



ISSN: 0975-833X

Available online at <http://www.journalcra.com>

INTERNATIONAL JOURNAL  
OF CURRENT RESEARCH

International Journal of Current Research  
Vol. 11, Issue, 11, pp.8110-8115, November, 2019

DOI: <https://doi.org/10.24941/ijcr.37200.11.2019>

## RESEARCH ARTICLE

### EFFECT OF POLYETHYLENE CONTENT ON PHYSICAL PERFORMANCE OF CARBON BLACK FILLED ELASTOMER/POLYETHYLENE BLENDS

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#### ARTICLE INFO

##### Article History:

Received 04<sup>th</sup> August, 2019  
Received in revised form  
18<sup>th</sup> September, 2019  
Accepted 15<sup>th</sup> October, 2019  
Published online 26<sup>th</sup> November, 2019

##### Key Words:

EPDM, LDPE,  
Blending, Swelling,  
Mechanical Properties.

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Citation: El-Feky, A.A., Osman M.M. and Hamdy, A. 2019. "Effect of polyethylene content on physical performance of carbon black filled Elastomer/polyethylene blends", *International Journal of Current Research*, 11, (11), 8110-8115.

#### ABSTRACT

Modification of polymers is widely used nowadays. Among the most important and practical methods of modifying polymers is blending. Ethylene propylene diene monomer (EPDM) was blended with low density polyethylene (LDPE) in five different ratios for each (100/0, 90/10, 80/20, 70/30, and 60/40) respectively with loading the blends with carbon black filler. Effect of polyethylene content in the blend on its swelling behavior and Mechanical properties was investigated. The critical content of PE in the blend was 0.3 weight fraction. All the blends showed excellent swelling behavior in sea water medium. It was found that addition of PE into EPDM matrix enhances the mechanical properties of the blends such as: tensile strength and modulus of elasticity. Also EPDM/PE blends show sufficient degree of toughness to be utilized in industrial applications efficiently.

#### INTRODUCTION

Polymers are materials of very high molecular weight that are found to have multifarious applications in our modern society (Sahnoune, 2003). World consumption of polymers has significantly increased in the past twenty years because of their numerous advantages including excellent corrosion resistance, competitive cost, low weight, easiness of installation, and higher specific strength than many other materials (Chang, 2006). Exploitation of many of these unique properties of polymers had made them extremely useful to mankind and hence, they represent an area where chemists continue to make major important contribution (Maryam, 2019). Polymers are used extensively in food packaging, clothing, home furnishing, transportation, medical devices, information technology and they have along commercial history in coatings and adhesives industries. Polymeric construction consists of several structural units bound together by covalent bonds. They can be subdivided into three main categories (Power, 2002): (i) Thermoplastics which consist of individual long chain molecules where any product can be reprocessed by chopping it up and feeding it back into the appropriate machine, (ii)

Thermo sets which include an infinite three dimensional networks that can be created only when the product is in its final form and cannot be broken down by reheating, (iii) The last category is rubber which contain looser three dimensional networks, where the chains are free to change their shapes. Ethylene propylene diene monomer (EPDM) can be considered as one of the most interesting elastomers that appear in recent years. EPDM is an artificial rubber prepared by random copolymerization of ethylene with propylene in presence of diene monomer (Bicerano, 1996). The most important characteristic of EPDM is its high oxygen, ozone, and UV resistance and hence, it is favorable for outdoor applications. Also EPDM has excellent adhesion to metals, excellent electrical, resistance characteristics as well as favorably good tensile properties, hence the areas of application where the use of EPDM is now accepted are numerous (Antunes, 2012). Polyethylene has many excellent characteristics which have led to its wide spread use such as low cost, easy process ability, excellent electrical insulation properties, toughness and flexibility (even at low temperatures), freedom from odor and toxicity, and reasonable clarity of thin films (Nikesh, 2018) Major markets of LDPE are in packaging and sheeting, also, cross linked LDPE foam have been used in the automotive industry for carpeting, boot mats, sound deadening, and pipe insulation, and as flotation media for oil carrying and dredging hose (Suleiman, 2008). With the advancement of technology, the utilization of polymeric materials becomes complex and require the introduction of new additives.

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Hence Modification of polymers is widely used nowadays. Among the most important and practical methods of modifying polymers is blending (Reyhaneh, 2016; Ming, 2019; Wu, 2005). Polymer blending is effective, inexpensive, and easier route to develop polymer formulations that can target a diverse set of applications (Ulug, 2012; Sionkowska, 2011). Blending of polymers together has proved to be one of the methods that have been applied successfully for obtaining new polymeric materials with special or specific properties, i.e., up grading its quality. Product performance requirements will dictate the initial selection of formula ingredients (Kucharczyk, 2012; Fu, 2008). These materials must be environmentally safe, meet occupational health and safety requirements, process able in the product manufacturing facilities, and must be cost effective. Fillers or reinforcement aids are added to rubber formulations to meet materials property targets such as tensile strength and abrasion resistance. There are many well known types of fillers like silica, silicates, clays, ceramic, non black fillers, and carbon black which is the most reinforcing agent used for rubber. Carbons black (CB) are amorphous carbons characterized with high surface area and are utilized frequently in manufacture of tire, wire, cable, and aviation industries. CB is usually added to PE to modify its dielectric properties and to improve its weathering resistance and; whereas it is frequently used as reinforcing filler in EPDM rubber based formulations. (Dubey, 2014). Carbon black is made by incomplete combustion of hydrocarbon such as natural gas or heavy aromatic residue oils from petroleum or coal. Reinforcement by carbon black involves an increase in the resistance of rubber to abrasion, tearing, and other types of tensile failure. Also, it leads to an increase in the hardness, modulus of elasticity, and related viscoelastic properties (Ping, 2010; Chen, 2017). Electrically insulating rubbers and plastics can be rendered conductive by the incorporation of adequate loading of carbon black. The most important properties of carbon black that affect the applications are particle size, structure, physical and chemical nature, and porosity. The smaller the particle size the poorer the processibility and the higher the reinforcement. Also electrical conductivity and rate of cure are affected to a considerable degree by the surface chemistry of carbon black (Huang, 2002). This study explores the effect of polyethylene ratio on the swelling behavior and mechanical properties of CB-filled PE/EPDM blends.

## Experimental

### MATERIALS

**EPDM:** Ethylene propylene diene monomer (EPDM), is the raw rubber used in this work which contains a small proportion of diene to confer unsaturation and make sulfur vulcanization possible. The commercial grade of (EPDM) used in this work is Vistalon 5600 with Diene content equals 4-7 % and 60 %Ethylene content.

**Thermoplastics:** Low-density polyethylene (LDPE) is the thermoplastic used in this work, where some of its important parameters are listed in Table (1)( Meister, 2000). LDPE film has a good balance of mechanical properties, and it is a good barrier to water and water vapor, also, its resistance to acids, alkalis, and inorganic salt solutions is high (Whelan, 1981; Jianjunlu, 2002).

**Carbon black:** Most rubber articles produced are compounded with carbon black (CB) as a filler typically in the amount of at

least 50 parts of carbon black per hundred parts of rubber by weight (50 phr). There are far too many types of carbon black available to the rubber compounder (23), fast extrusion furnace (FEF), is the type used in this work. FEF black is characterized by medium particle size and by giving moderate abrasion resistance and improved processing.

**Curing agents:** The curing system used is sulfur system, which is composed of: i) Activator (stearic acid + zinc oxide), ii) Accelerators (2 – dibenzothiazole disulphide (MBTS), tetra methylthiuram disulphide (TMTD) and phenyl –  $\beta$  naphthyl amine (PBN)) and iii) Elemental sulfur. Also, dioctylphthalate (DOP) and processing oil are used as softeners.

**Blend Preparation and Vulcanization:** Blends of PE and EPDM in different proportions were prepared in a Brabender Electronic Plastic order at a temperature of 170 °C which was adjusted to be above the specific melting temperature of PE, and rotor speed of 30 rev/min. Thereafter, the curing agents were mixed with the PE / EPDM blend at a temperature not exceeding 70 °C on a two - roll mill, the blend was passed through the rolls twice without banding at a roll opening of about 0.2 mm and then it was banded with a mill opening of about 1.5 mm, 3-4 cuts were made every ½ minute alternatively from each side. The composition of working blends, is given in Table (2). The weight of the blends was checked after mixing to ensure that the loss in weight does not exceed 5%. The compounded blend was left at room temperature over night before vulcanization. Then vulcanization was carried out over 170 °C  $\pm$  2 °C at 4 MPa for 25 minutes. The stock was sheeted out and compression molded in an electrically heated hydraulic press . The optimum cure time was derived from the oscillating disc rheometer (Mönsanto ODR - 100) measurements (24).

## METHODS

**Swelling test:** Swelling tests were conducted on the polymer blends. The blends were prepared as previously stated, after curing they were cut into thin 5 mm radius discs with thickness 2 mm. The samples were weighed, using an electronic balance, then they were immersed in natural sea water. The samples were reweighed weekly over 40 weeks. Before weighing, the samples were removed from the solution with tweezers, rinsed with distilled water and blotted dry to remove any excess water. The water uptake by the sample can be defined as the amount of water absorbed at a certain moment of time per unit weight of dry polymer. The swelling degree (Q %) was calculated as follows:

$$(Q)\% = \frac{W_t - W_i}{W_i} \times 100$$

where,  $W_t$  is the weight of the sample at a certain moment of time and  $W_i$  is the initial weight of the same sample.

**Mechanical measurements:** Stress - strain behavior in uniaxial tension was measured using a Zwick universal testing machine (model 1445) (Zwick, Germany). Dumbbell shaped specimens were cut from blends sheets according to ASTM 415 - 80 using a steel die of standard width 4 mm, length 50 mm, and 2 mm thick.. The thickness of the test specimens was determined by a gauge graduated to one hundredth of mm.

The tensile strain ( $\epsilon$ ) and tensile stress ( $\sigma$ ) were calculated as follows:

$$\epsilon = \frac{L - L_0}{L_0}$$

$$\sigma = \frac{F}{A}$$

Where,  $L_0$ ,  $L$  are the lengths of undeformed and deformed samples,  $F$  is the force, and  $A$  is the cross sectional area of strained samples obtained from the relation:

$$A = \frac{L_0 - A_0}{L}$$

Where,  $A_0$  is the cross sectional area of unstrained sample. From stress – strain diagrams, many useful parameters were deduced such as tensile strength, modulus of elasticity, elongation at break, permanent set, and toughness.

## RESULTS AND DISCUSSION

**Swelling Behavior:** As the solvent is absorbed in the polymer, swelling begins, and its duration depends on the chemical and physical nature of the solvent-polymer pair. The mass and dimensions of polymer system may be changed due to the penetration of the solvent into swollen specimen, so swelling process may lead to deformation or destruction of the sample microstructure (Gent, 1991). The time dependence of the degree of swelling for various EPDM/PE blends, with different blend ratios, is shown in Fig. (1). The general behavior may be approximated by an exponential growth function for all samples showing positive swelling mechanism of the form:

$$Q = Q_m [1 - \exp(-t / \tau)]$$

where,  $Q$  is the degree of swelling at time  $t$ ,  $Q_m$  is the degree of equilibrium swelling and  $\tau$  is a characteristic time which depends on both the polymer matrix and the cross linking density as is observed in Table (3). The data shown in table (3) indicate that addition of polyethylene (PE) to EPDM is accompanied by a decrease in the values of ( $\tau$ ). Fig. (1) Shows the dependence of the degree of water uptake by the samples (represented in percent form i.e.  $Q$  %) on the blend ratio. It can be noticed that increasing PE content in the blend leads to a slight increase in water uptake. This can be attributed to the fact that the swelling of polymer networks in suitable liquids is found to be large for three dimensional network polymers (Osanaie, 1986).

Given that polyethylene chains are not flat, but they have a three dimensional shape, where the hydrogen atoms being arranged along an inner zigzag chain of carbon atoms (Hatada, 1997), this may explain the increase in the extent of water uptake by the sample upon incorporation of polyethylene into rubber matrix. However, it can be noticed that this negative effect is very weak, and we can simply regard all EPDM / PE blends, even with high PE content, resistant to water uptake but with slightly different degrees. It can be noticed from Fig (1) that the initial rate of water absorption is followed by nearly a horizontal behavior, which indicates early saturation of the samples. This saturation phenomenon can be explained by the fact that the extent of swelling represents a competition between two forces, the first one is the free energy of mixing

**Table 1. Some parameters of low density polyethylene (LDPE)**

Parameter	LDPE
Specific gravity ( $\text{g} / \text{cm}^3$ )	0.91 – 0.94
Glass transition temperature	about - 80 °C
Molding temperature	about 250 °C
Annealing temperature	100 °C
Crystalline melting point, $T_m$	100 – 125 °C
Swelling %	(15 – 40)
Dielectric constant	2.2 at $10^3$ HZ
Dissipation factor	0.0003 at $10^3$ HZ
Volume resistivity	$10^{16} \Omega \cdot \text{cm}$

which, causes the solvent to penetrate and tries to dilute the polymer saturation. The second opposite force is generated when the polymer chains in the cross-linked polymer network begin to elongate, under the swelling action of the solvent, this force is an elastic retroactive force in opposition to deformation. We can say that the swelling reaches steady state when these two forces are balancing (Florian, 1998). Also this saturation behavior of EPDM / PE blends can be explained regarding the solvent effect. It is well confirmed that, when polymers are exposed to a salt solution the curve leveled off, after an initial high rate of water absorption, and eventually equilibrium is attained. This can be attributed to the fact that the presence of salts in water inhibited the uptake of water by varnishes, and the stronger the salt, the less is the amount of water absorbed (Walter, 1998). Generally, the horizontal part of the curves means that the rubber blend degradation or extraction of its soluble ingredients is very small. The dependence of the degree of maximum swelling for EPDM/PE blends on temperature was investigated, and the results of blend 60/40, as represent able sample, are shown in Fig. (2). It can be noticed that the rising of temperature from 30 to 50 °C has a slight effect on the swelling degree of the samples, while samples exposed to 70 °C show a noticeable increase in swelling degrees with progress of time, which can be explained by the fact that the rising of temperature leads to an increase in the internal stress caused by the diffused liquid (30). When this internal stress becomes high compared with the forces between macromolecules, it enhances the swelling and leads to higher degree of swelling at higher temperatures. Also the thermal expansion caused by rising temperature creates, an additional free volume, which is considered as another reasonable explanation for the observed increase of swelling.

**Mechanical properties of EPDM / PE blends:** Mechanical properties are important parameters that characterize polymers. They provide basis for quality control and comparative evaluation of various polymers (Seymour, 1990), hence investigation of stress – strain behavior of polymer blends is essential. An important parameter that has been found to be effective in modification of the mechanical properties of the blend is its composition (Nielsen, 1974) (i.e. the blend ratio), therefore it has been found desirable to present the changes occurring in mechanical properties as a function of blend ratio. Stress – strain measurements of the samples have been carried out at working temperature (30°C) and the obtained results are shown in Fig. (3). Generally, it can be seen that blending EPDM with PE improves mechanical properties of this elastomer as will be discussed later in details.

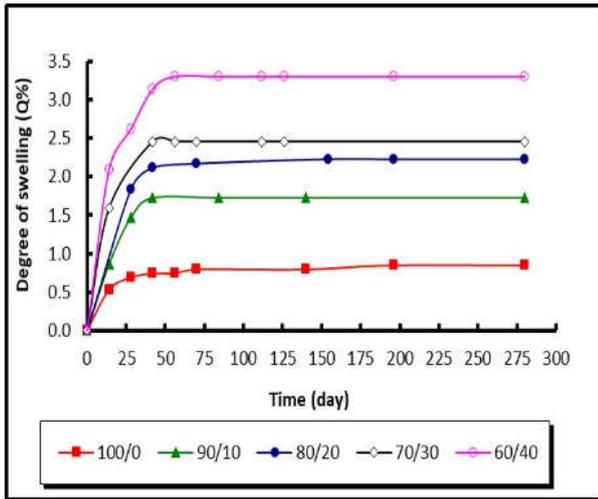
**Tensile strength:** The results illustrated in Fig. (4) Show the variation of the tensile strength at break (TS) as a function of weight fraction of PE, where two regions can be noticed. Region (I) represents the TS of the matrix of low PE content (0, 0.1 and 0.2 weight fraction) in which the addition of PE to

**Table 2. Composition of the working polymer blends**

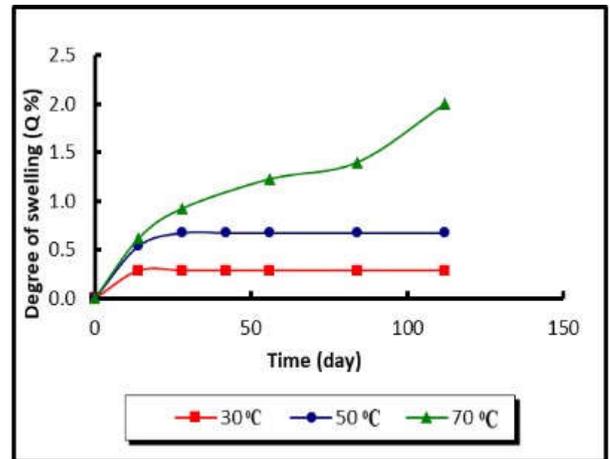
Ingredients	Blend (1)	Blend (2)	Blend (3)	Blend (4)	Blend (5)
EPDM	100	90	80	70	60
LDPE	0	10	20	30	40
Stearic acid	2	1.8	1.6	1.4	1.2
ZnO	5	4.5	4	3.5	3
Processing oil	30	27	24	21	18
DOP	0	1	2	3	4
FEF	70	70	70	70	70
MBTS	1.5	1.35	1.2	1.05	0.9
TMTD	1.5	1.35	1.2	1.05	0.9
PBN	1	0.9	0.8	0.7	0.6
S	3	2.7	2.4	2.1	1.8

**Table 3. The values of  $Q_m$  and  $\tau$  for EPDM / PE blends**

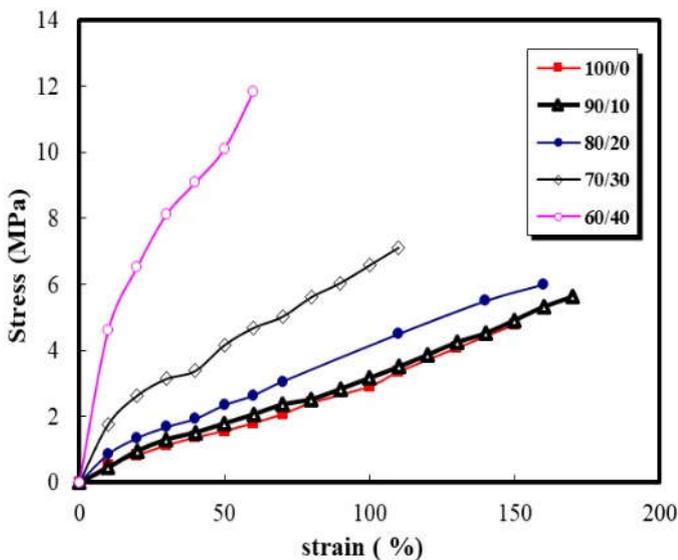
Blend ratio	$Q_m$	$\tau$ (days)
100 / 0	0.008465	56.3211
90 / 10	0.017250	18.4798
80 / 20	0.022284	14.6067
70 / 30	0.024625	14.4687
60 / 40	0.033018	13.4427



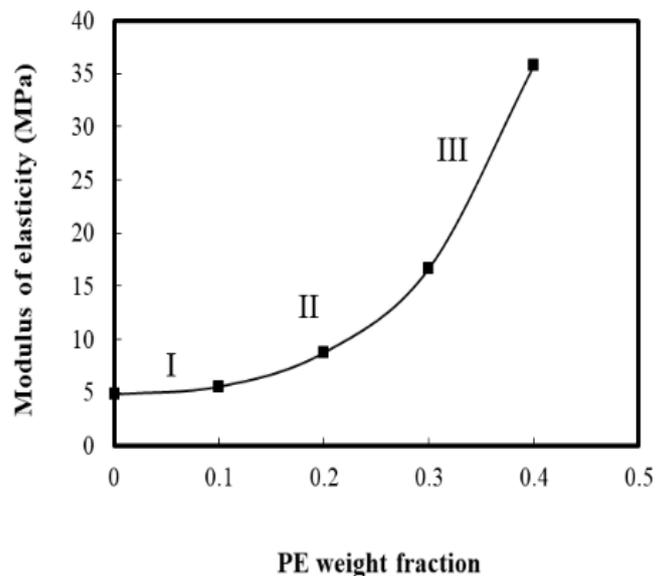
**Fig. 1. Dependence of the degree of swelling (Q %) on time for prepared PDM/PE blends**



**Fig. 2. Variations of swelling degree of EPDM/PE blend (60/40) in sea water at different temperatures**



**Fig. 3. Stress-strain curves for EPDM/PE blends at 30 °C**



**Fig.4. Tensile strength of EPDM/PE blends as a function of PE weight fraction**

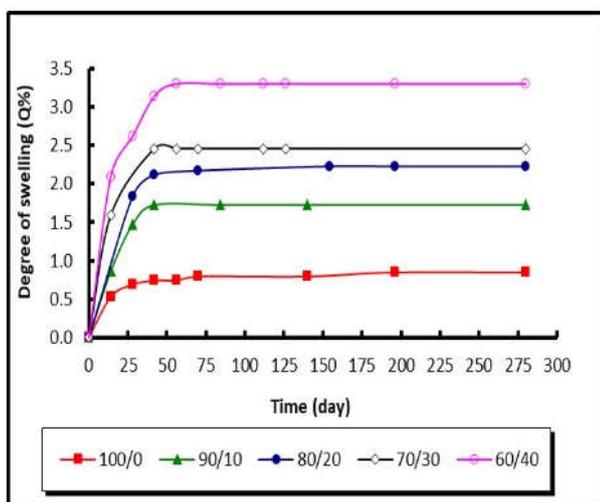


Fig. 5. Variations of elongation at break of EPDM/PE blends as a function of PE weight fraction

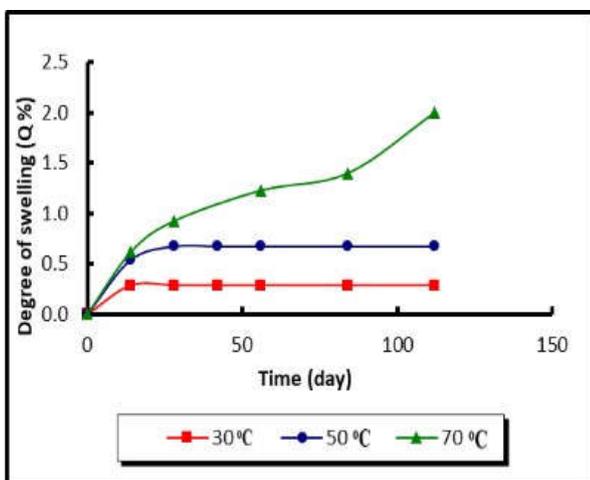


Fig. 6. Modulus of elasticity for EPDM/PE blends as a function of PE weight fraction

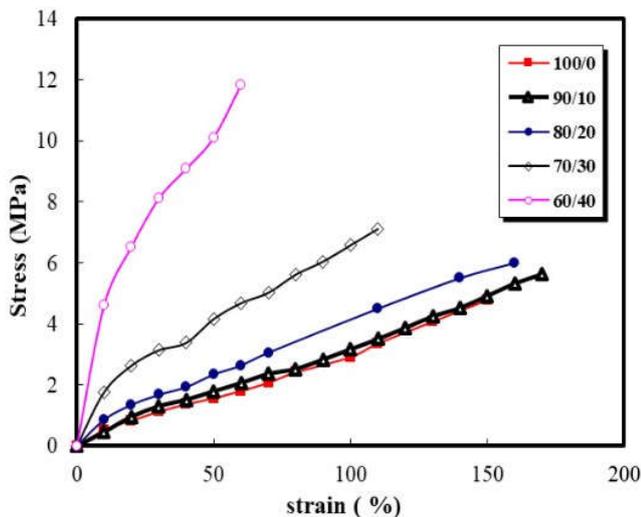


Fig. 7. Variations of toughness of EPDM/PE blends as a function of PE weight fraction

EPDM has no effect on the tensile strength, while region (II) shows that incorporation of PE with greater weight fraction (0.3 and 0.4) leads to an increase in TS values of the blend, this increase can be attributed to the predominance of the crystalline phase in the matrix.

Also the occurrence of intercross linking, either between the macromolecules of each component or between the macromolecules of the amorphous phase of the two components, leads to an increase in the degree of cross linking, which enables the blend to bear higher stress level and, hence, shows high tensile strength values. The linearity obtained at region (II), may be taken as an indication for the reasonable compatibility of the two polymers at this stage (Krausse, 1972; Manson, 1976).

**Elongation at break:** The variation of the values of elongation at break ( $E_b$ ) as a function of weight fraction of PE is presented in Fig. (5). It can be noticed that at low concentration of PE the values of  $E_b$  undergo a slight decrease, while for high weight fraction of PE (higher than 0.2), a remarked decrease of  $E_b$  with noticeable rate is observed. These results can be explained by the fact that elongation is a type of deformation, and as it is well known that, the crystalline polymers resist deformation, it would be expected that the elongation behavior of the blend will be a function of the crystallinity percentage of the plastic component. Thus increasing the weight fraction of PE will increase the crystallinity contribution in the blend hence; it will be accompanied with a decrease in the elongation property of the blend. However it is very interesting to note that polyethylene have a similar tendency with elastomeric materials, even though polyethylene would show plasticity rather than elasticity. Therefore, the approximation may be made that as polyethylene shows essentially energetic elasticity, it can be treated with the same method as those of rubbery materials showing entropic elasticity (Nitta, 2002). However, we can say that although incorporation of PE into EPDM matrix leads to a decrease in elongation at break ( $E_b$ ), this decrease is not marked one.

**Modulus of elasticity:** Modulus is the ability of a sample of a material to resist deformation (Oadian and George, 1991). The modulus of elasticity for each EPDM / PE blend was obtained from the initial slope of the stress strain curves, then plotted as a function of polyethylene content in Fig. (6), where it becomes clear that three distinct regions are observed. Region (I) doesn't show any change in the modulus of the blend on adding low weight fraction of PE to EPDM matrix while, a gradual increase in modulus can be noticed in region (II) with increasing PE content up to 0.3 weight fraction. Whereas the third region is characterized by a marked increase of modulus upon the addition of higher weight fraction of PE (above 0.3). Taking in our account that the modulus indicates the stiffness of the material, we can explain these results by the fact that EPDM / PE blend consists of two phases: amorphous and crystalline and the composite behavior of the blend is controlled by the mechanical interaction of the two phases (Abdel, 1999). So, we can consider blends with low concentration of PE as rubber matrix reinforced with partially crystalline LDPE, and as the soft amorphous phase is the dominant phase, this leads to the low modulus values represented in region (I). On the other hand, at high values of PE weight fraction, the blend may be visualized as a matrix of plastic that has been toughened by the amorphous elastomer, i.e., the hard plastic phase is the dominant phase, this explains the high modulus of the blends with high PE concentration observed in regions II and III of Fig. (6).

**Toughness:** Toughness is a measure of the sample ability to absorb mechanical energy without breaking (Fengkui, 2001).

Toughness values of EPDM / PE blends (as obtained from the area underneath a stress – strain curve) are plotted against PE weight fraction, where the results obtained are shown in Fig. (7). A monotonic decrease in toughness can be observed with increasing PE content in the blend.

It is well confirmed that for many common applications polyethylene is a tough polymer due to its elastic behavior (Knight, 1996), this elastic behavior in semi-crystalline polymers, such as polyethylene, can be explained with a cluster network model in which these clusters are connected by means of tie molecules and operate as junction points (Kilian, 1984). The elasticity is reflected as a reasonable degree of toughness, hence addition of PE to a tough elastomer (EPDM) show a slight decrease in toughness as indicated in Fig. (7). So, we can say that EPDM / PE blends have a reasonable degree of toughness to provide good performance in industrial applications.

## Conclusion

- All the prepared EPDM/PE blends showed excellent swelling behavior and very low values of penetration rate and average diffusion coefficient in sea water medium.
- Increasing PE content in the blend leads to a slight increase in water uptake.
- Rising temperature from 30 to 50° C has a slight effect on the swelling degree of the prepared samples, noticeable increase in swelling degrees is observed when the samples exposed to 70°C.
- Addition of PE to EPDM improves the mechanical properties of its vulcanizates, as it leads to an increase in tensile strength and modulus of elasticity. So, EPDM / PE blends have a very good resistance against mechanical damage and good tolerance to possible mechanical stress.

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