. The temperature of the three discs (T_A, T_B, and T_C) is measured by using a thermometers placed inside them. After reaching thermal equilibrium, the temperatures were recorded.

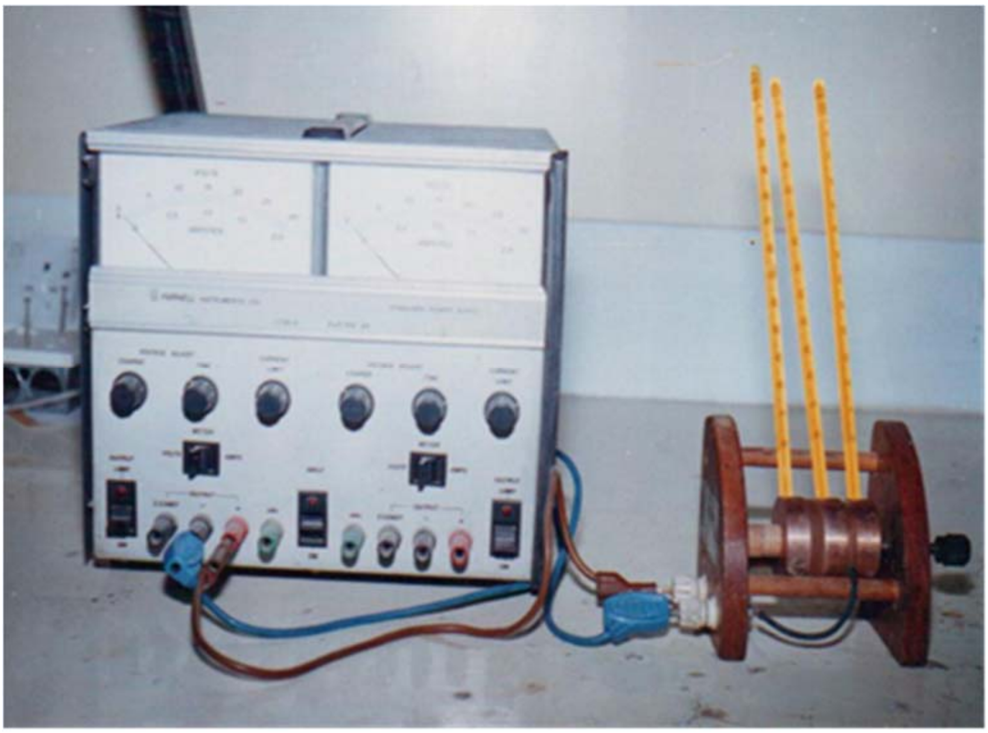


Fig. (3-9)Thermal Conductivity Test Instrument

The value of thermal conductivity is determined by using the following equation [74]:

$$k\left[\frac{T_B - T_A}{d_S}\right] = e\left[T_A + \frac{2}{r}(dA + \frac{1}{4}d_S)T_A + \frac{1}{2r}d_S T_B\right] \quad \ldots\ldots \text{(3-2)}$$

Where:

K: The thermal conductivity coefficient (W/m. °C).

T_A, T_B, & T_C: Temperature of the metal discs A, B, C respectively (°C).

d_A, d_B & d_C: Thickness of the discs A, B & C respectively (mm).

d_S: Sample's thickness (mm).

r: disc's radius (mm).

e: The quantity of heat flowing through the cross sectional area of the specimen per unit time ($W/m^2.°C$) is calculated from the following equation [74]:

$IV = \pi r^2 (T_A + T_B) + 2\pi re\,[d_A T_A + d_S (1/2)(T_A + T_B) + d_B T_B + d_C T_C]$ …(3-3)

Where:

I= Current through the heater (Ampere)

V= Applied voltage (Volt)

3-3-2 Thermogravimetric Analysis Instrument (TGA):

Figure (3-10) shows the (TGA) instrument model TGA4000 manufactured by (PerkinElmer/Germany).

It is Consists of a sample pan that is supported by a precision balance as shown in figure (3-11). That pan resides in a furnace and is heated or cooled during the experiment. The mass of the sample is monitored during the experiment. A sample purge gas controls the sample environment. This gas may be inert or a reactive gas that flows over the sample and exits through an exhaust.

Essentially, separate measurements are carried out at different linear heating rates (2, 5, 10, 20, 40 C°/min) for all samples. The sample weight about (20-30 mg).

Fig. (3-10)Thermogravimetric instrument

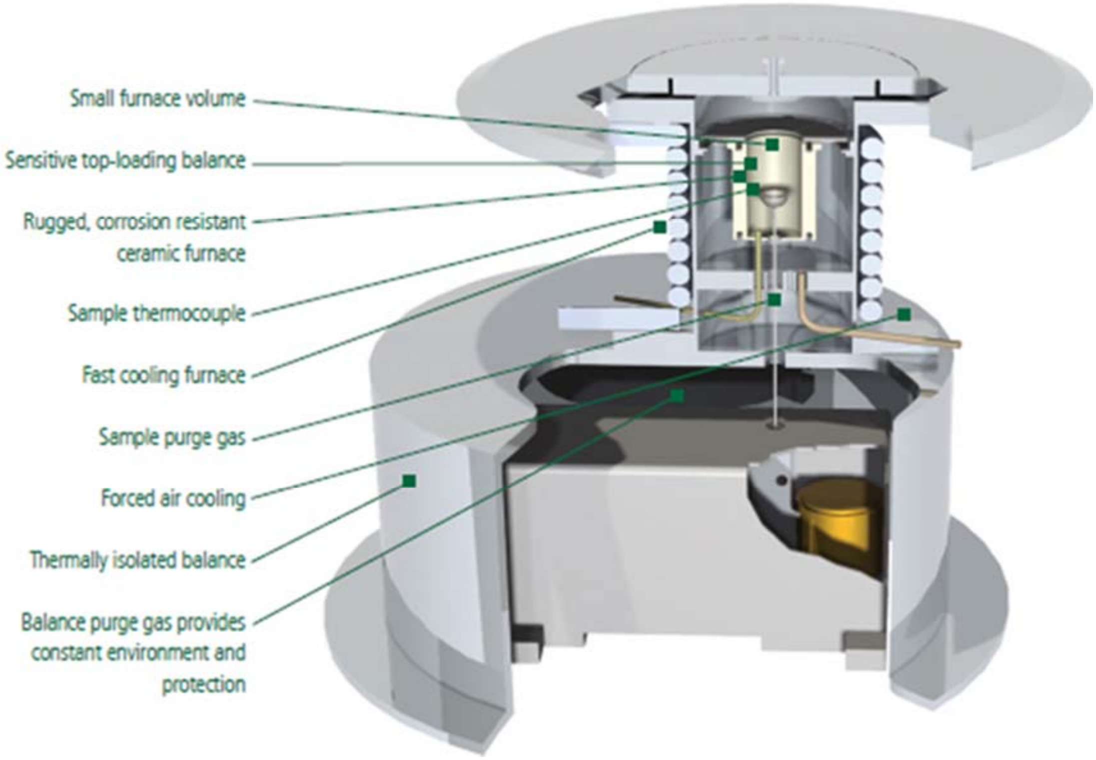


Fig. (3-11) recording thermobalance

3-3-3 Thermomechanical analysis (TMA):

All of the thermomechanical experiments (expansion and penetration mode) presented in this work carried out by LINSEIS, TMA PT1000/German origin. Preparation of samples as cylinder with approximate dimensions of (20 mm height and 5 mm diameter). The samples were securely place on the sample holder and a (50mN) force was applied to the sample in the penetration mode only, followed by a heating rate of 5˚C/min from ambient up to 120˚C.

In addition, the temperature controller was linked to the software governing the mechanical movement of the test frame. This connection allowed the thermal and mechanical profiles to be interconnected and required less real-time control by the operator. Figure (3-12) shows this instrument.

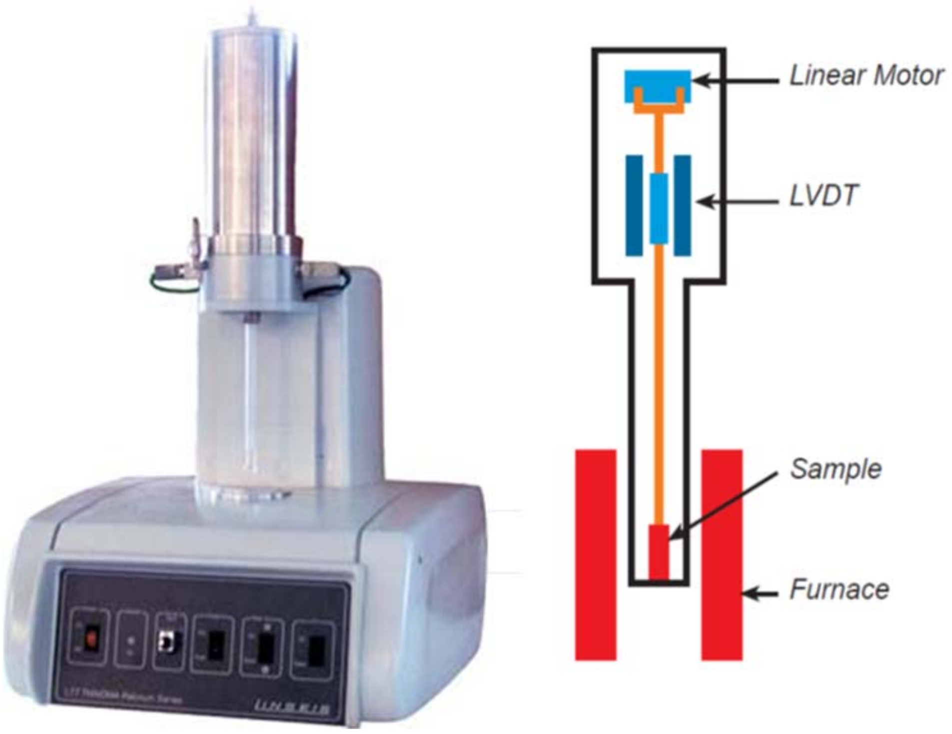


Fig. (3-12) TMA test instrument, (LVDT) linear voltage differential transformer [75]

Chapter Four

Results and Discussion

4-1 Mechanical Tests :

4-1-1 Tensile test:

Stress-strain curves of PVC pipes (K.S.A, Egypt) and doors (Firat, Nova) are shown in figures (4-1) and (4-2) respectively.

From these tensile curves for PVC (pipes and doors), we can get young's modulus, ultimate tensile strength, elongation at max. and elongation at break, which display in tables (4-1) and (4-2).

From figure (4-1) and table (4-1), we can show that K.S.A pipe has higher young's modulus and higher tensile strength than Egypt pipe. While the Egypt pipe has higher value of elongation at break than K.S.A pipe and the elongation at max. are approximately equal. This is due to elongation at break decreases with the increase of filler/stabilizer content [27].

It is quite clear that Egypt pipe has large area under the stress-strain curve compared with K.S.A pipe as shown in fig. (4-3) that means Egypt pipe has much toughness than K.S.A pipe which are in agreement with [62], [68].

From figure (4-2) and table (4-2), the Firat door overcomes clearly on the Nova door in all tensile properties. Firat has much toughness than Nova sample, as shown in fig. (4-4).

All results of the tensile properties are in good agreement with their values in literatures [76].

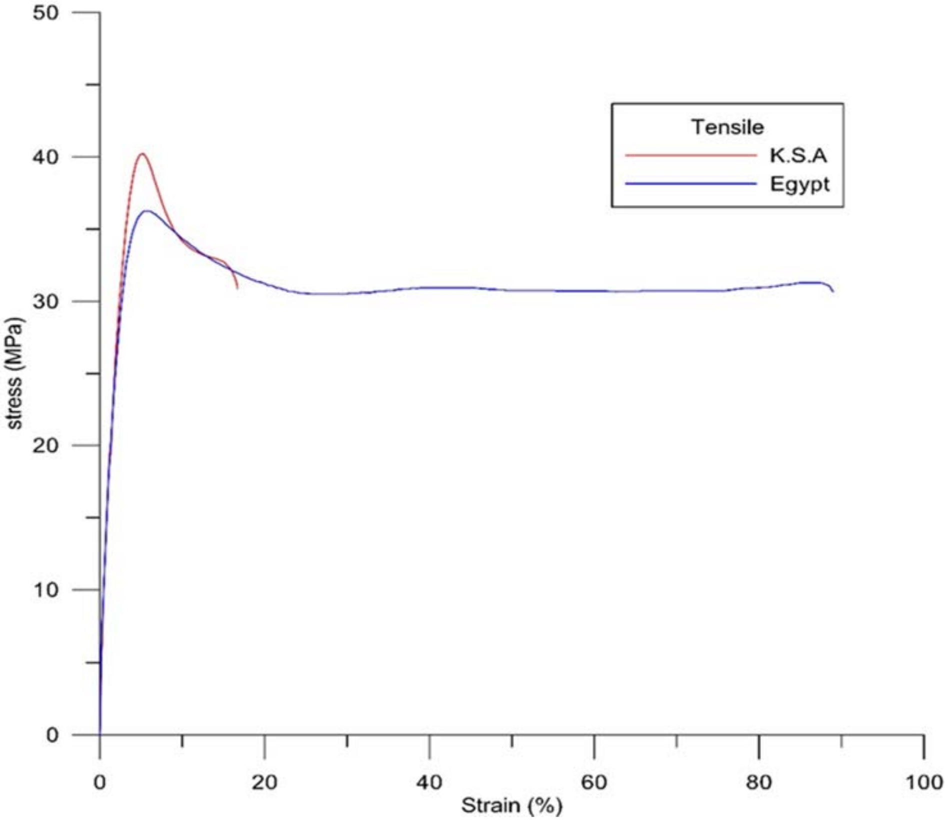


Fig. (4-1) stress-strain curves of K.S.A and Egypt pipes

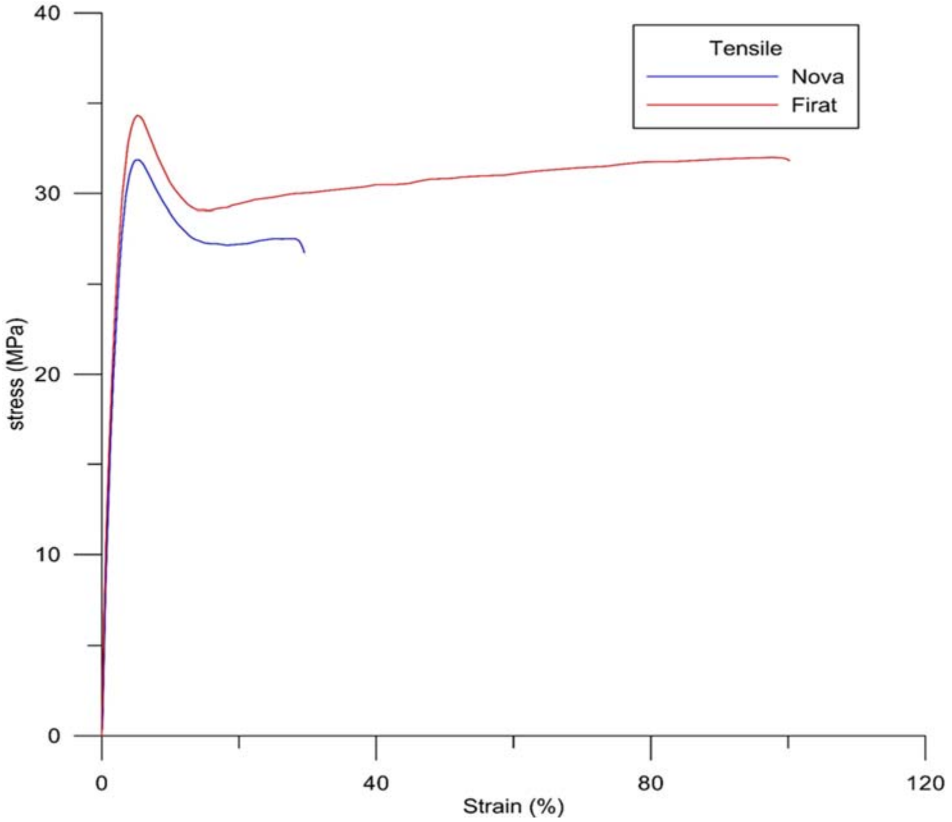


Fig. (4-2) stress-strain curves of Firat and Nova doors

Table (4-1) tensile properties of K.S.A Egypt pipes

NO.	Tensile properties	K.S.A.	Egypt
1	Young's Modulus (MPa)	1588.785	1053.361
2	Ultimate Tensile Strength (MPa)	40.19	36.25
3	Elongation at max (%)	4.87	5.19
4	Elongation at Break (%)	16.68	89.014

Table (4-2) tensile properties of Firat and Nova doors

NO.	Tensile properties	Firat	Nova
1	Young's Modulus (MPa)	1246.827	1214.642
2	Ultimate Tensile Strength (MPa)	34.5	32.25
3	Elongation at max (%)	5.456	5
4	Elongation at Break (%)	100.18	28.268

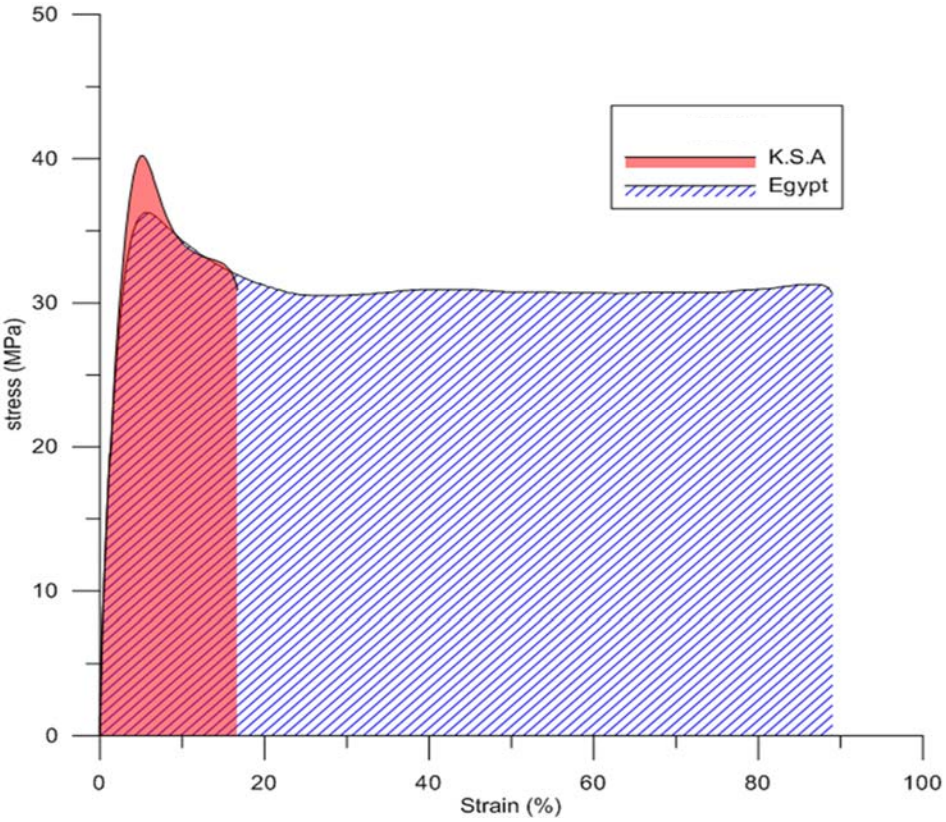


Fig. (4-3) the toughness comparison of K.S.A and Egypt pipes

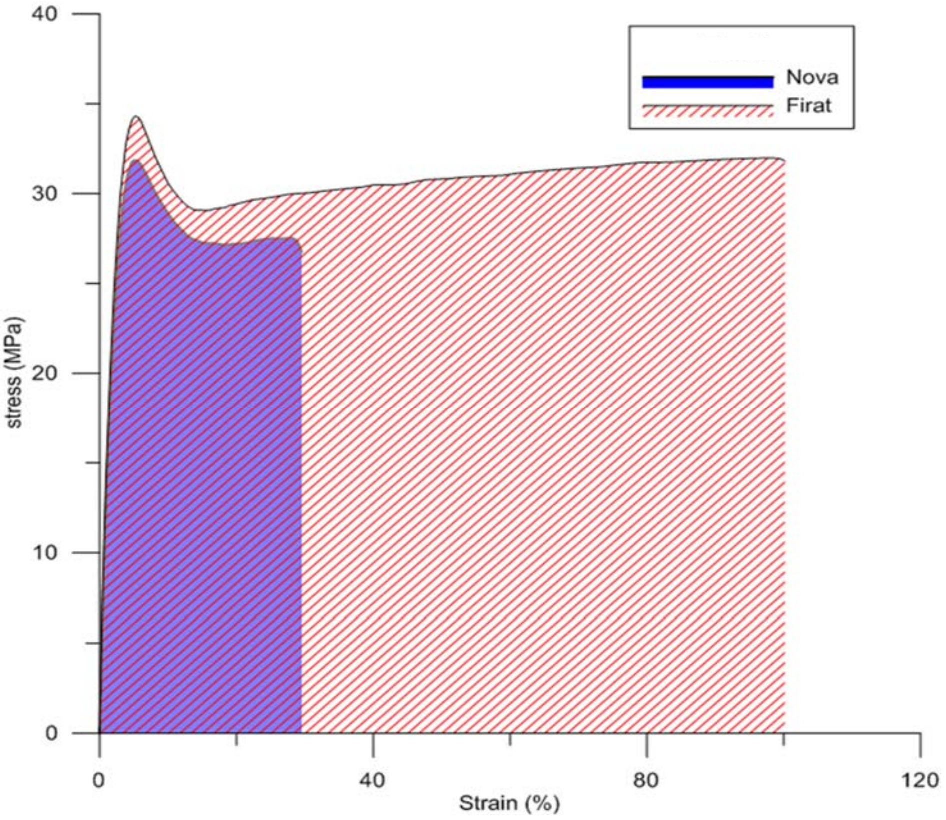


Fig. (4-4) the toughness comparison of Firat and Nova doors

4-1-2 Bending test:

From figures (4-5) and (4-6) the flexural strength is determined, using the relation (2-1).

Table (4-3) displays the values of flexural strength of pipes and doors. The flexural strength is about (76 MPa) for the two kinds of pipes, which agreement with [77].

The Firat door has higher flexural strength (56.6 MPa) than Nova door (54.8 MPa). This difference in the flexural strength because there is difference in the bonding between the chains [78].

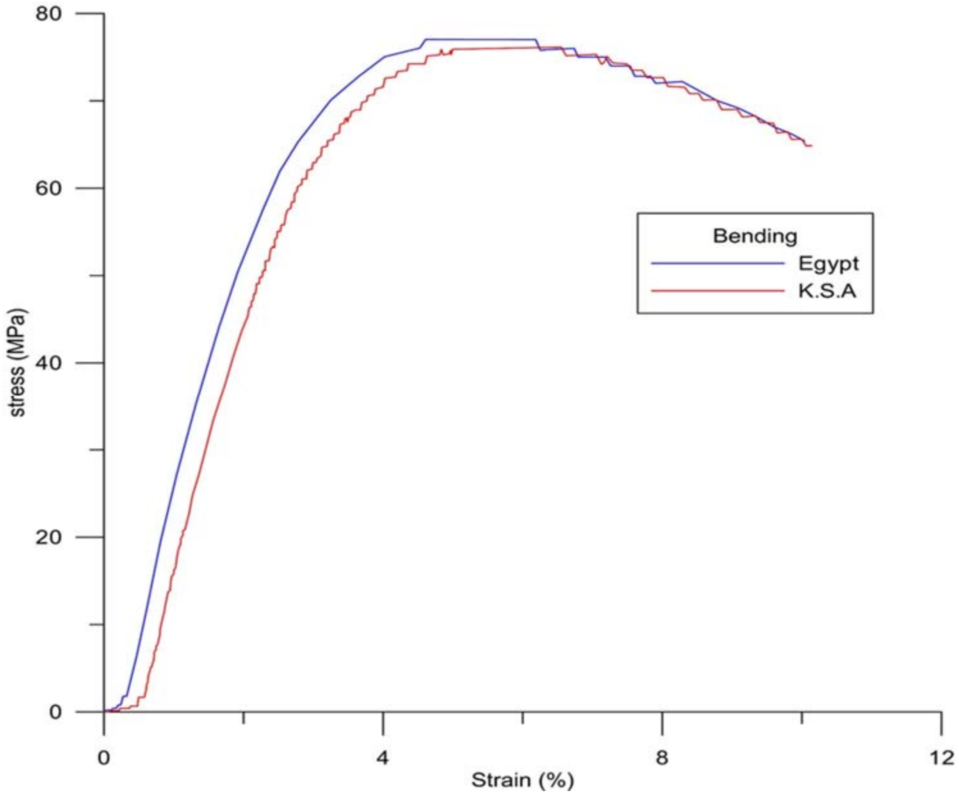


Fig. (4-5) the comparison of bending stress-strain curve of K.S.A and Egypt pipes

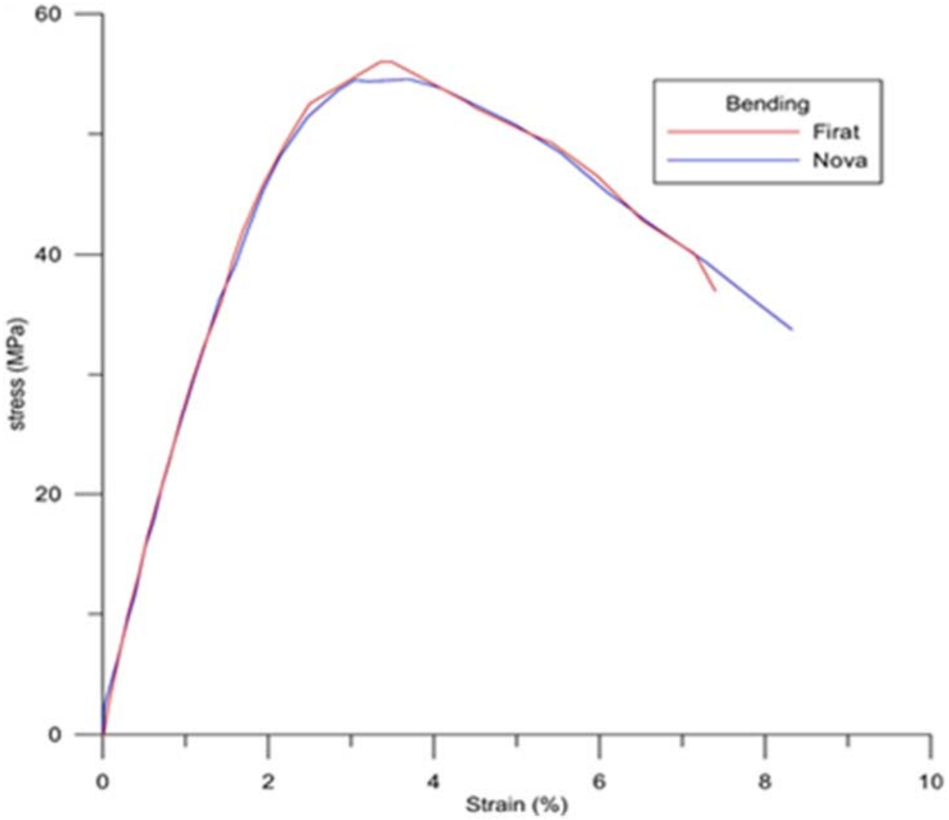


Fig. (4-6) the comparison of bending stress-strain curve of K.S.A and Egypt pipes

Table (4-3) Flexural strength of pipes and doors

NO.	Sample	Flexural strength (MPa)
1	K.S.A.	76
2	Egypt	76.55
3	Firat	56.6
4	Nova	54.8

4-1-3 Impact Test:

The impact properties of materials are directly related to overall toughness, the impact strength of samples are determined by using eq. (2-2).

The table (4-4) shows that Egypt pipe has impact strength value (14.83 KJ/m^2) higher than that of K.S.A (11.18 KJ/m^2), this is natural, because of the area under stress-strain curve (fig. (4-3)) is greater for Egypt pipe than that K.S.A.

The Firat door has impact strength value (30.8 KJ/m^2) more than Nova door (21.7 KJ/m^2), because of the area under stress- strain curve (fig. (4-4)) is greater for Firat door than Nova.

All the values of impact strength of pipes and doors in table (4-4) are good agreement with [79].

Table (4-4) Comparison between Impact strength of different PVC samples.

NO.	Specimen	Impact strength (KJ/m^2)
1	K.S.A.	11.1898
2	Egypt	14.832
3	Firat	30.883
4	Nova	21.780

4-1-4 Hardness Shore D:

The hardness of Egypt pipe is equal to that of two types of doors (Firat and Nova) but the hardness of K.S.A pipe has a higher value than other types. Table (4-5) illustrates the values of hardness, which are in agreement with [27].

The K.S.A pipe has greater tensile strength among them, where the tensile strength is relevant to the hardness by direct proportion [35], [40].

Table (4-5) Hardness Comparison shore D of different PVC samples

NO.	Specimen	Shore D
1	K.S.A.	83.7
2	Egypt	80.8
3	Firat	80.7
4	Nova	80

4-2 Thermal Properties

4-2-1 Thermal Conductivity:

From Table (4-6), it can be noticed, that the average value of the thermal conductivity of this material is less than (0.45W/m.°C) which reflects the high resistance of those materials to heat transfer. This means, their suitability would be used as thermal insulators, this low thermal conductivity is due to the fact that these polymers have no free electrons and low phonon velocity [59].

The table (4-6) shows that Firat has thermal conductivity value higher than Nova. The results of thermal conductivity of PVC are in agreement with [80].

Table (4-6) Thermal conductivity of doors

Type	Thermal conductivity (W/m.˚C)
Firat	0.21525
Nova	0.19545

4-2-2 Thermal stability and Degradation:

TGA curves of PVC pipes and doors are shown in figures (4-7), (4-8), (4-9) and (4-10) respectively, at different heating rates (2, 5, 10, 20 and 40 ˚C/min). From these curves, we can:

i. **Identify the kind of polymer** from their decomposition pattern [81].
ii. **Thermal stability:**

The reaction is characterized by two temperatures, T_i and T_f, which are called the procedural decomposition temperature and the final temperature, respectively.

According to Figures (4-7) to (4-10), the weight loss curve has been shifted to the higher temperatures when the heating rate is increased. In all experiments, final temperature was 800 ˚C, from 240 – 350 ˚C the polymer degrade but above 350 ˚C the sample degrade with 50% [82], although the decomposition curves of PVC pipes and doors are close to each other at the heating rate range of (5-20 ˚C/min). Decomposition takes place above 250 ˚C; the sample first suffers a loss of about 2% due to moisture and then, as expected decomposes in two steps [83]. At high heating rate, fast pyrolysis was observed [84].

Figures (4-11) (4-12), (4-13), (4-14) and (4-15) show that the Egypt pipe has higher thermal stability than K.S.A pipe at different heating rates.

Figures (4-16), (4-17), (4-18) and (4-19) show that Firat door has higher thermal stability than the Nova door at different heating rates.

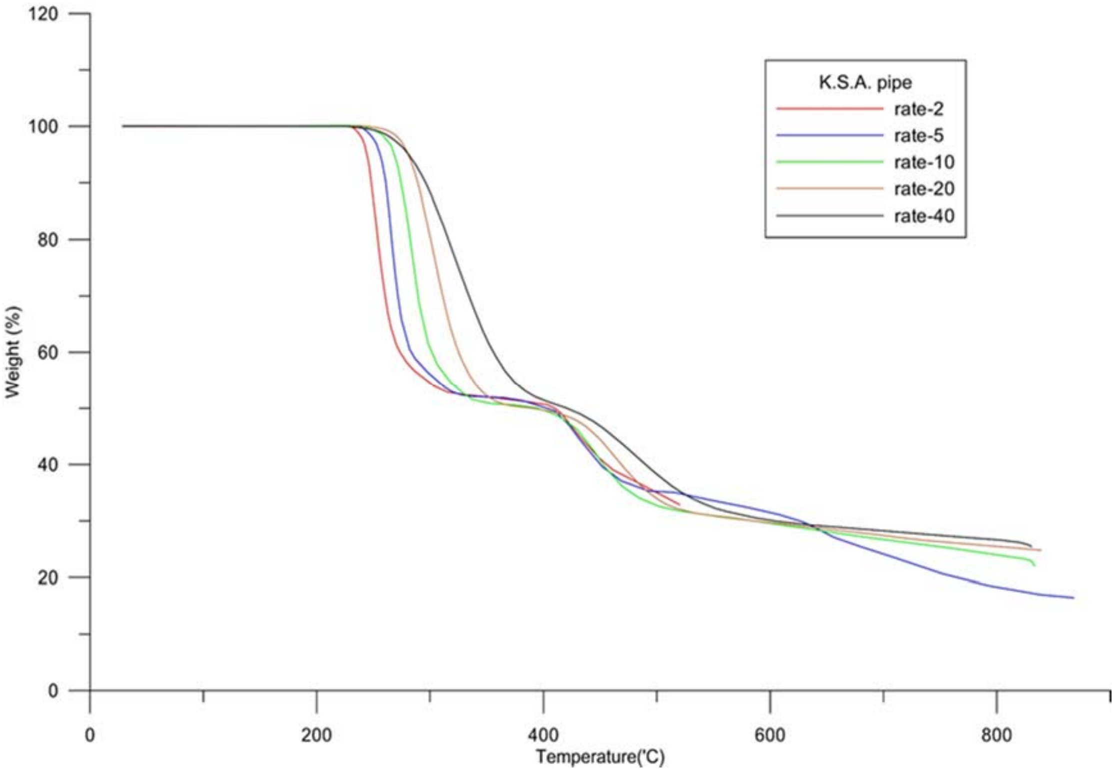


Fig. (4-7) the relation of weight% VS temp. of K.S.A pipe at different heating rates

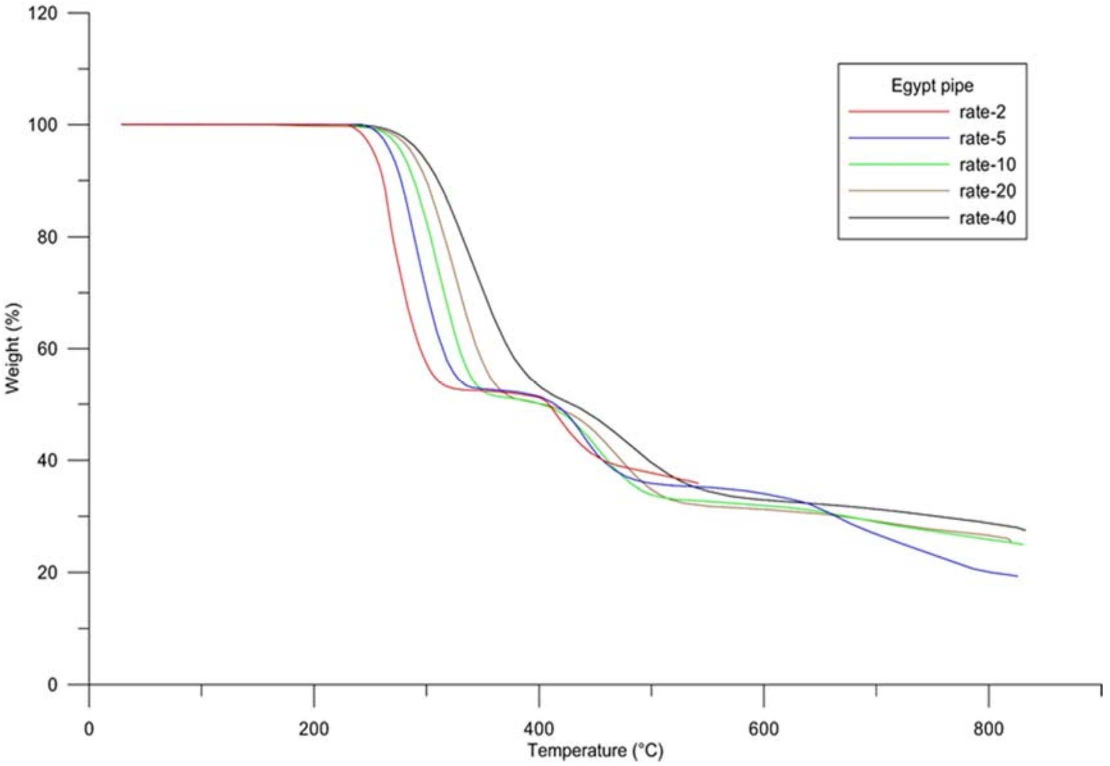


Fig. (4-8) the relation of weight% VS temp. of Egypt pipe at different heating rates

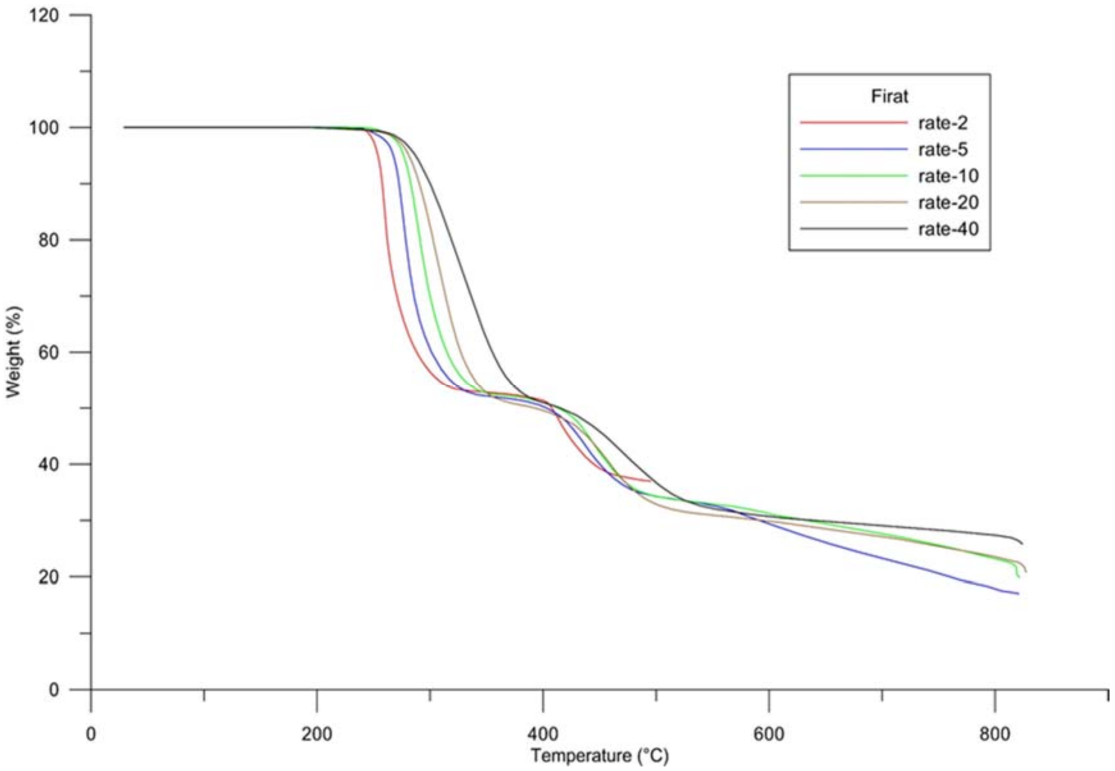


Fig. (4-9) the relation of weight% VS temp. of Firat door at different heating rates

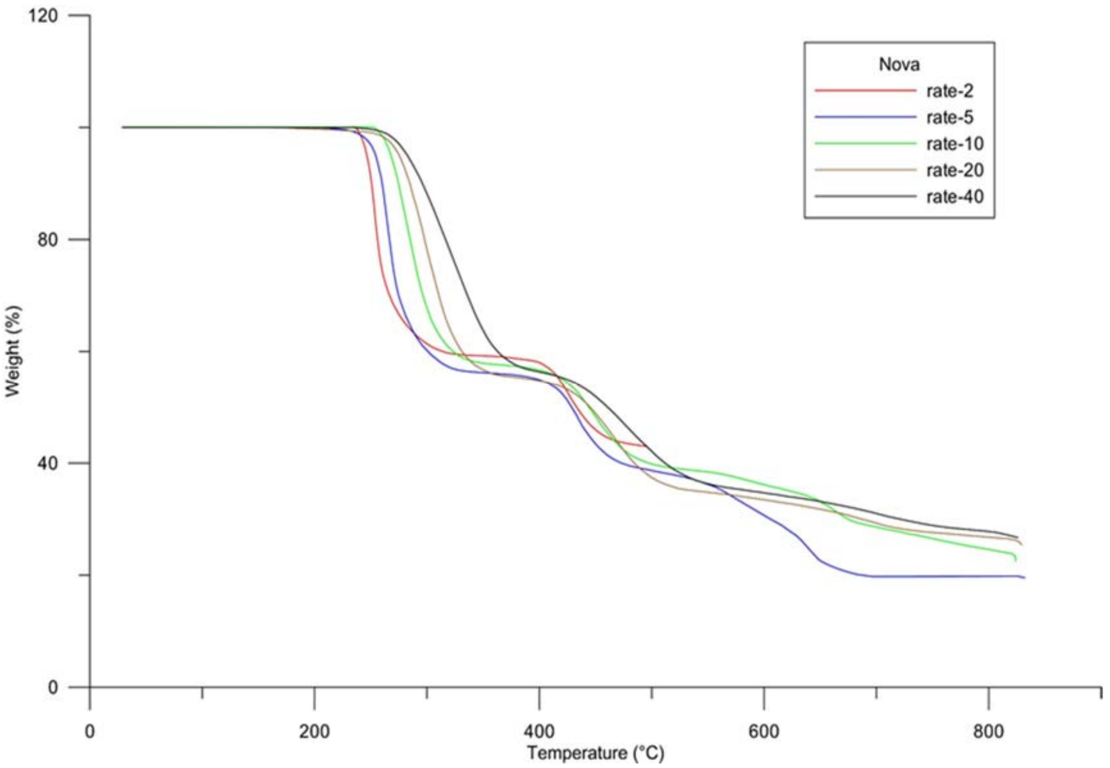


Fig. (4-10) the relation of weight% VS temp. of Nova door at different heating rates

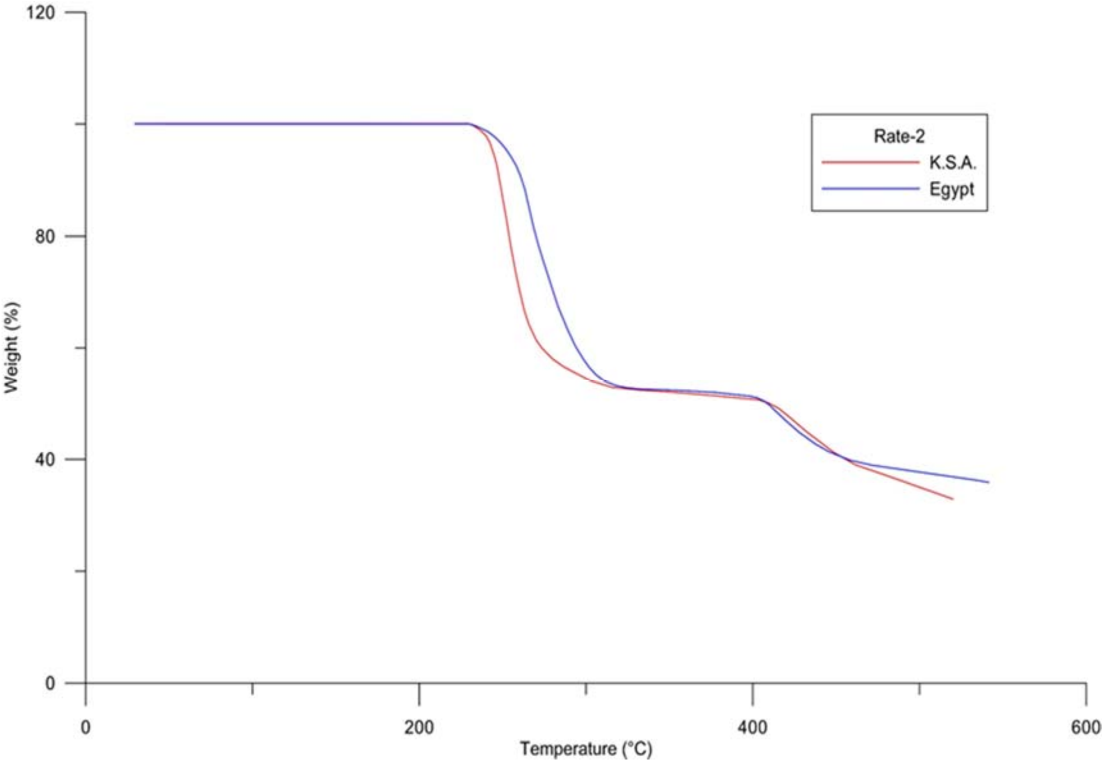


Fig. (4-11) the thermal stability of K.S.A and Egypt pipes at heating rate (2 ˚C/min)

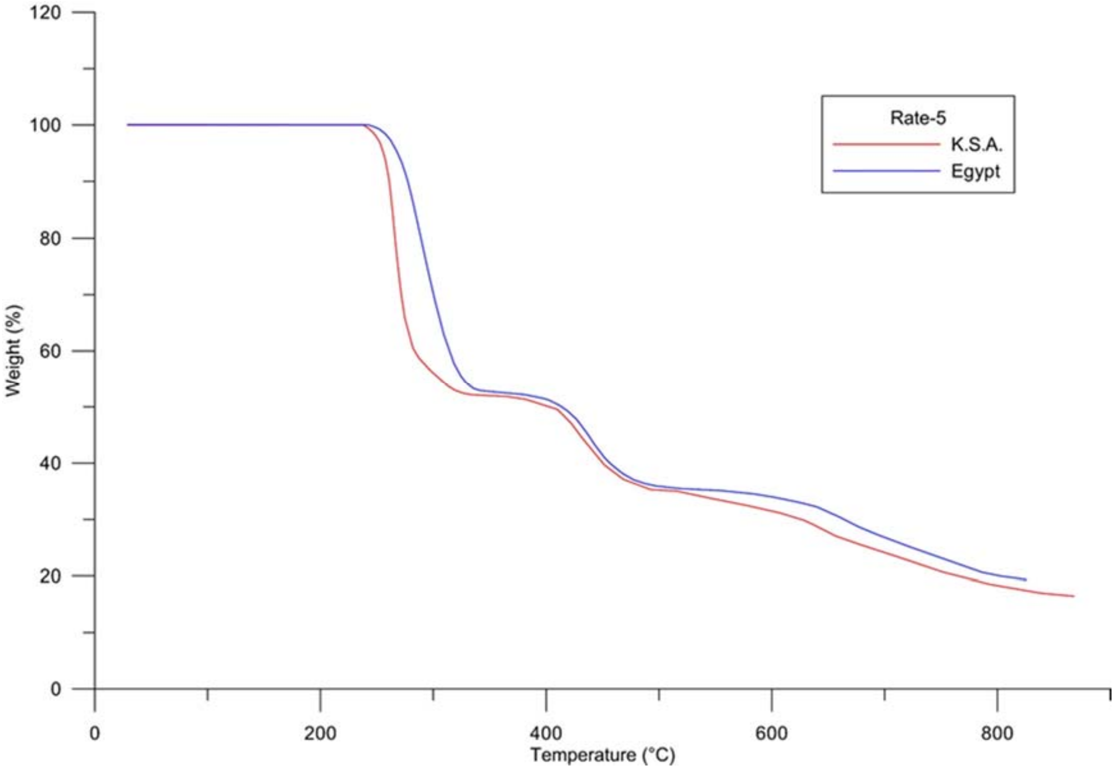


Fig. (4-12) the thermal stability of K.S.A and Egypt pipes at heating rate (5 ˚C/min)

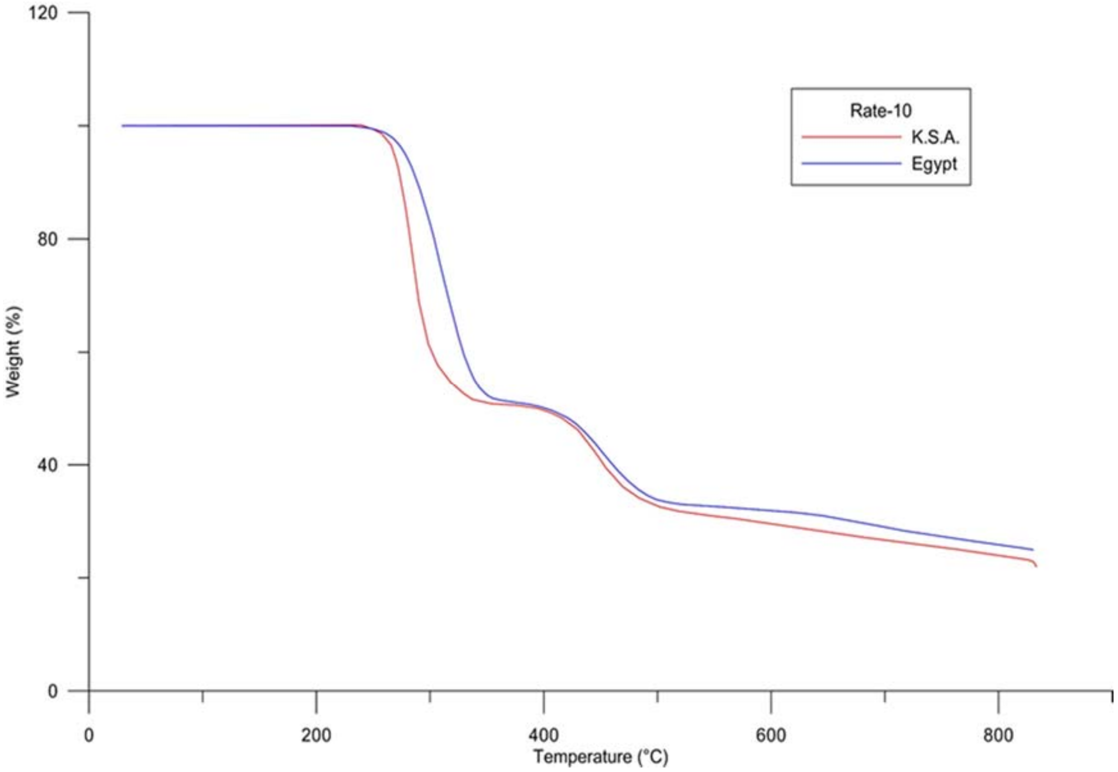


Fig. (4-13) the thermal stability of K.S.A and Egypt pipes at heating rate (10 ˚C/min)

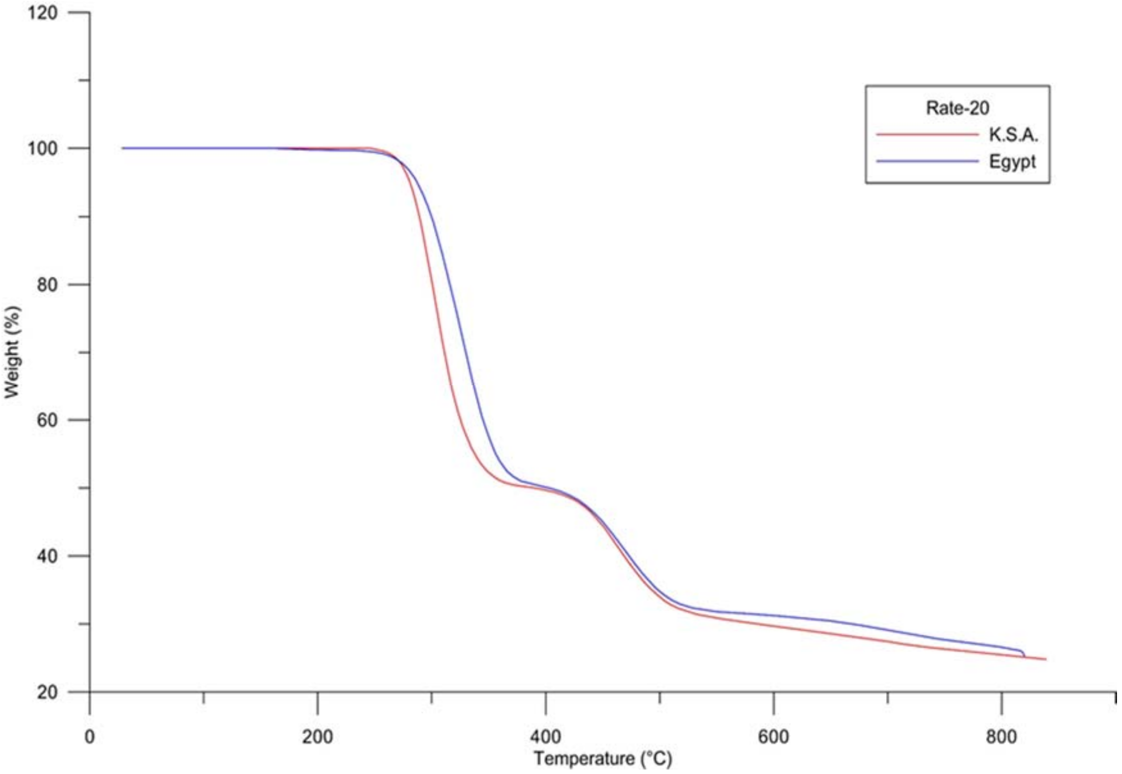


Fig. (4-14) the thermal stability of K.S.A and Egypt pipes at heating rate (20 ˚C/min)

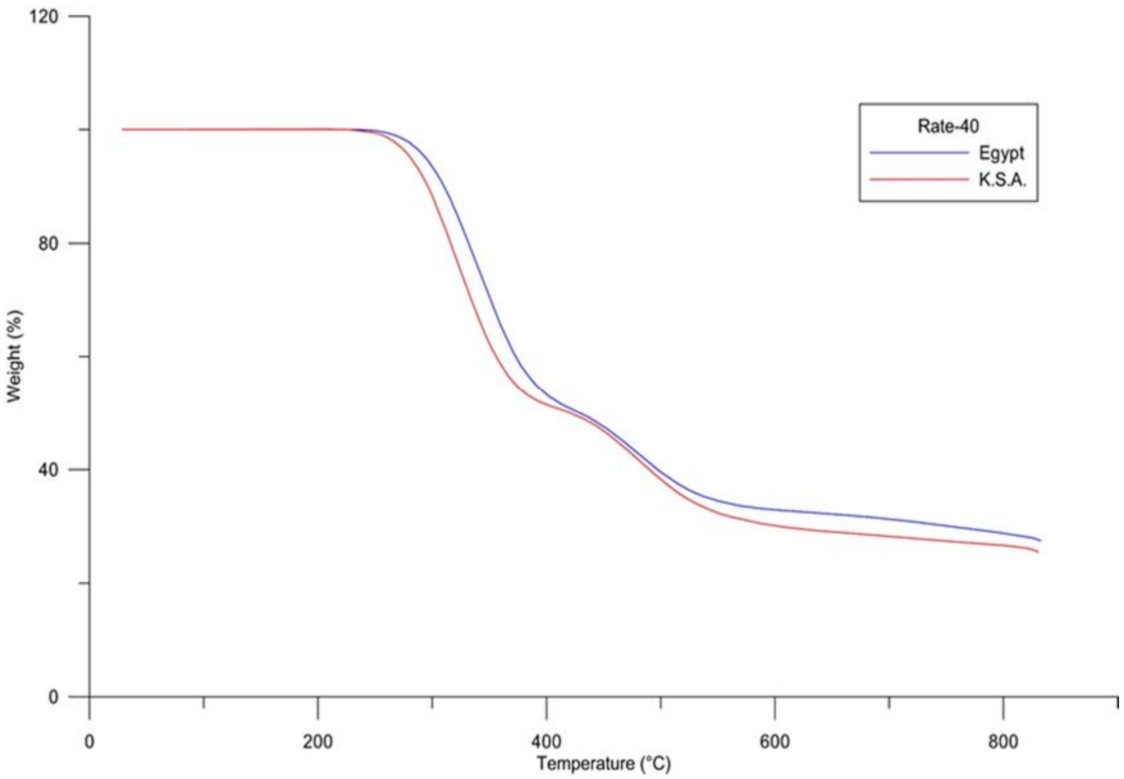


Fig. (4-15) the thermal stability of K.S.A and Egypt pipes at heating rate (40 ˚C/min)

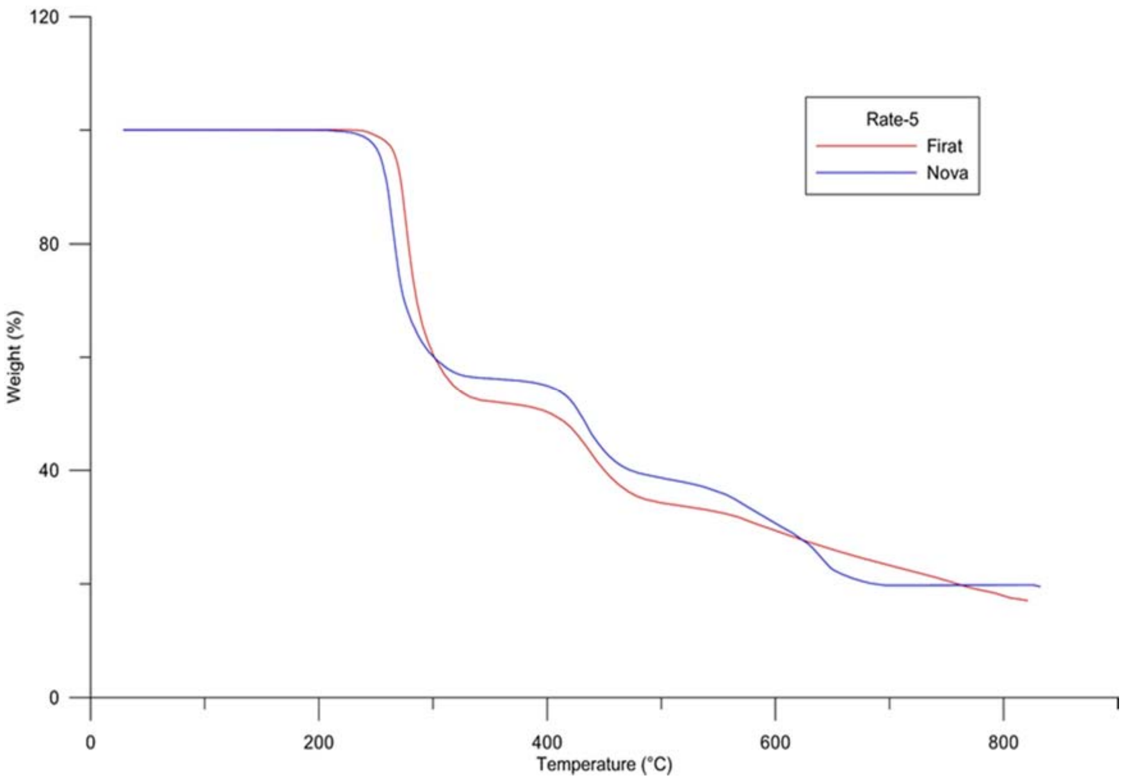


Fig. (4-16) the thermal stability of Firat and Nova doors at heating rate (5 ˚C/min)

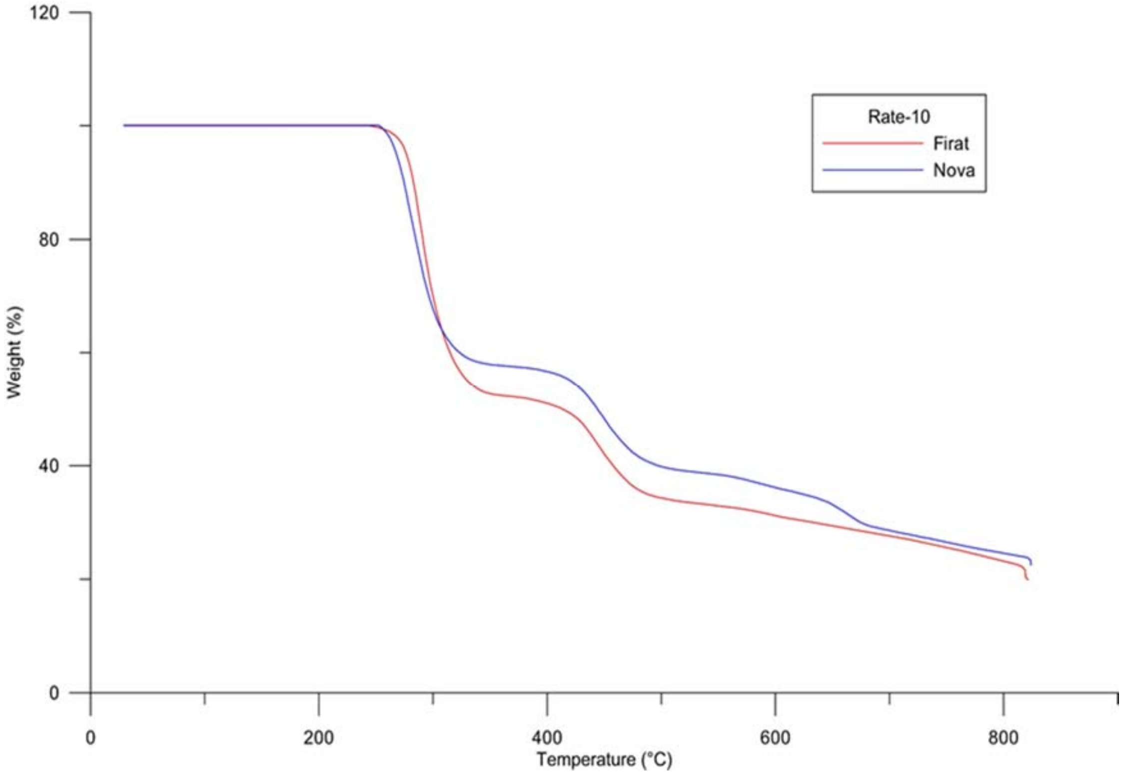


Fig. (4-17) the thermal stability of Firat and Nova doors at heating rate (10 ˚C/min)

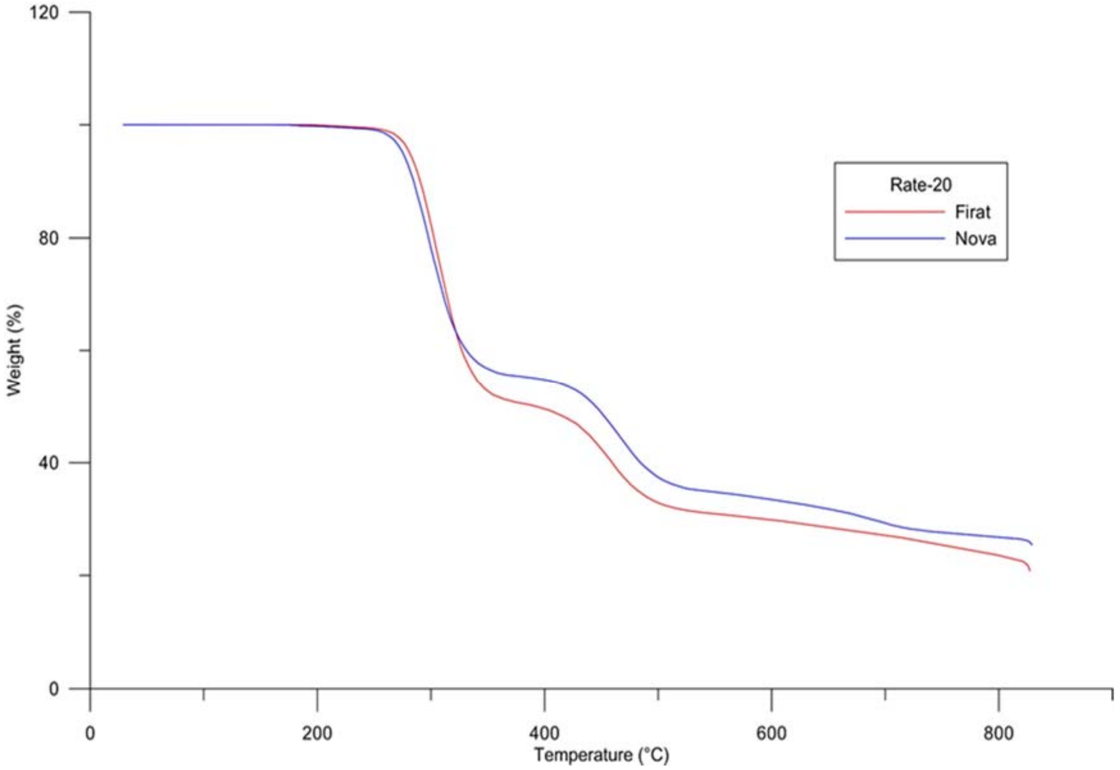


Fig. (4-18) the thermal stability of Firat and Nova doors at heating rate (20 ˚C/min)

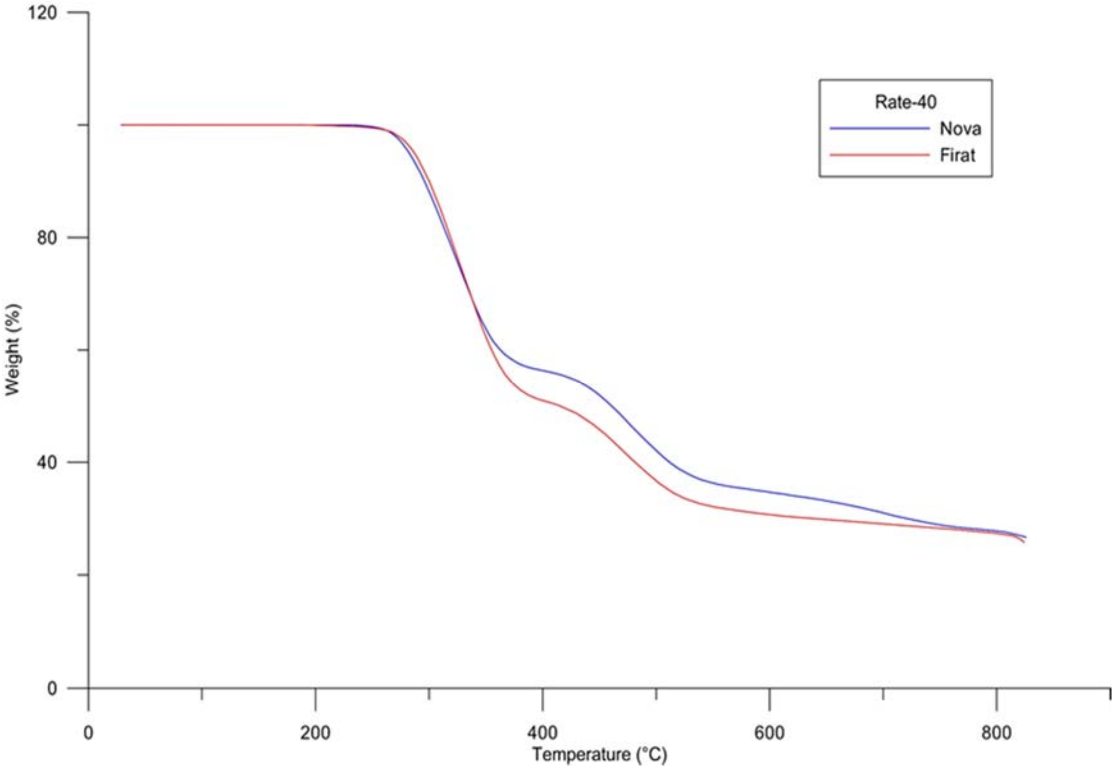


Fig. (4-19) the thermal stability of Firat and Nova doors at heating rate (40 ˚C/min)

iii. Activation Energy:

TGA curves are used to determine the kinetics of thermal decomposition of polymer like activation energy of polymer. For estimating the activation energy at certain conversion levels (1%, 2.5%, 5%, 10%, 20%, 30%, 40%), where the weight loss of sample is reduced at (99%, 97.5%, 95%, 90%, 80%, 70% and 60%) respectively. The conversion temperatures are determined for pipes and doors at different conversion levels. As in tables (4-7), (4-8), (4-9), (4-10), where the conversion temperature are increased with increase of the heating rates.

From the slope of figures (4-20) and (4-21) for pipes and figures (4-22) and (4-23) for doors, with using equation (2-12) we estimate the activation energy of pipes and doors at different conversion levels.

When the value of slopes would be nearly constant, the conversion level is chosen to determine the activation energy, because the mechanism of degradation becomes stable.

As shown in tables (4-11) and (4-12) for pipes and (4-13) and (4-14) for doors, the results are in good agreement with the values of activation energy at (30%) with [85].

Table (4-7) conversion levels of K.S.A pipe

conversion	β (˚C/min)	T_c (˚C)	T_c(K)	$10^3/T_c(K^{-1})$
1%	2	237.2	510.7	1.958097
	5	244.8	518.3	1.929385
	10	251.72	525.22	1.903964
	20	262.93	536.43	1.864176
	40	251.72	525.22	1.903964
2.5%	2	241.86	515.36	1.940391
	5	250	523.5	1.91022
	10	260.34	533.84	1.87322
	20	272.41	545.91	1.831804
	40	268.96	542.46	1.843454
5%	2	245.34	518.84	1.927376
	5	255.17	528.67	1.891539
	10	265.51	539.01	1.855253
	20	277.58	551.08	1.814619
	40	279.31	552.81	1.80894
10%	2	248.837	522.337	1.914473
	5	260.34	533.84	1.87322
	10	272.41	545.91	1.831804
	20	287.06	560.56	1.78393
	40	295	568.5	1.759015
20%	2	255.81	529.31	1.889252
	5	265.517	539.017	1.855229
	10	279.31	552.81	1.80894
	20	298.27	571.77	1.748955
	40	313.79	587.29	1.702736
30%	2	261.627	535.127	1.868715
	5	270.689	544.189	1.837597
	10	287.06	560.56	1.78393
	20	308.62	582.12	1.717859
	40	333	606.5	1.648805
40%	2	274.418	547.918	1.825091
	5	282.75	556.25	1.797753
	10	300	573.5	1.743679
	20	321	594.5	1.682086
	40	355.2	628.7	1.590584

Table (4-8) conversion levels of Egypt pipe

conversion	β (˚C/min)	T_c (˚C)	T_c (K)	$10^3/T_c(K^{-1})$
1%	2	237.9	511.4	1.955417
	5	251.72	525.22	1.903964
	10	260.34	533.84	1.87322
	20	262.06	535.56	1.867204
	40	265.517	539.017	1.855229
2.5%	2	248.83	522.33	1.914498
	5	260.34	533.84	1.87322
	10	267.24	540.74	1.849318
	20	272.41	545.91	1.831804
	40	279.31	552.81	1.80894
5%	2	255.81	529.31	1.889252
	5	265.517	539.017	1.855229
	10	279.31	552.81	1.80894
	20	284.48	557.98	1.792179
	40	291.38	564.88	1.770287
10%	2	263.95	537.45	1.860638
	5	274.13	547.63	1.82605
	10	287.93	561.43	1.781166
	20	298.27	571.77	1.748955
	40	306.89	580.39	1.722979
20%	2	270.93	544.43	1.836783
	5	286.206	559.706	1.786652
	10	301.72	575.22	1.738465
	20	313.79	587.29	1.702736
	40	329.31	602.81	1.658897
30%	2	279	552.5	1.809955
	5	301.75	575.25	1.738375
	10	321	594.5	1.682086
	20	335.08	608.58	1.643169
	40	356.14	629.64	1.588209
40%	2	297.67	571.17	1.750792
	5	310.34	583.84	1.712798
	10	327.58	601.08	1.663672
	20	341.379	614.879	1.626336
	40	375.86	649.36	1.539978

Table (4-9) conversion levels of Firat door

conversion	β (˚C/min)	T_c (˚C)	T_c (K)	$10^3/T_c(K^{-1})$
1%	5	248.28	521.78	1.916517
	10	262	535.5	1.867414
	20	255.17	528.67	1.891539
	40	256	529.5	1.888574
2.5%	5	260.35	533.85	1.873185
	10	268.97	542.47	1.84342
	20	272.41	545.91	1.831804
	40	274.14	547.64	1.826017
5%	5	267.24	540.74	1.849318
	10	275.86	549.36	1.8203
	20	279.31	552.81	1.80894
	40	282.76	556.26	1.79772
10%	5	272.41	545.91	1.831804
	10	281.72	555.22	1.801088
	20	287.93	561.43	1.781166
	40	298.28	571.78	1.748924
20%	5	278.28	551.78	1.812317
	10	286.21	559.71	1.78664
	20	301.72	575.22	1.738465
	40	318.28	591.78	1.689817
30%	5	284.48	557.98	1.792179
	10	298.28	571.78	1.748924
	20	312.1	585.6	1.70765
	40	336.21	609.71	1.640124
40%	5	300	573.5	1.743679
	10	315.52	589.02	1.697735
	20	327.59	601.09	1.663644
	40	355.17	628.67	1.59066

Table (4-10) conversion levels of Nova door

conversion	β (˚C/min)	T_c (˚C)	T_c(K)	$10^3/T_c(K^{-1})$
1%	5	231	504.5	1.982161
	10	258.62	532.12	1.879275
	20	244.82	518.32	1.92931
	40	253.79	527.29	1.89649
2.5%	5	250	523.5	1.91022
	10	263.79	537.29	1.861192
	20	265.51	539.01	1.855253
	40	272.41	545.91	1.831804
5%	5	255.17	528.67	1.891539
	10	267.24	540.74	1.849318
	20	274.14	547.64	1.826017
	40	279.31	552.81	1.80894
10%	5	260.34	533.84	1.87322
	10	274.14	547.64	1.826017
	20	283.62	557.12	1.794945
	40	294.83	568.33	1.759541
20%	5	265.52	539.02	1.855219
	10	284.48	557.98	1.792179
	20	296.55	570.05	1.754232
	40	315.52	589.02	1.697735
30%	5	275.86	549.36	1.8203
	10	296.55	570.05	1.754232
	20	308.62	582.12	1.717859
	40	332.76	606.26	1.649457
40%	5	300	573.5	1.743679
	10	325.86	599.36	1.668446
	20	331.03	604.53	1.654178
	40	365.51	639.01	1.564921

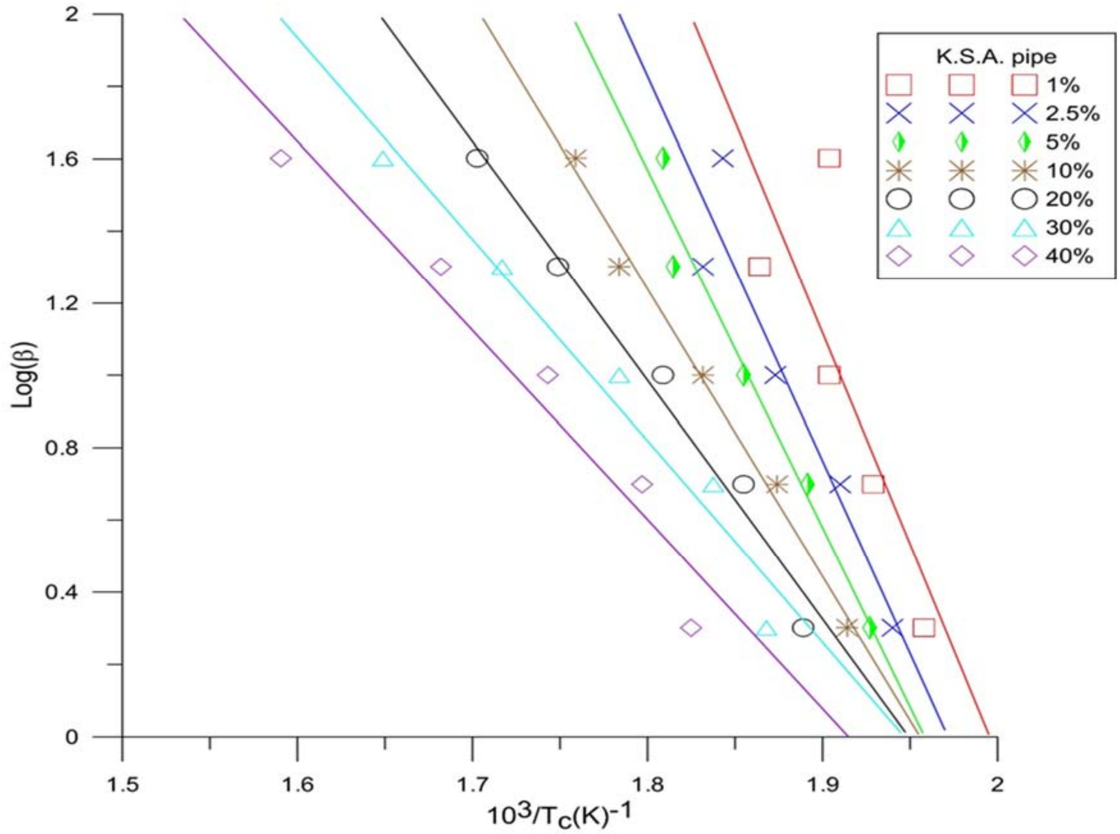


Fig. (4-20) temperature dependance of Logβ VS reciprocal absolute temperature for various weight loss for K.S.A pipe

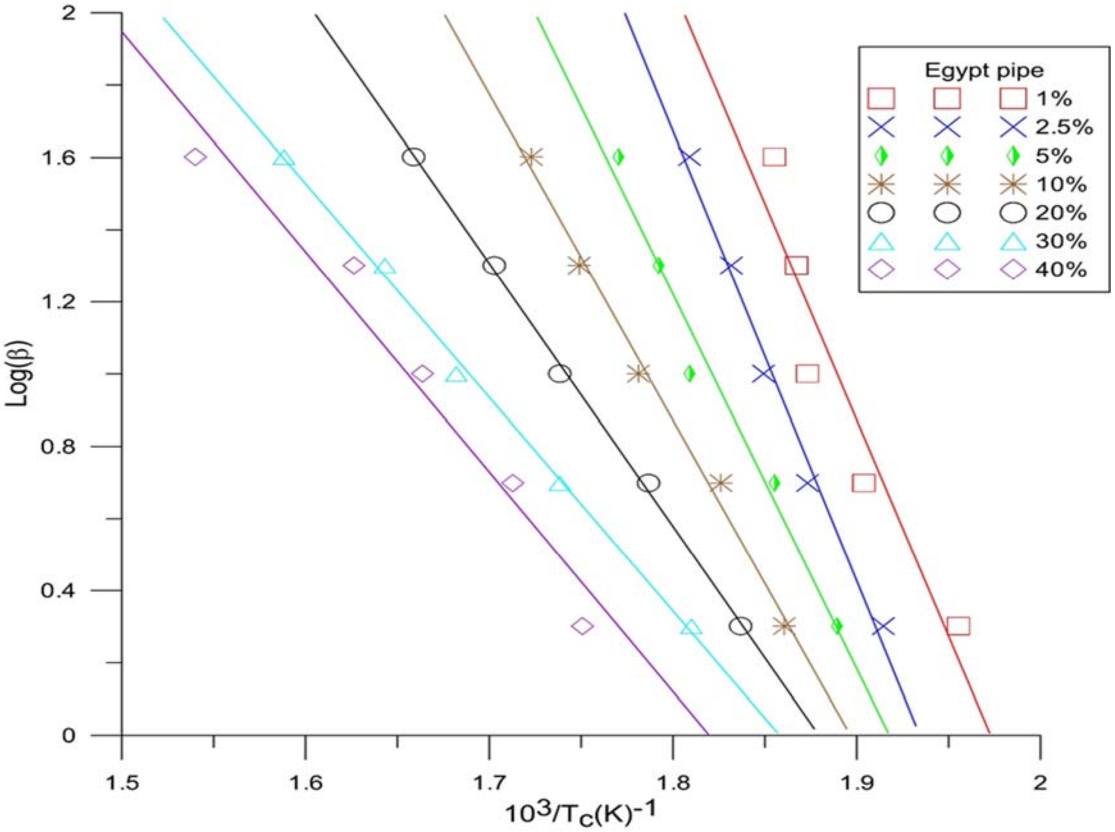


Fig. (4-21) temperature dependance of Logβ VS reciprocal absolute temperature for various weight loss for Egypt pipe

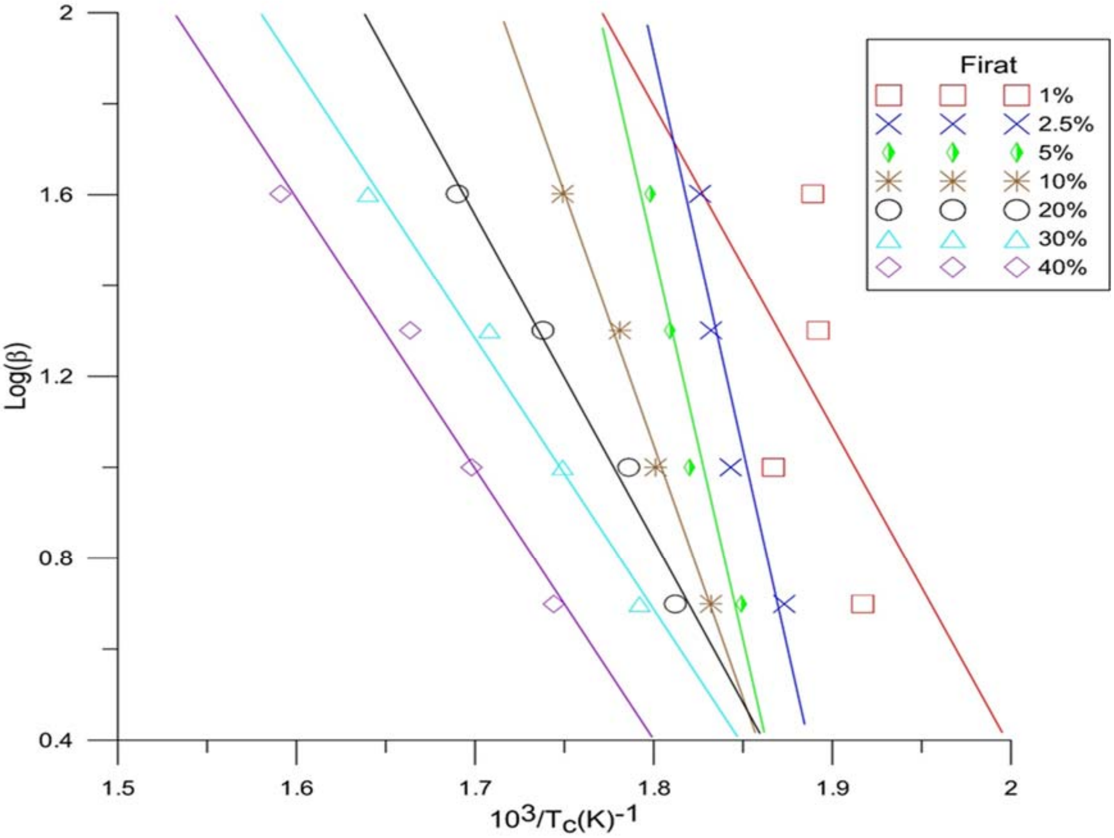


Fig. (4-22) temperature dependance of Logβ VS reciprocal absolute temperature for various weight loss for Firat door

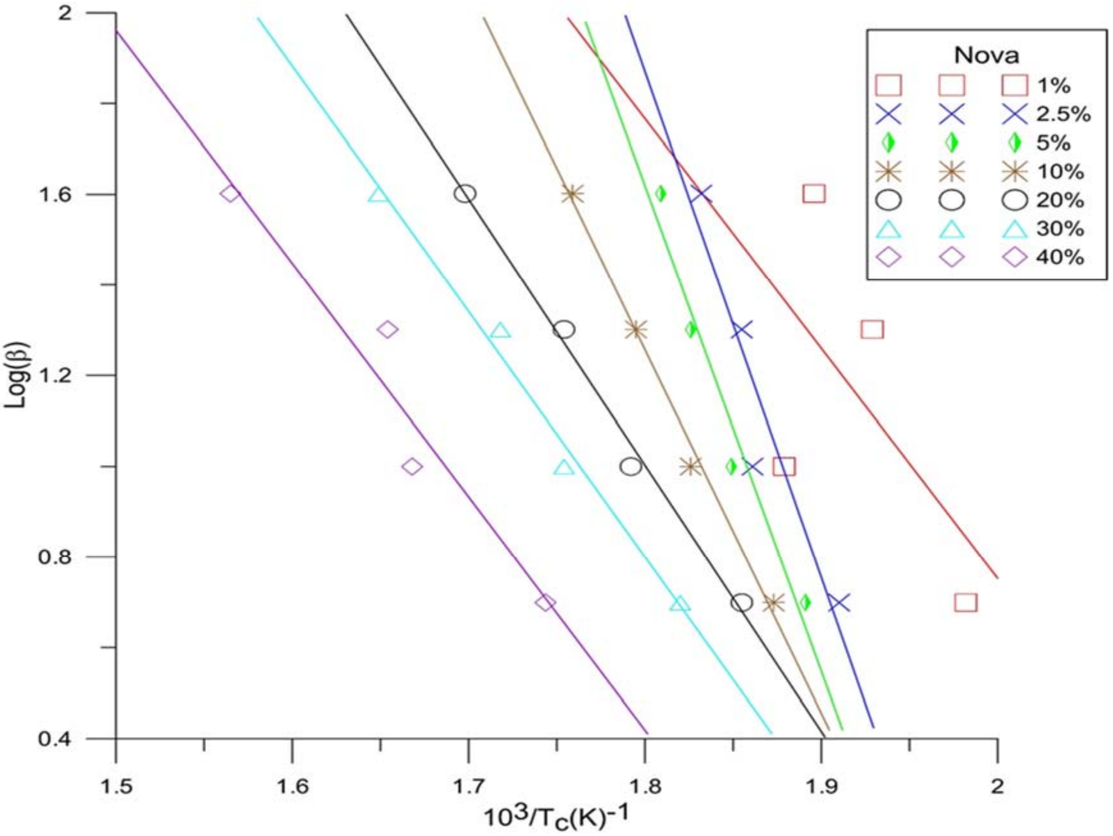


Fig. (4-23) temperature dependance of Logβ VS reciprocal absolute temperature for various weight loss for Nova door

Table (4-11) the values of activation energy of K.S.A pipe at different conversion levels

Conversion %	slope	Ea (KJ/mole)
1	-11.69	212.489
2.5	-10.64	193.403
5	-9.905	180.043
10	-7.956	144.616
20	-6.607	120.095
30	-5.578	101.39
40	-5.23	95.06

Table (4-12) the values of activation energy of Egypt pipe at different conversion levels

Conversion %	slope	Ea (KJ/mole)
1	-12.02	218.487
2.5	-12.51	227.39
5	-10.36	188.313
10	-9.06	164.68
20	-7.254	131.85
30	-5.961	108.35
40	-6.102	110.91

Table (4-13) the values of activation energy of Firat door at different conversion levels

Conversion %	slope	Ea (KJ/mole)
1	-7.067	128.45
2.5	-17.47	317.55
5	-17.12	311.19
10	-11.11	201.94
20	-7.15	129.96
30	-5.961	108.35
40	-5.954	108.225

Table (4-14) the values of activation energy of Nova door at different conversion levels

Conversion %	slope	Ea (KJ/mole)
1	-5.069	92.139
2.5	-11.15	202.67
5	-10.70	194.49
10	-8.010	145
20	-5.867	106.64
30	-5.411	98.35
40	-5.14	93.466

iv. Lifetime predictions:

The aging of polymers implies some type of degradation process usually involving the breaking of covalent bonds [82].

At conversion level 30% of the lifetime are estimated for pipes and doors by using eq.

$$t = \frac{Tc - Ti}{\beta} \quad \ldots\ldots\ldots\ldots\ldots (4\text{-}1)$$

Where T_c is conversion temperature.

T_i is procedural temperature.

t is time degradation at Tc

Tables (4-15), (4-16), (4-17) and (4-18) show that the time is decreased with the increase of the heating rates.

From the figures (4-24), (4-25), (4-26) and (4-27) we can determine the lifetime of pipes and doors at different serves temperatures as displayed in table (4-19) and (4-20) for pipes and doors respectively. The lifetime is decreased with increase of temperature [68].

At serves temperatures from 60-80˚C the lifetime of Egypt pipe is better than the K.S.A pipe, and the lifetime of Firat door is better than Nova door.

Table (4-15) time values of first conversion level of K.S.A at different heating rates

β(C°/min)	T_i (C°)	T_c(C°)	t (min)	t (hr)	Log t
2	30.23	261.627	115.698	1.92830	0.285176
5	27.586	270.689	48.620	0.81034	-0.09133
10	27.586	287.06	25.947	0.43245	-0.36406
20	27.586	308.62	14.051	0.23419	-0.63042
40	27.586	333	7.635	0.12725	-0.89532

Table (4-16) time values of first conversion level of Egypt at different heating rates

β(C°/min)	T_i (C°)	T_c(C°)	t (min)	t (hr)	Log t
2	30.23	279	124.385	2.073083	0.316617
5	28.858	301.75	54.578	0.90964	-0.04113
10	27.586	321	29.341	0.489023	-0.31067
20	27.586	335.08	15.374	0.256245	-0.59134
40	27.586	356.14	8.213	0.136898	-0.8636

Table (4-17) time values of first conversion level of Firat at different heating rates

β(C°/min)	T_i (C°)	T_c(C°)	t (min)	t (hr)	Log t
5	27.60	284.48	51.376	0.85626	-0.06739
10	27.60	298.28	27.068	0.45113	-0.3457
20	27.60	312.10	14.225	0.23708	-0.6251
40	27.60	336.21	7.7152	0.12858	-0.89083

Table (4-18) time values of first conversion level of Nova at different heating rates

β(C°/min)	T_i (C°)	T_c(C°)	t (min)	t (hr)	Log t
5	27.60	275.86	49.652	0.8275	-0.0822
10	27.60	296.55	26.895	0.44825	-0.34792
20	27.60	308.62	14.051	0.23418	-0.63045
40	27.62	332.76	7.629	0.12715	-0.89568

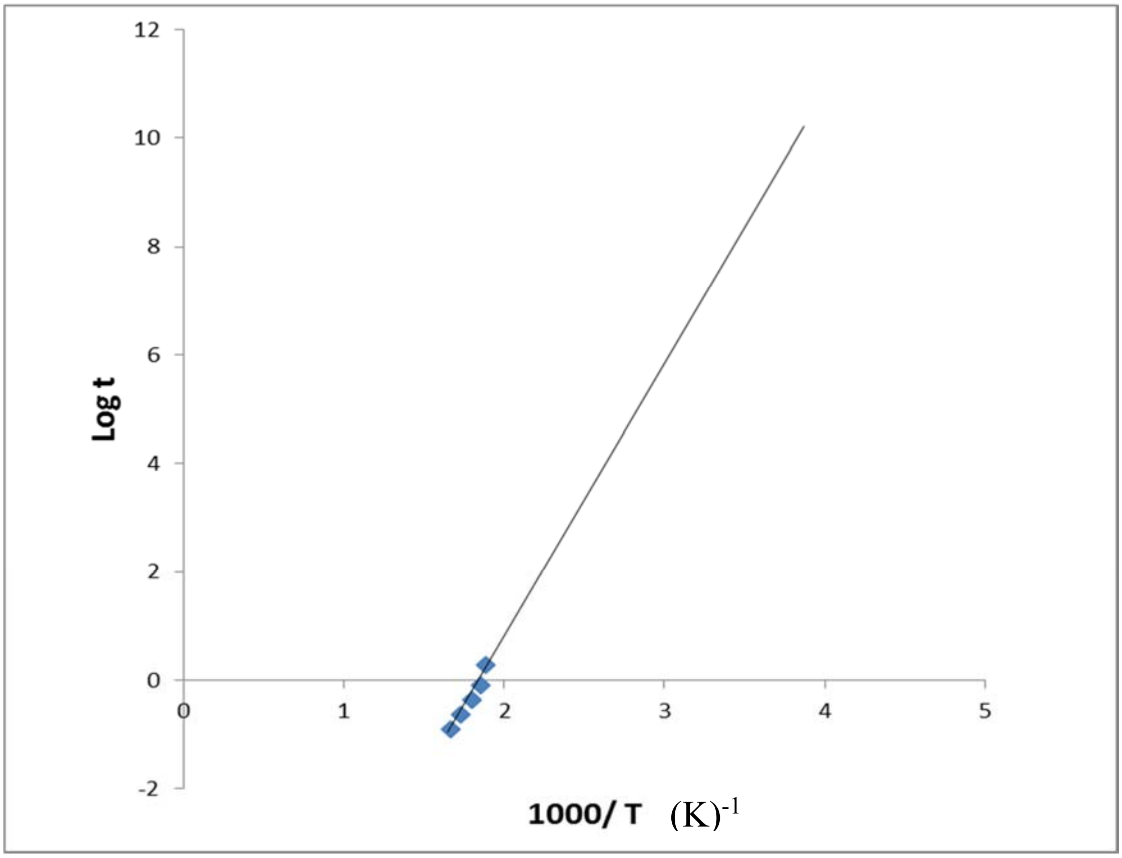


Fig. (4-24) the extrapolation relation of Logt VS $10^3/T$ for K.S.A pipe

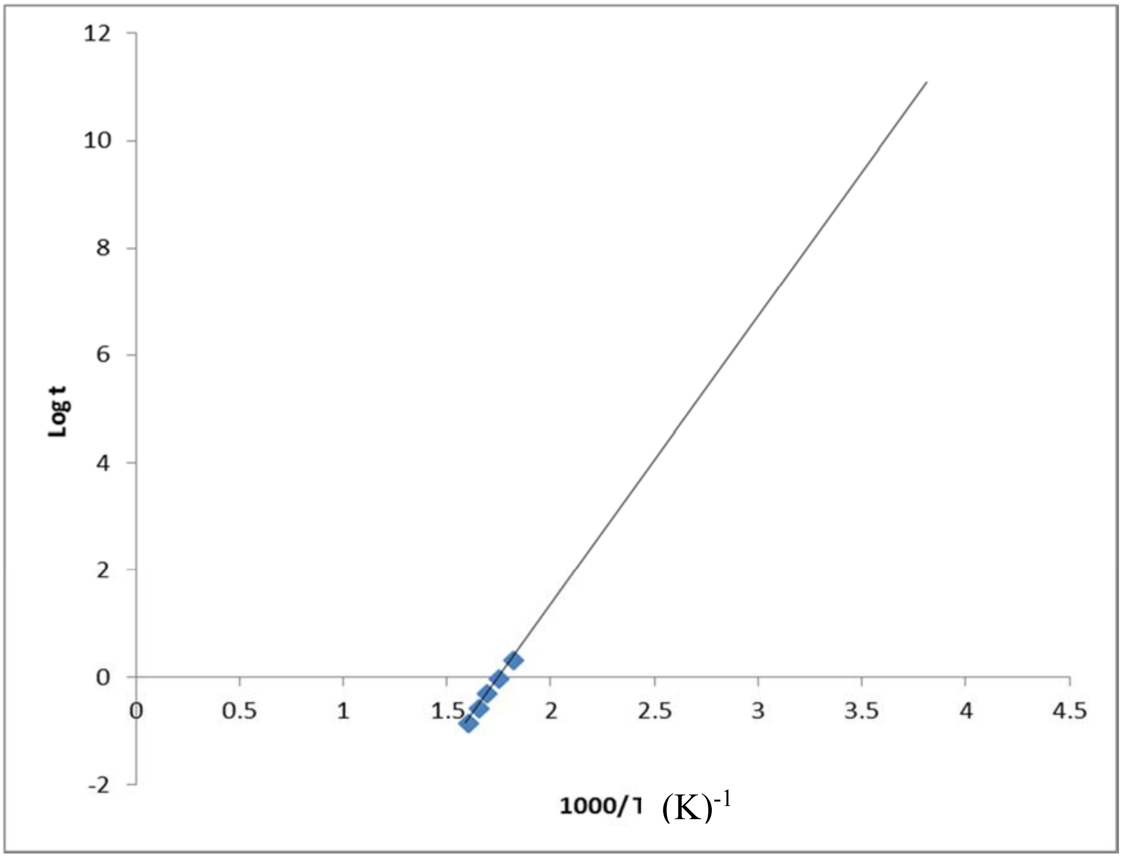


Fig. (4-25) the extrapolation relation of Logt VS $10^3/T$ for Egypt pipe

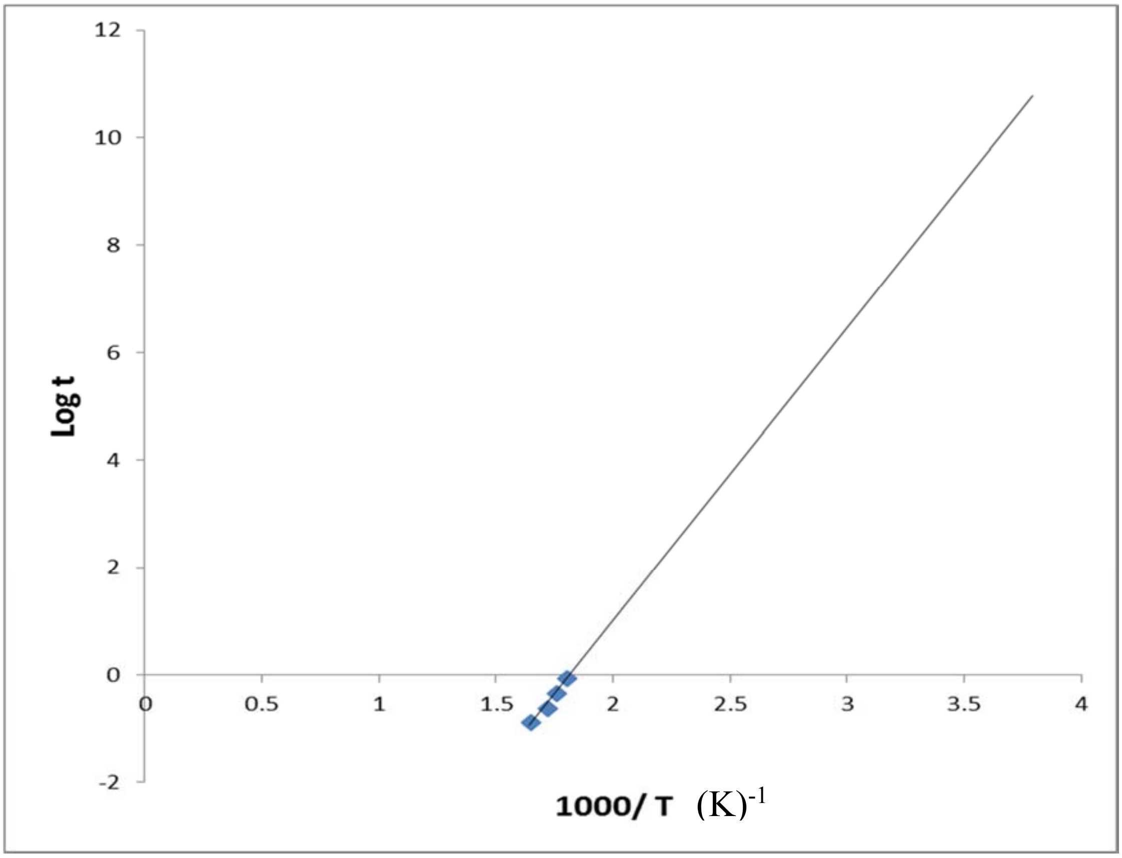


Fig. (4-26) the extrapolation relation of Logt VS 10^3/T for Firat door

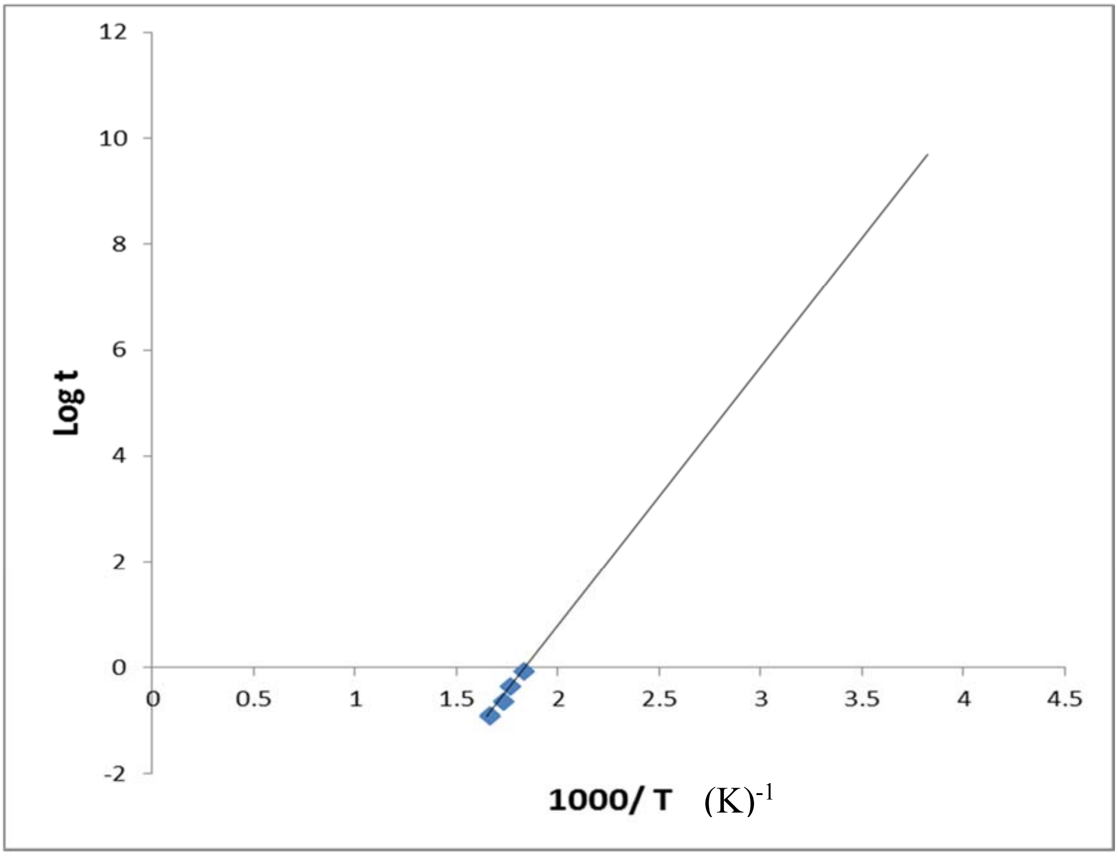


Fig. (4-27) the extrapolation relation of Logt VS 10^3/T for Nova door

Table (4-19) estimation values of lifetime of PVC pipes based on a mass loss of 30% at different temperatures

Pipes	Lifetime (years) at temperature		
	60 ˚C	70 ˚C	80 ˚C
K.S.A	47.73	20.98	8.36
Egypt	400	99.35	57.17

Table (4-20) estimation values of lifetime of PVC doors based on a mass loss of 30% at different temperatures

doors	Lifetime (years) at temperature		
	60 ˚C	70 ˚C	80 ˚C
Firat	137.15	71.97	23.71
Nova	45.41	20.56	4.54

4-2-3 Thermomechanical Analysis (TMA) test:

The TMA test including two procedures (expansion mode & penetration mode):

I - *Expansion Mode:* The transition temperature derived from this procedure considered the glass transition temperature.

The occurrence of an abrupt positive change in the slope of the linear thermal expansion that indicates a transition of the material from one state to another figures (4-28) to (4-31) [86].

The higher glass transition temperature is that of (K.S.A) pipe as shown in table (4-21), while the other types which have glass transition temperature approximately equal.

II - *Penetration Mode*: The transition temperature derived from this procedure is referred to as the softening point, *Ts*. For most materials *Ts* is close to the *Tg* as measured in the expansion mode or as measured by differential scanning calorimetry [86]. See Figures (4-32) to (4-35).

The value of Ts may be affected by the applied force and the probe contact area. Table (4-22) shows the higher value of softening point for K.S.A pipe. The Tg and Ts values are in agreement with [76].

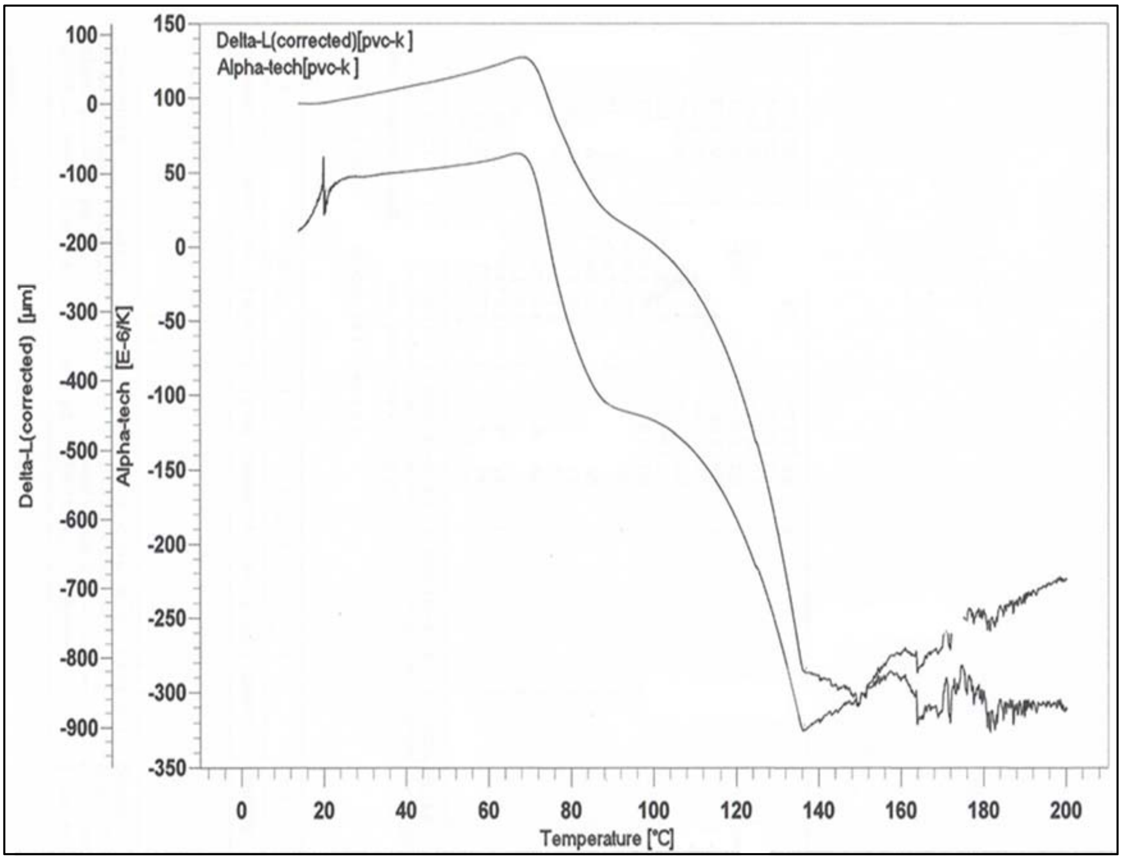


Fig. (4-28) Expansion Mode of K.S.A pipe

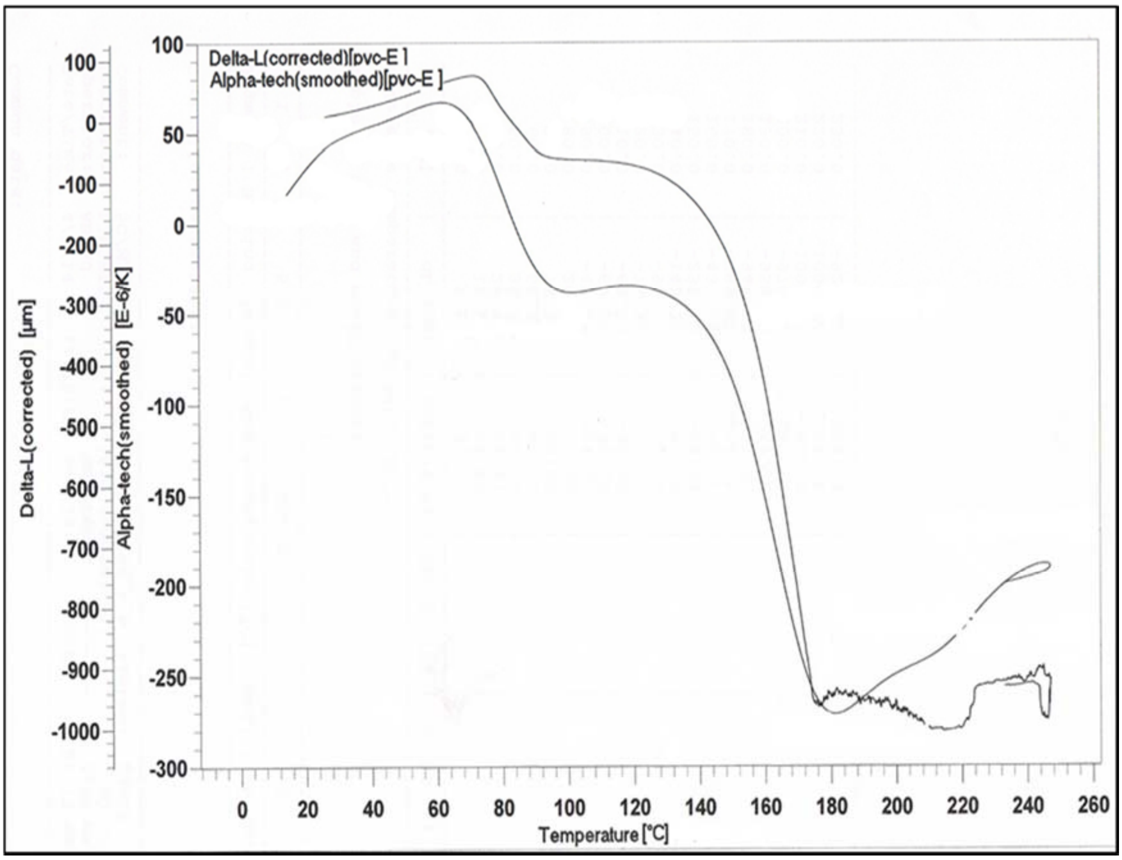


Fig. (4-29) Expansion Mode of Egypt pipe

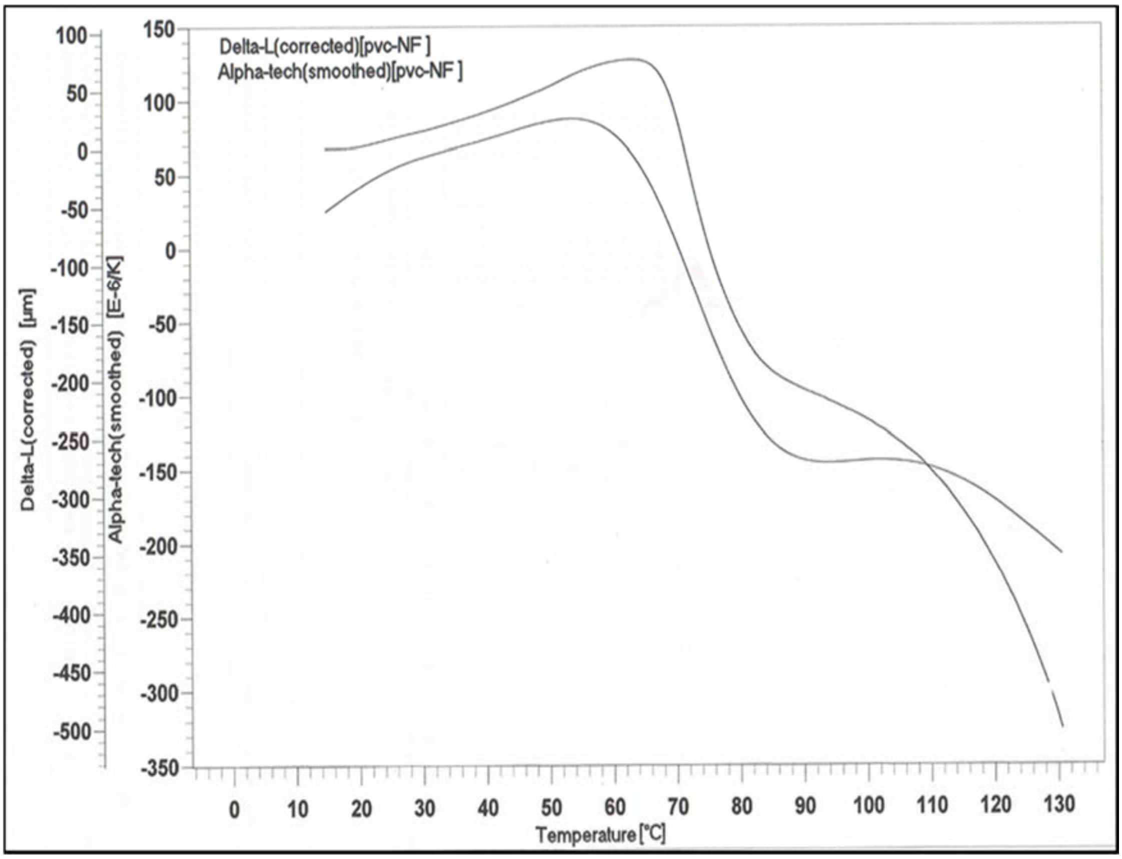


Fig. (4-30) Expansion Mode of Firat door

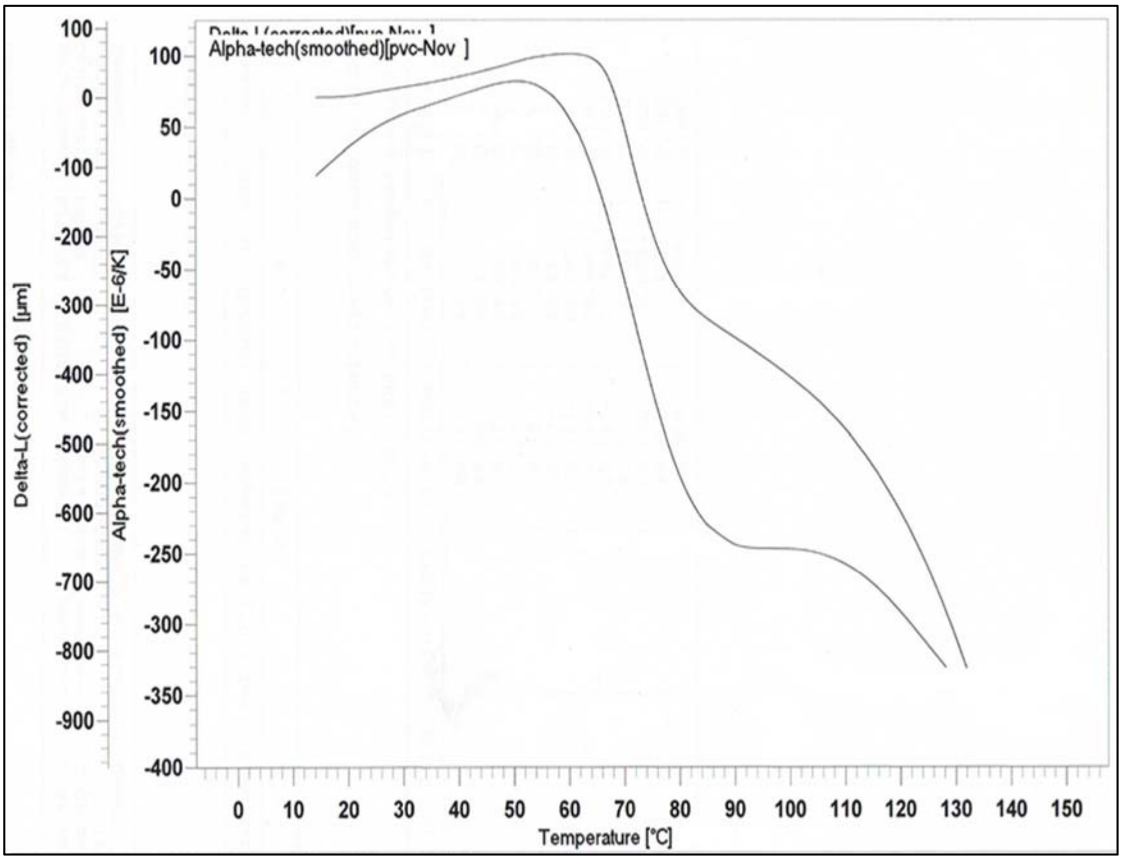


Fig. (4-31) Expansion Mode of Nova door

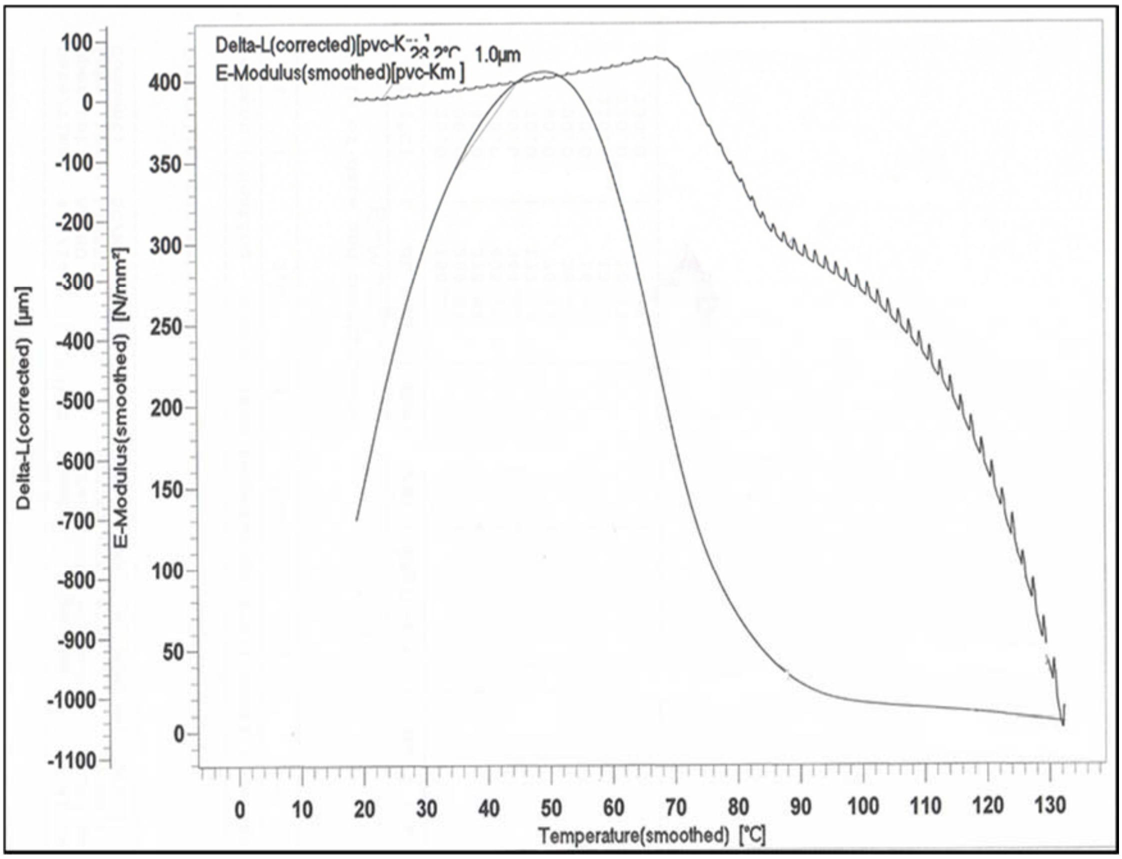


Fig. (4-32) Penetration Mode of K.S.A pipe

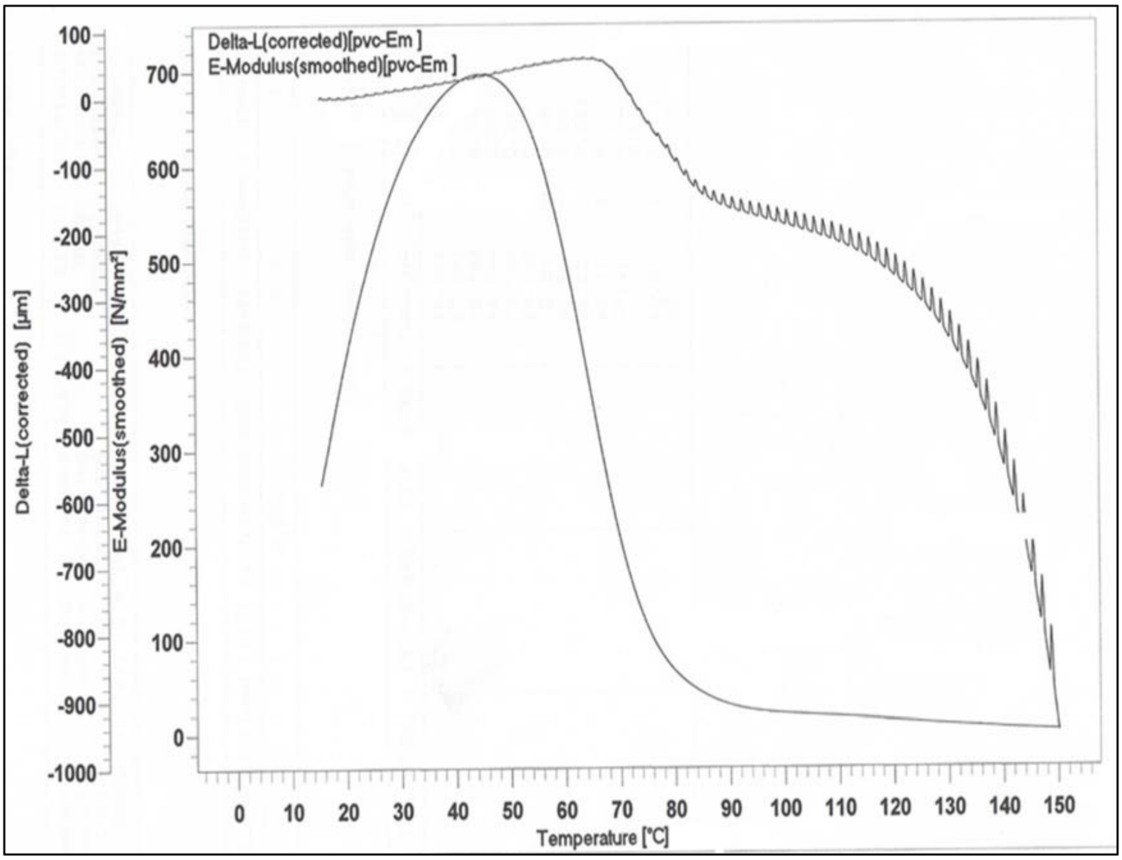


Fig. (4-33) Penetration Mode of Egypt pipe

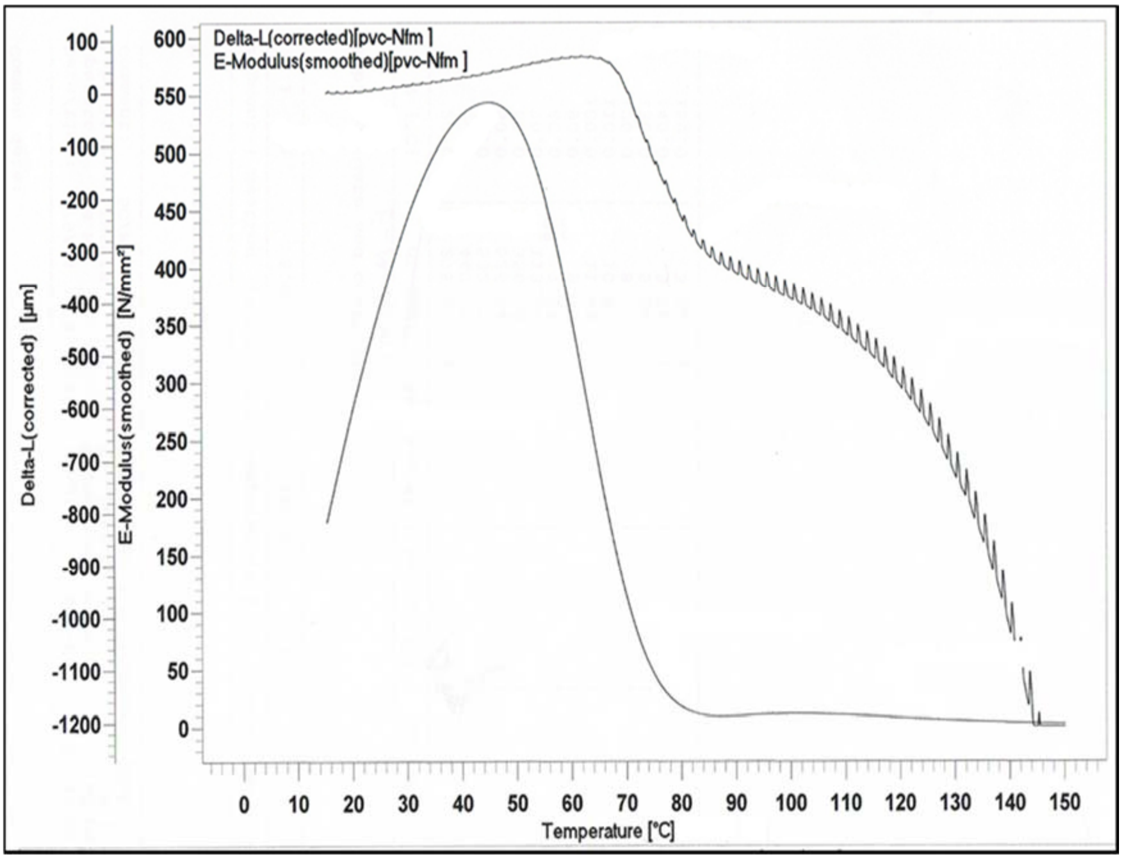


Fig. (4-34) Penetration Mode of Firat door

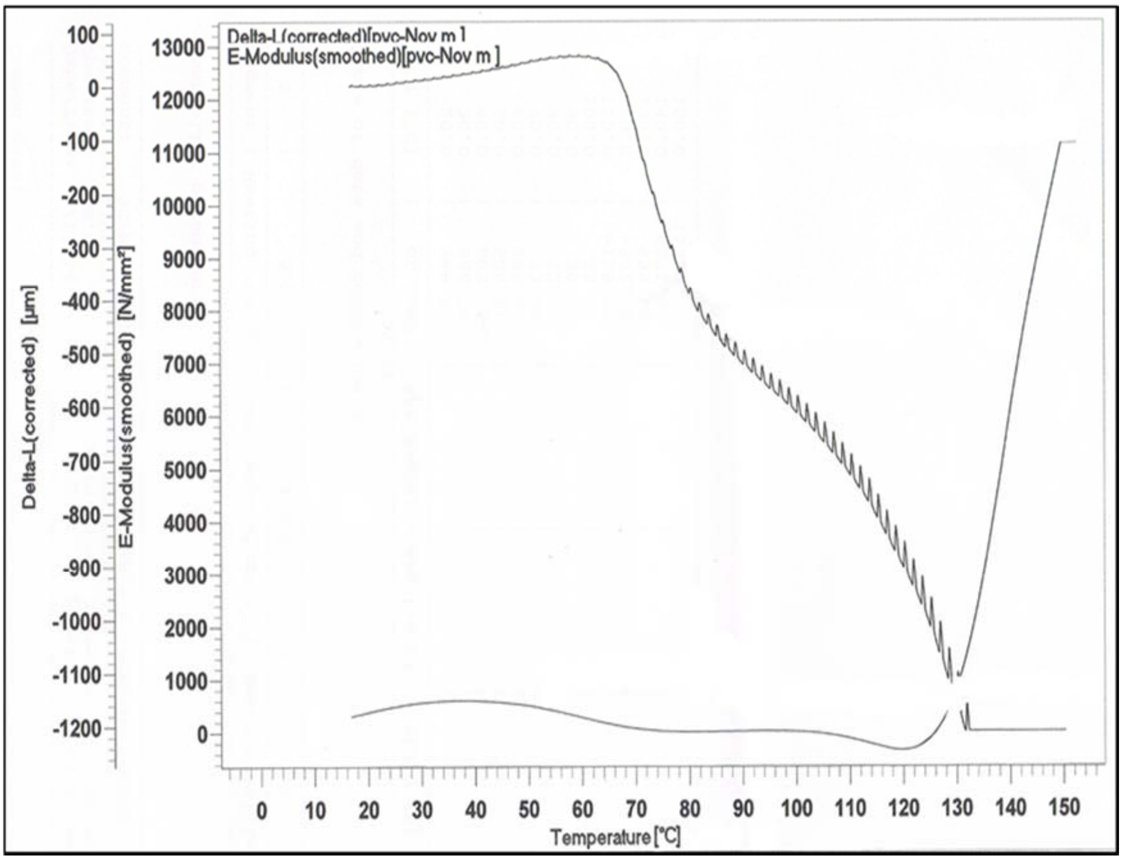


Fig. (4-35) Penetration Mode of Nova door

Table (4-21) T_g values of pipes and doors

NO.	Specimen	T_g (C°)
1	K.S.A.	68.1
2	Egypt	66.27
3	Firat	65.64
4	Nova	65.23

Table (4-22) T_s values of pipes and doors

NO.	Specimen	T_s (C°)
1	K.S.A.	68.8
2	Egypt	67.07
3	Firat	66.62
4	Nova	66.17

Chapter Five

Conclusions and Suggestions for Future Work

5-1 Conclusions:

1- K.S.A pipes exhibited maximum ultimate tensile strength and Young's modulus than the Egypt pipes. However, they have lower value of elongation at Max. and elongation at break than Egypt ones.

2- All the tensile properties of Firat doors are much than that of Nova doors.

3- The flexural strength of Firat doors is greater than that of Nova doors, but have the same values of K.S.A and Egypt pipes.

4- The Firat doors are tough (more impact strength) than Nova doors, while the Egypt pipes have more toughness than K.S.A pipes.

5- The hardness of K.S.A pipes is more than for Egypt pipes, but Firat and Nova doors have the same values of hardness.

6- The thermal conductivity of Firat doors is greater than Nova doors. In general it is in good insulation.

7- Firat doors have more thermal stability than Nova doors at different heating rates.

8- Egypt pipes have more thermal stability when compared with K.S.A pipes at different heating rates.

9- The 30% conversion level is best level for determining the activation energy and lifetime.

10- The activation energy of Egypt pipes and Firat doors is (108.35 KJ/mole) while it is (101.39 KJ/mole) for K.S.A pipes and (98.35 KJ/mole) for Nova doors.

11- The lifetime at 70°C of Egypt pipes is (≈100 years) but it is (≈21 years) for K.S.A pipes, and when comparing the doors at same temperature the lifetime of Firat is (≈72 years) but it is (≈21 years) for Nova.

12- The K.S.A pipes have values for T_g and T_s higher than that of Egypt pipes, while the Firat and Nova doors have the same values.

5-2 Suggestions for future work:

From the present study, the following fields could be suggested for the future work:

1- Study the mechanical wear of pipes and doors.
2- Determine the glass transition using (DSC).
3- Study the weathering outdoor for pipes and doors.

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الخلاصة

تم في هذا البحث دراسة السلوك الميكانيكي لمنتوجات من مادة الـ PVC، المتضمنة نماذج أنابيب من منشأين مختلفين (السعودي والمصري) ونماذج أبواب تركية المنشأ من شركتين مختلفتين (فرات ونوفا) باعتبارها الأكثر رواجاً في الأسواق المحلية العراقية.

ولقد تضمنت الخواص الميكانيكية المدروسة في هذا البحث (أقصى متانة للشد، معامل المرونة، الاستطالة عند أقصى شد، الاستطالة عند القطع، مقاومة الصدمة، الانحناء، الصلادة) لكل من الأنابيب والأبواب.

أظهرت النتائج العملية أن الأنابيب السعودية تمتلك متانة شد ومعامل مرونة أفضل من الأنابيب المصرية، في حين أن الأنابيب المصرية تمتلك استطالة عند أقصى شد واستطالة عند القطع أفضل من الأنابيب السعودية. بينما كانت جميع خواص الشد لأبواب الفرات متغلبة على نوفا.

من خلال مقارنة متانة الانحناء لكل من الأنابيب والأبواب، وجد أن الأنابيب لها متانة إنحناء متساوية تقريباً، بينما أبواب الفرات لها متانة إنحناء أفضل مقارنتاً مع نوفا.

أظهرت النتائج أيضاً أن الأنابيب المصرية ذو متانة صدمة أفضل مما هي عليه في الأنابيب السعودية، وكذلك أبواب الفرات أيضاً لها متانة صدمة أفضل من النوفا.

وكانت أعلى قيمة صلادة (Shore D) كانت للأنابيب السعودية (83.7) بينما كانت النتيجة هي نفسها للنماذج الأخرى.

في هذا البحث أيضاً تم دراسة خاصيتي التوصيلية الحرارية والإستقرارية الحرارية، والتي أظهرت أن أبواب الفرات تمتلك توصيلية حرارية أعلى من أبواب النوفا. وكذلك من خلال القياس الوزني الحراري (TGA) عند معدلات تسخين مختلفة (2, 5, 10, 20, 40 °C/min) تبين أن أبواب الفرات لها إستقرارية حرارية أفضل من النوفا. بينما الأنابيب المصرية أظهرت تفوق واضح في الإستقرارية الحرارية على الأنابيب السعودية.

بوساطة منحنيات القياس الوزني الحراري تم تحديد طاقة التنشيط لمستويات تحلل مختلفة. عند مستوى التحلل 30% عندما تصبح ميكانيكية التحلل مستقرة تكون طاقة التنشيط للأنابيب المصرية وأبواب الفرات لها نفس القيمة (108 KJ/mole) بينما تكون (101.39 KJ/mole) للأنابيب السعودية و (98.35 KJ/mole) لأبواب النوفا.

وكذلك أظهرت نتائج اختبار العمر الافتراضي التي تم إجرائها بوساطة منحنيات (TGA) أن العمر الافتراضي يقل بزيادة درجة الحرارة، حيث أظهرت النتائج عند درجة حرارة (70°C) أن العمر الافتراضي للأنابيب المصرية بحدود (100 سنة) بينما السعودية (21 سنة). وعند درجة الحرارة ذاتها كان العمر الافتراضي للأبواب الفرات بحدود (72 سنة) بينما النوفا كان العمر الافتراضي لها بحدود (21 سنة).

أظهر الفحص الميكانيكي الحراري (TMA) أن الأنابيب السعودية تمتلك قيمة تحول زجاجي (T_g) ودرجة لدونة (T_s) أعلى من الأنابيب المصرية بينما الأبواب كانت قيمها متقاربة.

جمهورية العراق
وزارة التعليم العالي والبحث العلمي
جامعة بغداد
كلية التربية للعلوم الصرفة ابن الهيثم
قسم الفيزياء

دراسة الصفات الحرارية والميكانيكية وعمر الأداء لبوليمر PVC

رسالة قدمت الى كلية التربية للعلوم الصرفة ابن الهيثم – جامعة بغداد
كجزء من متطلبات نيل شهادة الماجستير في علوم الفيزياء
تقدم بها الطالب

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بإشراف

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