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Characteristic of low-density polyethylene reinforcement with nano/micro particles of carbon black: a comparative study

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ABSTRACT

Purpose: Low density polyethylene is commonly used polymer in the industry because of its unique structure and excellent overall performance. LDPE, is relatively low mechanical properties and thermal stability can sometimes limit its application in industry. Therefore, the development of particulate reinforced polymer composites is one of the highly promising methodologies in the area of next generation engineering products.

Design/methodology/approach: Nano and Micro composite from low density polyethylene LDPE reinforced with different weight fraction of carbon black particles (CB) (2, 4 and 8)% prepared by first dispersion Nano and Micro carbon black particles CB in solvent and then mixing manually with low density polyethylene LDPE pellet and blended by twin-screw extruder, the current research study the mechanical properties (tensile strength, elastic modulus, and hardness), FTIR, DSC, and thermal conductivity of prepared nano and micro composites using two methods and the morphological properties of nano-micro composites.

Findings: The tensile strength of the LDPE/CB nano and micro composites improved at 2% and 4%, respectively, and decreasing at 8%, addition of carbon black nanoparticles led to increase the tensile strength of pure low-density polyethylene from 13.536 MPa to 19.71 MPa, and then dropping to 11.03 MPa at 8% percent, while the elastic modulus of LDPE/CB nano and micro composites shows an improvement with all percentages of CB. The results show that the mechanical properties were improved by the addition carbon black nanoparticles more than addition micro- carbon black. FTIR show that physical interaction between LDPE and carbon black. The thermal conductivity improvement from 0.33 w/m.k for pure LDPE to 0.62234 w/m.k at 2% CB microparticle content and the reduced to 0.18645 w/m.k and 0.34063 w/m.k at (4 and 8)% micro-CB respectively, The thermal conductivity of LDPE-CB nano-composites is low in general than that the LDPE-CB microcomposite. DSC result show improvement in crystallization temperature T_c , melting temperature and degree of crystallization with addition nano and micro carbon black. Moreover, SEM images revealed to uniform distribution and good bonding between LDPE and CB at low percentages and the presence of some agglomeration at high CB content.

Research limitations/implications: This research studied the characteristics of both nano and micro composite materials prepared by two steps: mixing CB particles with solvent and then prepared by twin extruder which can be used packaging material, but the main limitation was the uniform distribution of nano and micro CB particles within the LDPE matrix. In a further study, prepare a blend from LDPE with other materials and improve the degradation of the blend that used in packaging application.

Originality/value: LDPE with nanocomposites are of great interest because of their thermal stability, increased mechanical strength, stiffness, and low gas permeability, among other properties that have made them ideal for applications in the packaging and automotive industries. LDPE reinforcements nano-sized carbon black can have better mechanical and thermal properties than micron, resulting in less material being needed for a given application at a lower cost.

Keywords: Low-density polyethylene, Nano-micro carbon black, Thermal conductivity, Nanocomposites and SEM

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MATERIALS

1. Introduction

Lightweight and low-cost polymers with superior electrical insulation have been widely used as packaging materials in electrical devices because of their good processability. Nonetheless, polymer thermal conductivity is typically poor (i.e. 0.10–0.25 W/m. K), and it has long been known that increasing the thermal conductivity of these polymers is important to minimize energy loss and improve device stability. The manufacturing of insulating polymer materials with high thermal conductivity has become important as the demand for high energy transmission and high-density power in electronic devices has grown [1]. Polymer nanocomposites, which are made up of a polymer matrix and micro-sized fillers, have stronger physical and mechanical properties than traditional composites. As a consequence, a number of polymer matrixes, such as polypropylene (PP), polyethylene, polystyrene, poly(ethylene oxide), and polyamide-6, have been studied. PP nanocomposites are of great interest because of their thermal stability, increased mechanical strength, stiffness, and low gas permeability, among other properties that have made them ideal for applications in the packaging and automotive industries [2]. Nano-sized reinforcements can have greater mechanical and physical characteristics than micron or larger sized reinforcing materials, resulting in less material being needed for a given application at a lower cost. Nanocomposite polymeric materials can be used in addition to the plastics industry as technical textiles in a variety of industries, including automotive, aerospace, defense,

construction, and others. They can act as load-bearing structural materials either directly or indirectly. Nanomaterials including carbon black, carbon nanotube, silica and clay have all been researched and used in the development of nano polymer composite materials [3]. The electrical resistivity of carbon black (CB) or carbon fibers (CF) filled polymer composites is highly dependent on the filler material. At low filler concentrations, the composite resistivity nearly matches that of the polymer matrix. Clusters of conductive particles agglomerate in the composite. With increasing filler content, the size and number of clusters grow larger and larger until they reach a critical stage, known as the percolation threshold, where the cluster becomes infinite and the material becomes conductive [4]. Photons, electrons, and phonons transport energy in solids. Due to the fact that most polymers prevent electrons from moving freely, the primary heat conduction mechanism is phonons, quantized modes of vibration occurring in a rigid crystal lattice. Carbon-based fillers tend to be the most promising, as they combine the lightweight with high thermal conductivity [5]. Traditional carbon-based fillers include graphite, carbon fiber, and carbon black. Carbon black particles are aggregates of graphite microcrystals with particle sizes varying from 10 to 500 nm and surface areas ranging from 25 to 150 m²/g. Thermal conductivity, rather than electrical conductivity, is said to be aided by carbon black [6,7]. The development of LDPE/inorganic nanocomposites with improved barrier and mechanical properties is expected to enhance its usage in this direction even further. Because of the branched

macromolecules (in LDPE) [8], which prevent the polymer chains from penetrating into the clay galleries, LDPE nanocomposite structures with heavily scattered inorganic nanoparticles are far more difficult to achieve than HDPE nanocomposite structures [9]. It is well known that the geometric shape, dispersion state, particle size, particle size distribution, and surface properties may all have a major effect on composite properties. Polymer nanocomposites are gaining popularity as a result of the recent commercial availability of nanoparticles. Mechanical properties such as strength, modulus, and dimensional stability, as well as permeability to hydrocarbons, water, and gases, chemical resistance, flame retardancy, thermal stability, and electrical, dielectric, magnetic, and optical properties, have all improved significantly [10].

This work-study the effects of micro and nanoparticles of CB at various weight percentage, filled LDPE mixing in twin screw extruder on the morphological, thermal and mechanical properties of composites and compared with pure polyethylene (PE), SEM device is used to check the homogeneity of CB particles inside LDPE matrix and thermal conductivity is tested in two methods also elastic modulus tested.

2. Materials and experimental method

2.1. Materials

The matrix is made of low-density polyethylene (LDPE). Micro carbon particles (CB) from the Babylon tire factory and nanocarbon particles (CB) from Shijiazhuang Sun Power Technology Co., Ltd, China, were used as reinforcement materials, with particle sizes ranging from 95 nm to 5 μm for microcarbon and 20-30 nm for nanocarbon.

2.2. Micro and nanocomposites polymer preparation

Both nano and micro-CB particles melt mixed with LDPE pellets in weight ratios of 2%, 4%, and 8% to obtain the necessary micro and nano – polymer composites. Twin-screw extruder with a speed of 12 rpm and temperature 190°C was used for composites preparation. The CB was combined with acetone to create a suspension that was then blended with LDPE before going through the extruder to get a more homogenized mixture. After mixing in extruder the resulting sheets were hot-pressed by using thermal-hydraulic pressing type (XLB-plate vulcanizer), at temperature (150°C), pressure (5 MPa) and time (15 min) to produce a

uniform sheet for produce the samples according to ASTM for each test.

3. Characterization

3.1. Fourier Transformation Spectroscopy (FTIR)

FTIR test was carried out using Fourier transform infrared spectrometer type IR Affinity-1, this test was achieved to study the interaction between the matrix and reinforcement phase.

3.2. Tensile test

The tensile test on the specimens was carried out using universal testing machine according to ASTM D 638-03 at a strain rate of 10 mm/min (WDW-5E machine).

3.3. Hardness test

Hardness of micro and nanocomposites were measured using shore D hardness tester under ASTM D 2240, by taking five reading for each sample and then take the average.

3.4. Thermal conductivity test

Thermal conductivity is one of test important packaging test, since thermal abuse is a major problem when shipping temperature-sensitive commodities including food, pharmaceutical, electronics, and horticulture products. Insulated packaging can help keep product temperatures within acceptable ranges and prevent product deterioration in transportation until it reaches the customer [11].

Two different methods with two different devises are used to measure the thermal conductivity of CB micro and nanocomposites

Method (A): The thermal conductivity, thermal diffusivity, and specific heat capacity are measured across a temperature range of -60°C to 200°C by TPS 500 using the equation below[12]:

$$\Delta T = P_0 / (\pi^{3/2} r k) D(\tau) \quad (1)$$

where: ΔT is the rise in temperature, P_0 is the generated electric heat, r is the disk radius, and k is the measured thermal conductivity. τ and $D(\tau)$ are functions. The thermal conductivity can be calculated using the slope of Equation. To reduce errors, ensure good thermal contact between the sample and the heating element when using this method [11].

Method (B): Thermal conductivity again test using YBF-3 thermal coefficient meter instruction, the thermal coefficient of elastic is calculated from the equation below:

$$\lambda = \frac{mchB}{(\pi RB^2(V_1 - V_2)) \cdot ((2hp + 2Rp - \pi r^2)) / ((2Rp + 2hp))} \quad (2)$$

$$\Delta V / \Delta t \quad | \quad T = T_2$$

where: c is the specific heat capacity of the copper plate is $c = 385 \text{ J/kg} \cdot ^\circ\text{C}$, hp is the thickness of the radiating disk (the lower copper plate) $hp = 7.01 \text{ mm}$; Rp the radius of the radiating disk (the lower copper plate) $Rp = 65.00 \text{ mm}$; the mass m of the radiating disk (the lower copper plate) $m = 824 \text{ g} = 0.824 \text{ kg}$; the thickness hB of the sample and RB the radius of the sample.

In order to measure the thermal coefficient of the material, start the measurement from the definition of thermal conductivity and its physical meaning. The law of thermal conductivity indicates: if heat conducts along with Z direction, take a vertical sectional d at any position Z_0 on Z -axis.

3.5. Differential Scanning Calorimetry (DSC) test

DSC model TA-60WS has been used to study thermal characteristics of LDPE and its composites. The samples with a weight of $(8-10 \text{ mg} \pm 0.5)$ were tested under inert gas atmosphere with a heating and cooling rate $10^\circ\text{C}/\text{min}$ from 30 to 250°C and then cooled to 30°C . The degree of crystallinity (X_c) for LDPE polymer and its composites calculated from the equation [13]:

$$X_c = (\Delta H_m / \Delta H_o) * W_f * 100\% \quad (3)$$

where: ΔH_m : is the melting enthalpy recorded from DSC, ΔH_o : is the melting enthalpy for fully crystalline PE (290 J/g) [13], W_f : is the weigh fraction of LDPE in composite.

3.6. Scanning Electron Microscope (SEM test)

SEM analyses were carried out in the Tescan scanning electron microscope machine model INSPECT S50 made in Cheeky. The surface of the samples was coated with gold. The samples were tested utilizing 5 kV at different magnification to compare them for each sample.

4. Results and discussion

4.1. FTIR result

Figure 1 shows the FTIR spectrum for LDPE, LDPE shows many bands. The bands at 2924.09 and 2854.65 cm^{-1}

for CH_2 stretching, the band at 1465.90 cm^{-1} for CH_2 bending, and the bands at 717.52 and 540.07 cm^{-1} for CH_2 rocking [8]. The FTIR analysis of LDPE/nano-CB and LDPE/micro-CB composites shows there are no changes in the spectrum of LDPE which indicates that there is no chemical interaction between LDPE and CB and the presence of only physical interaction with a slight shifting in the bands of LDPE [14]. The changes in band values intensities or shiftings are shown in Table 1.

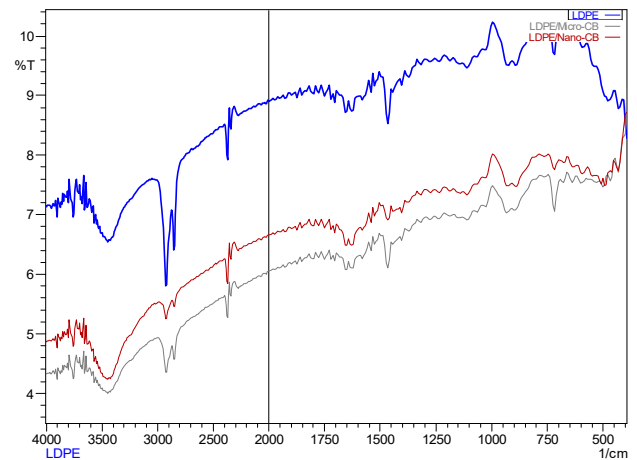


Fig. 1. Show FTIR Spectrum of Pure LDPE, LDPE/Nano-CB and LDPE/Micro-CB

Table 1.

FTIR transmission bands spectrum for LDPE, LDPE/nano-CB, and LDPE/micro-CB

Bond type	LDPE standard [8]	LDPE experimental	LDPE/CB nano	LDPE/CB micro
CH_2 stretch	2939-2850	2924.09-2854.65	2924.09-2846.93	2916.37-2854.65
CH_2 bend	1470	1465.90	1465.90	1465.90
CH_2 rock	720	717.52	717.52	717.52

4.2. Tensile test result

Tensile experiments were used to evaluate the reinforcing effect of nanofillers on nanocomposites' mechanical characteristics as shown in Figure 2. According to the findings, the nanofillers altered the properties of LDPE. The tensile strength of the LDPE/CB nanocomposites increased with the addition of CB nanoparticles. Tensile strength was increased from 13.536 MPa for pure LDPE to (15 MPa , 19.71 MPa) at 2% and 4% nano-

CB, respectively, also the tensile strength increased for the same percent (2%, 4%) from micro-CB to 14 MPa at 2% micro-CB, and 17 MPa at 4% micro-CB. This increase in tensile strength correlated with an increase in the degree of crystallization that led to improved intermolecular bonding between particles and matrix, that later appear in DSC results. While increasing CB content (in both nano and micro), the tensile strength decreases to 11.03 at 8% nano-CB, and 9.8 at 8% micro-CB, this results due to particle content rose, and agglomeration was formed that led to decrease in particle-matrix interfaces, and reduce the stress transfer from matrix to particle and a rise in particle-particle interfaces induced a change in the failure mechanism, resulting in a decrease in tensile strength [15-17]. The rate increment in tensile strength at (2%, 4%) nano-CB is about 10.8%, 40.7% respectively, and the rate increment at (2%, 4%) micro-CB is about 3.7%, 25.9% respectively. Conclude the values of tensile strength with addition nano-CB higher than tensile strength with addition micro-CB.

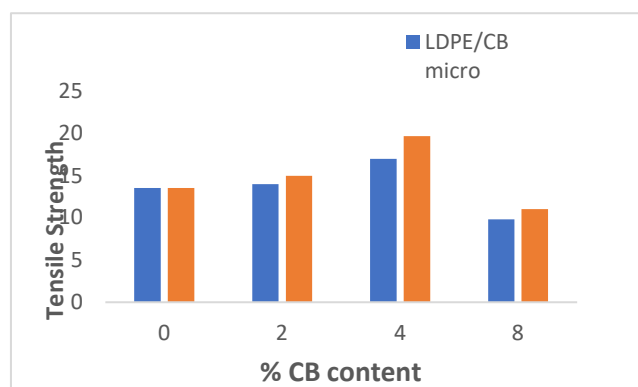


Fig. 2. Tensile strength of micro and nanocomposites of LDPE/CB

Figure 3 demonstrates the effects of particle loading on the elastic modulus of LDPE/CB composites. It demonstrates that particle size loading has an effect on the modulus in the range studied. The elasticity modulus of LDPE/CB nanocomposites increased with increase CB nanoparticle content, the modulus of elasticity increase from 15 MPa to 61.25 MPa at 8%wt. nano-CB. The rate of increase in the elasticity as follows: (60%, 96%, 308%) at (2%, 4%, 8%) nano-CB respectively. This means increasing the stiffness of LDPE with the addition of CB due to increases of the toughness of polymer LDPE. Furthermore, since the rigidity of inorganic reinforcement is typically much higher than that of organic polymers, adding rigid particles such as CB nanoparticles to a polymer matrix LDPE will easily increase the modulus. In general, when the particle size decreases

reached a critical size, the elastic modulus increases (in research done by Fu et al. [18] they assumed that the critical size such as 30 nm has an obvious effect of particle size on the modulus predicted theoretically).

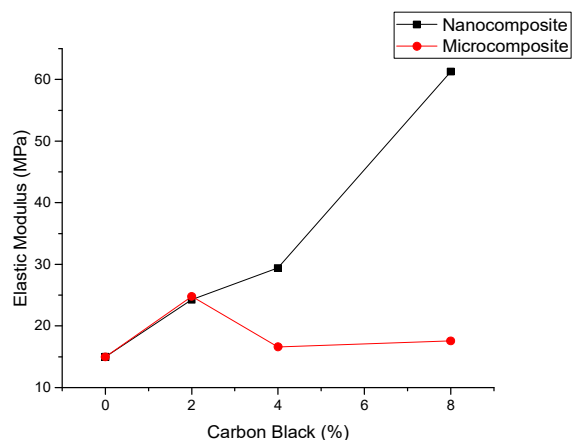


Fig. 3. Show the behavior of elastic modulus of LDPE mixed with nano and micro CB particles

In the present study, we have obtained an increase in elastic modulus with increasing particle loading of CB nanoparticles with a particle size in a range of 20-30 nm, which proves the theory that assumed by Fu et al. [18]. In some cases, elastic modulus decreases at a critical weight percentage at which agglomeration may occur. This results partially agreements with Gacitua, et al. [19]. The effects of particle loading on the elastic modulus of LDPE/CB composites. The modulus is shown to be influenced by particle size in the range studied.

Figure 3 also shown effect microparticles of CB on the elasticity modulus of LDPE, the results show that the elasticity keeps stable as the filler concentration increases from 0 to 8% CB, except at 2% micro-CB where the peak has occurred, the modulus increased from 15 MPa (for pure LDPE) to 24.818 MPa at 2% CB, this is maybe due to the weak adhesion between fillers and the LDPE matrix. The rate of increase in the elasticity as follows (65%, 11%, 17%) at (2%, 4%, 8%)wt. of micro-CB respectively. It was conclude that the elastic modulus improvement for LDPE with the addition nanoparticle more than addition microparticle.

4.3. Hardness test result

Figure 4 show the variation of shore D hardness of LDPE with two sizes (nano and micro) carbon black particles. It is obvious that the addition of CB particles improves the hardness of LDPE composites as the amount of CB increase,

the hardness of LDPE has raised by 5 points when nano-CB particles was added to LDPE while the hardness of LDPE raised by 3 points when micro-CB particles was added, this improvement is due to the smaller sizes of nano-CB particles and the better dispersion in LDPE matrix also the less agglomeration of nano particles.

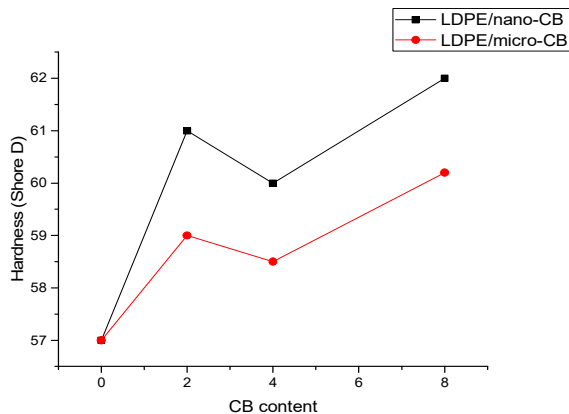


Fig. 4. show shore D hardness of LDPE/CB micro- and nanocomposite

4.4. Thermal conductivity result

Figure 5 shows the effects of CB nano and microparticles on the thermal conductivity of composites and compared with pure LDPE using TPS 500 these results of two methods are obtained and compared. Incorporation of nano-CB increased thermal conductivity from 0.33 W/m.K (for pure LDPE) to maximum value 0.436 W/m.K at 2%wt. ,also the addition of micro-CB led to increase thermal conductivity to maximum value 0.7663 W/m.K at 2%wt. this belongs to good dispersion of CB in the matrix of LDPE also there are maybe not high agglomerations obtained. The increasing of thermal conductivity at low carbon content because of the good dispersion of CB

Thermal conductivity has been decreased to (0.245, 0.1613) W/m.K at (4%, 8%) wt. from nano-CB, and Figure 5 also shows the addition of micro-CB reduced the thermal conductivity to (0.4775, 0.4524) W/m.K at (4%, 8%) wt. from micro-CB ,but remains higher than pure LDPE, this happen due to agglomerations of CB and which leads to decreased the electrical activity beside to minimum dispersion of CB micro and nanoparticles so that the insulation matrix surrounding the conductive fillers leading to decreasing the thermal conductivity. We conclude by incorporating nano-CB into the polymer matrix, thermal conductivity is lower compared with addition micro-CB, the material used for food packaging should be thermally

insulating and this was achieved by adding nanoparticle carbon black to LDPE .

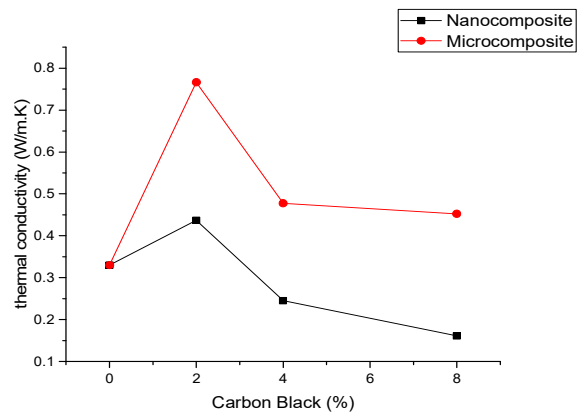


Fig. 5. show the behavior of thermal conductivity of LDPE mixed with nano and micro-composites CB particles using TPS500

Figure 6 shows the effects of CB nano and microparticles on the thermal conductivity of LDPE using YBF-3 thermal coefficient meter instruction. The thermal conductivity of microcomposite obtained a maximum value at 2% micro-CB (0.62234 W/m.K) and decreasing to (0.18645 W/m.K) at 4% micro-CB then return to the original value (0.3406 W/m.K) at 8% micro-CB. This behavior confirmed with that produced in Figure 5 for the same material.

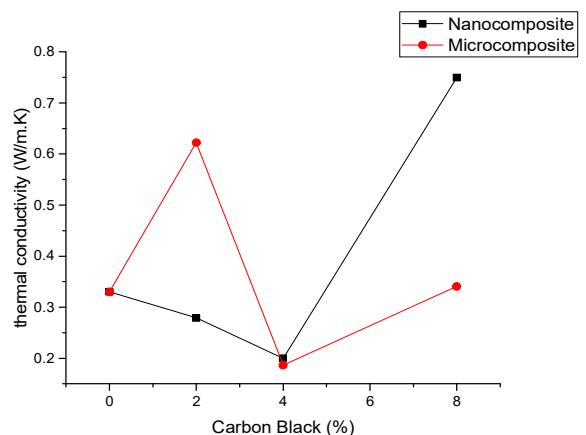


Fig. 6. show the behavior of thermal conductivity of LDPE mixed with nano and micro CB particles using YBF-3 thermal coefficient meter instruction at 60°C

Figure 6 also show the thermal conductivity of LDPE with addition nano-CB. Nanocomposites indicate decreasing in thermal conductivity up to (0.1999 W/m.K) at 4% nano-

CB after that this property reaches a maximum value (0.7494 W/m.K) at 8% nano-CB. The unconfirmed between this behavior and that in the Figure 5. for the same value may be due to the non-uniform distribution of nano CB particles in LDPE. Or due to the different temperatures applied in the two methods.

4.5. DSC result

The melting and crystallization temperature of LDPE and its nano and micro composite have been studied using DSC. Figure 7 shows the effect of both nano and micro carbon black on the melting temperature of LDPE, the melting temperature of neat LDPE is 112.8°C when nano-CB added to LDPE its found that melting temperature has been increased to 137.6°C, increment by 24.8°C, and when micro-CB added to LDPE its found that melting temperature increased to 133.5, increment by 20.7°C. Also, there is an improvement in crystallization temperature T_c as shown from Figure 8, this improvement is due to both nano and micro particles act as nucleating agent and improve thermal properties of LDPE composites. The degree of crystallinity of LDPE composites also enhanced with both additives (nano, micro) as shown in Table 2. This explains the improvement in mechanical properties.

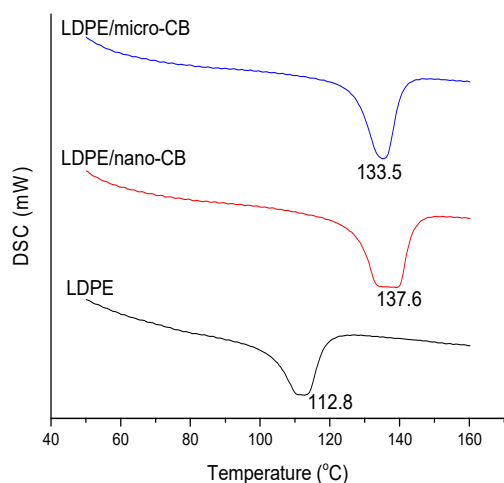


Fig. 7. Show The Melting Temperature(T_m) of LDPE, LDPE/Nano-CB and LDPE/Micro-CB

Table 2.

DSC data of LDPE, LDPE/Nano-CB, and LDPE/Micro-CB

Sample	T_m , °C	ΔH J/g	T_c , °C	X_c , %
LDPE	112.8	62.76	96.3	21.64
LDPE/nano-CB	137.6	88.45	116	28.06
LDPE/micro-CB	133.5	72.62	115.1	23.03

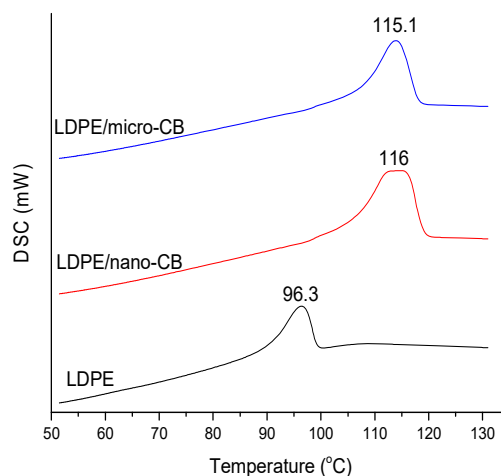


Fig. 8. Show The Crystallization Temperature(T_c) of LDPE, LDPE/Nano-CB and LDPE/Micro-CB.

4.6. Scanning Electron Microscope (SEM) results

SEM was used to analyze the morphology of the samples in order to gain a better understanding of the effect of increasing CB weight percent on the thermal and mechanical behavior of the LDPE, as well as investigate the distribution of CB within the LDPE matrix and its relationship to the composites' thermal behavior (Fig. 9). The images indicate that the CB nanoparticles were uniformly distributed in the matrix reinforcement system, but that the dispersion was not fully well coordinated, despite the fact that there was very little agglomeration of the nanoparticles. Also, there are clearly areas where the nano-particles are more concentrated, with a little agglomeration of CB nanoparticles seems to like very tiny grains noticeable from the matrix. There is a very even distribution of carbon black nanoparticles, with particles attached to the LDPE surface. The morphology of the nanocomposites was analyzed at different magnifications. As CB content rise, the adhesion between the phases weakens, and the nanoparticles act as holes, cavities, or nanostructured flaws, causing local stress concentrations and decreasing particle-matrix transfer [20,21]. Also, the effects of the magnification is shows in Figure 9 are measured at two scales for composites (20 and 50 μm) while the pure sample at 5 μm to see the effects of particles on the homogeneity and distribution in polymer matrix (LDPE), with increasing CB content that obtained smooth surface in both scales (20 and 50 μm) at 8% CB while at 2% CB and 4% CB can obtain surfaces with small roughness. However, due to their high surface energy, CB nanoparticles appear to agglomerate with increasing

concentration [22]. This is the reason for the decrease in mechanical properties of composite when the amount of CB nanoparticles increased to 8%. When comparing LDPE reinforced with microparticles to LDPE reinforced with nanoparticles, where the number of voids is less and homogeneity is higher, we notice that the amount of agglomeration is greater in micro composites, which is

reflected in mechanical properties that are less than those of the nano-composites as shown by Figure 2 and Figure 3 for tensile properties these result is in agreement with J. Zhoa and Q. Yang [23] which they study the effect of carbon black content on thermal properties of HDPE and its shown that the CB form aggregates as its content increase from 0 to 12%.

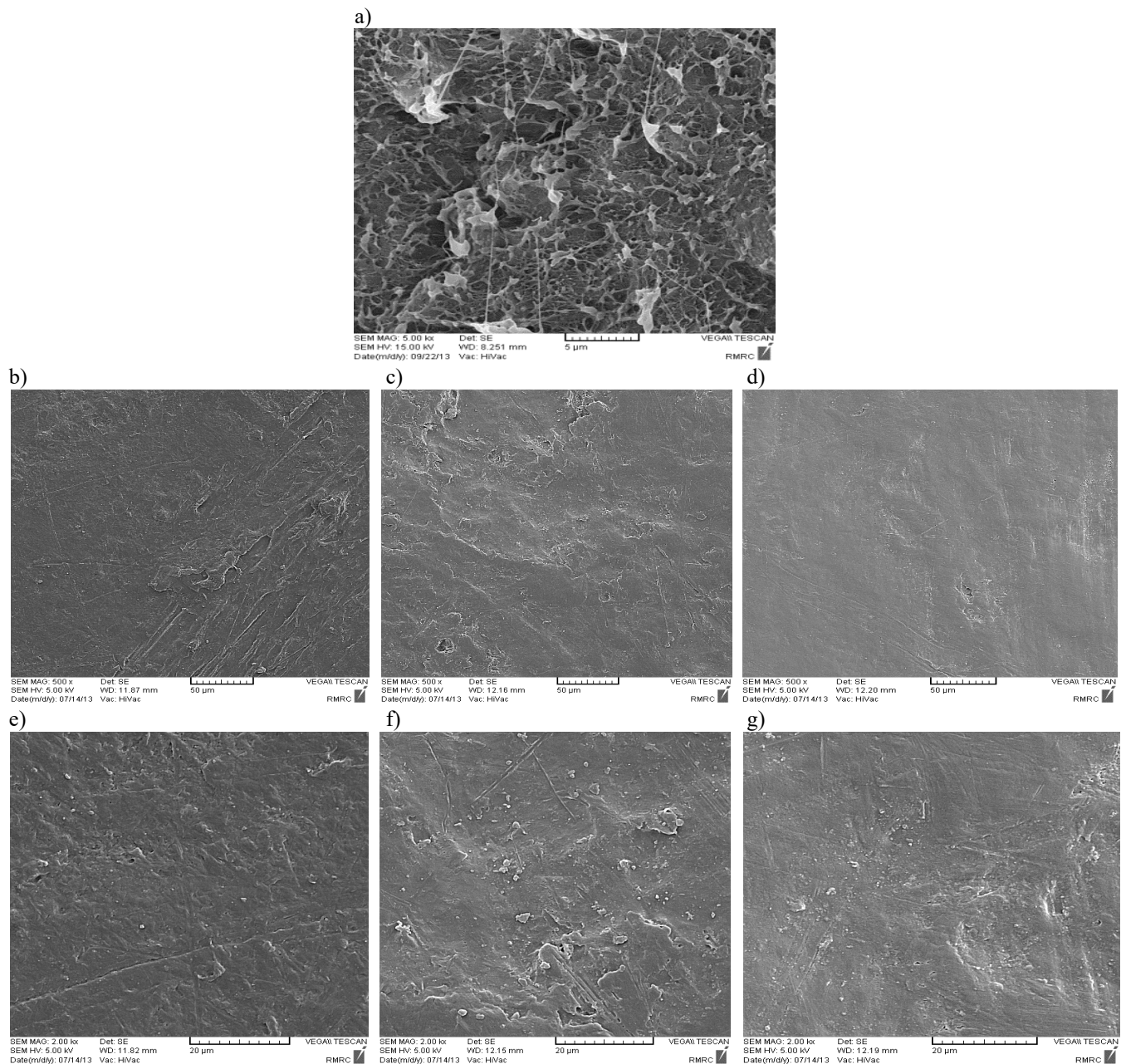


Fig. 9. SEM images of pure LDPE (a) and LDPE/CB Micro/Nano-composites at different CB percentages: b), e) 2%, c), f) 4%, d), g) 8%. The b), c), d) images for micro-composite. The e), f), g) show images of nanocomposites

5. Conclusions

1. The result of tensile test show the tensile strength increase with increasing nano/micro content carbon black, while the tensile strength decreasing at 8% for both nano and micro CB, the value of tensile strength of LDPE/CB nanocomposite higher than LDPE/CB micro-composites, Elastic modulus increases with nano CB particles increasing, while keeps stable and reach peak value at 2% for micro composites.
2. We conclude the mechanical properties were improved by the addition carbon black nanoparticles more than addition micro-carbon black.
3. We conclude by incorporating nano-CB into the polymer matrix, thermal conductivity is lower compared with addition micro-CB, the material used for food packaging should be thermally insulating and this was achieved by adding nanoparticle carbon black to LDPE.
4. FTIR show physical interaction between LDPE and nano/micro particles carbon black.
5. DSC result show improvement in crystallization temperature (T_c), melting temperature (T_m) and degree of crystallization with addition nano and micro carbon black, but for LDPE/nano-CB higher than LDPE/micro-CB.
6. The SEM images show smooth surfaces and no mixture separation with increasing CB content at different magnification.

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