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Mechanical and structural properties of polypropylene composites filled with graphite flakes

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ABSTRACT

Purpose: The goal of the work is to study the polymer matrix composite (PMC) with isostactic Polypropylene (iPP) as a matrix material and graphite as a filler.

Design/methodology/approach: Density and tensile strength have been carried out and the relationship between the graphite content and properties were examined. X-Ray Diffraction (XRD), Differential Scanning Calorimeter (DSC) and SEM facilities were applied on the polished and fractured surfaces of PMC.

Findings: Results showed that values of tensile were decrease by increasing graphite. Fractured surfaces showed that good interaction between the polypropylene and graphite particles. XRD and DSC investigations showed that the crystallization ratio changes in the iPP composites.

Research limitations/implications: Graphite addition was observed to be an alternative additive to the iPP. If suitable coupling agents could be added to graphite and iPP mixtures, the mechanical properties can possibly be increased.

Originality/value: It is thought that the nucleating effect of the graphite leads to an increased rate in orientation of the polymer.

Keywords: Polymer; Filler; Crystallization; Thermal; Microstructure

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1. Introduction

Several series of conductor-polymer composites were prepared from metal, graphite and conducting ceramics as filler materials, and epoxy, silicone rubber, polyethylene and polypropylene as polymer matrix. In the past several years, some composites containing dispersed conducting particles in an insulating polymer matrix have been studied for applications such as thermostats and pressure sensors [1]. PP is on the hand and ordinary commodity polymer with a very simple chemical structure which is probably the most used mineral filled thermoplastic polymer for injection molding applications [2,3]. The understanding of mechanical properties of the composites is very important in order to design pressure sensors.

Polymer- particle composites have received considerable interest in the materials field because of their potential for large gains in mechanical and morphological properties. Thermoplastic polymers and especially polyolefins are produced and consumed today in vast quantities. However, they are seldom used as pure polymers and are usually combined with mineral fillers. Fillers

find application in the polymer industry almost exclusively, e.g. to improve mechanical, thermal, electrical properties and dimensional-stability [4-9].

The morphological features of semi-crystalline polymers such as crystalline level, crystal thickness, etc. are mainly governed by the nature of the polymer. Crystalline morphology is also strongly sensitive to thermo-mechanical treatment and processing. The five major methods of determining crystallization are based on specific volume, X-ray diffraction, thermal analysis, nuclear magnetic resonance, and infrared spectroscopy [10-13].

In the present work, test materials have been prepared a series of filled iPP composites with graphite filler loading (10–50 wt. %), to study the effect of the filler content. The aim of this work is to study the effect of graphite on physical, mechanical and morphological of the injection molded iPP.

2. Experimental

In this study, in order to characterize the polymer/graphite composites that produced tensile testing, density, XRD, EDS and SEM were utilized. A series of filled iPP composites with graphite filler loading (10-30-50 wt. %) was prepared to study the effect of the filler content. Three different weight percentage of graphite was introduced to iPP to investigate the relationships between iPP and graphite combinations. The polymer matrix material that used in this study is a commercial grade iPP supplied by Turkish Petkim Polymer Industry. Graphite that approximately particle size was 29.4 μm obtained from the Graphite Kropfmühl AG were used in PMC.

For tests the samples were manufactured in the form of rectangle plates. For production of graphite filled polymers an injection apparatus was used which has four different heating stages of 175, 180, 185, 190 °C. Injection and molding pressure were chosen as 7 MPa and 11 MPa respectively. The mould temperature was fixed at 30 °C and the pressure was applied for 30 sec.

Tensile tests were carried out on an Instron 3367 universal test machine. The procedures were defined by the ASTM D 638 standards. ASTM D 792 standard was used for the density tests. Rigaku D/MAX/2200/PC model XRD analysis was done on PMC that prepared metallographically by polishing with 1000 grid emery paper. Perkin Elmer DSC system was employed for thermal characterization. The SEM and EDS studies of the graphite filled polymers were obtained by using Jeol JSM-6060LV Scanning Electron Microscopy. Before the SEM studies, the gold coating was applied on the samples surface for the conductivity of the samples.

3. Results and discussion

Physical and mechanical properties of graphite filled iPP are given in Table 1.

Processing conditions were chosen same for all the composites, the interaction between inorganic and organic materials is very slight. The addition of graphite the higher the elastic modulus and the density while the lower the max. tensile strength, the tensile strength to fracture and the tensile strain to fracture as expected. The reduction of elongation at break values with increasing graphite content is indirect evidence that implies the bonding strength is poor. The polymer chains have enough time and amorphous regions with applied load. After immediately orientation of chains during tests the crystallization start formation and this causes increase in elastic modulus of composite [7,14]. Also, the filler added to polymer matrix restriction the motion of polymer chains and thus lowers tensile strain to fracture sharply. It was found that the density of test materials increased with the graphite content. Results showed that almost there is a linear relationship between mechanical properties and graphite weight percent. It is clear from the experiments that tensile and density values are influenced mainly by the ratio of graphite fillers.

Table 1.
Physical and mechanical properties of graphite filled iPP

Graphite content (wt. %)	Tensile strength max. (MPa)	Tensile strength to fracture (MPa)	Tensile strain to fracture (%)	Elastic modulus (MPa)	Density (g/cm^3)
10	29.31	18.78	5.17	2476.34	0.98
30	20.72	8.55	1.60	4900.95	1.04
50	17.44	6.40	1.53	5538.80	1.67

XRD pattern of the graphite is given in Figure 1. It is seen that in Figure 1-a and Figure 1-b the iPP and the graphite are in the form of Graphite (2H-C) 41.1487 and Isotactic Polypropylene (C_3H_6) 45.1807, respectively. In Figure 1-a, two different crystal planes of the iPP material are shown with 1 and 3 peaks. XRD analysis is also used for determining the orientation relationships in the filled iPP. The diffraction peaks were observed in 2 theta angles of 13.9^0 and 16.7^0 for planes corresponding to 1 and 3 respectively [4,15]. The intensity ratios of these two peaks are identified as the orientation of the crystal paleness for iPP [11,13]. The X-ray diffraction analysis proved that introducing of graphite particles resulted in an interaction of crystallization structure of iPP. In this study, it is expected that the plates of graphite particles lie parallel to the surface of the mold, and this orientation can induce a preferential orientation in the morphology of iPP crystals placed near the filler [16,17]. As shown in Table 2 the crystal orientation ratio of the (I_2/I_1) planes produced a value of 0.79. The more addition of graphite increases the orientation ratio (3.60).

DSC analysis was applied to the unfilled iPP and the composites of iPP filled with graphite flakes. The peak temperatures of the scans were taken as the melting temperature of the polymer. The peak area was used to determine the melting enthalpy. The crystallization of iPP and the nucleation activity of the filler were calculated. The crystallization was calculated with the Equation 1.

$$Xc = \frac{\Delta H_m \cdot (m_c / m_p)}{\Delta H_o} x100 \quad (1)$$

Where ΔH_m is the melting enthalpy measured in the heating experiments, ΔH_o is the theoretical value of enthalpy of 100 % crystalline PP (ΔH_o = 207.1 J/g), m_c is the mass of the sample, and m_p is the mass of iPP in the sample.

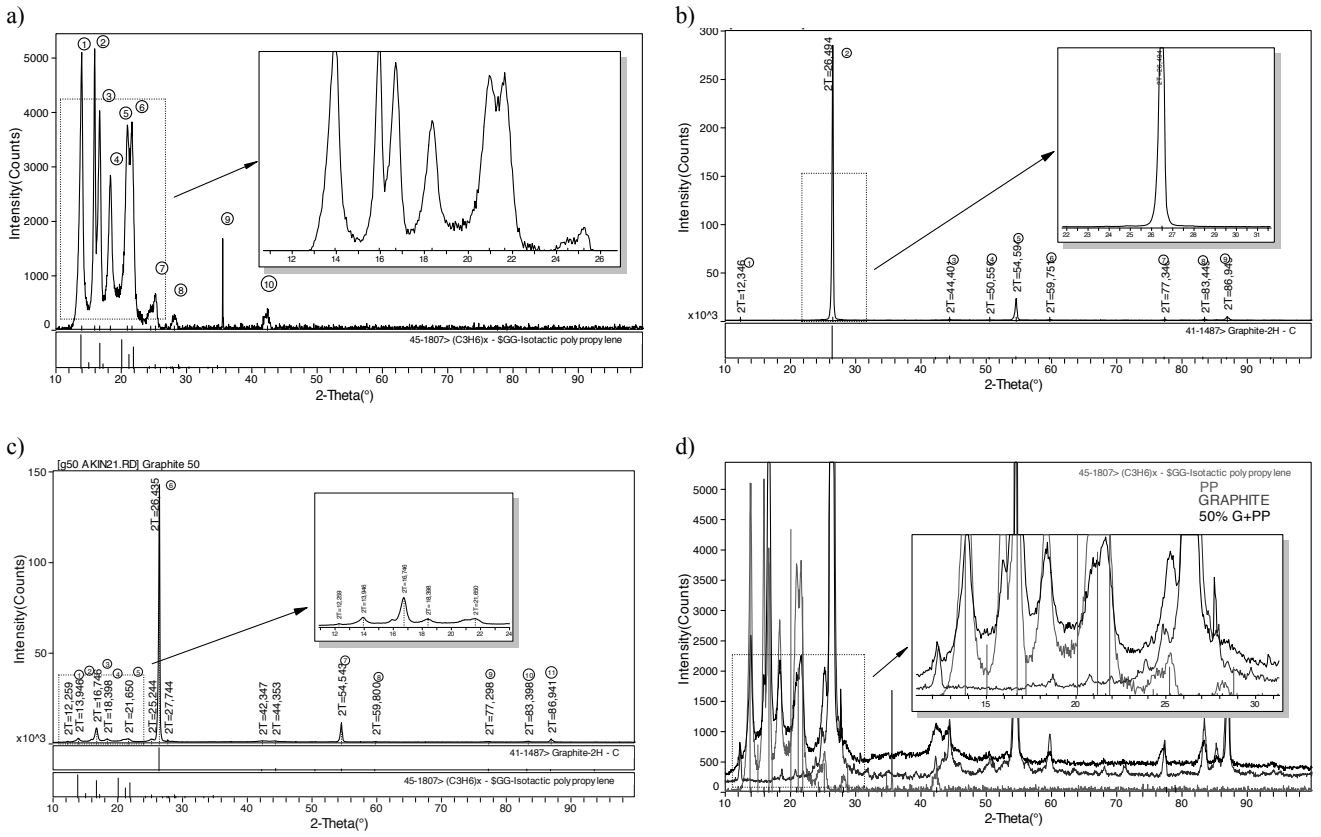

Fig. 1. XRD patterns of the pure iPP (a), graphite (b) and including 10–50 wt. % graphite iPP matrix composites (c), (d)

Table 2.
The peak Intensity (Counts) of the (1) and (2) planes and crystal orientation ratio I_2/I_1 for iPP composites

Graphite content (wt. %)	Intensity (Counts)		I_2/I_1	Melting Enthalpy ΔH (J/g)	Melting Temp. T_m (°C)	Reduced Melting Enthalpy ΔH (J/g)	Crystallization (%)
	I_1	I_2					
0	5100	4050	0.79	66.43	163.73	66.43	32.00
50	2500	9000	3.60	19.72	165.11	39.44	38.09

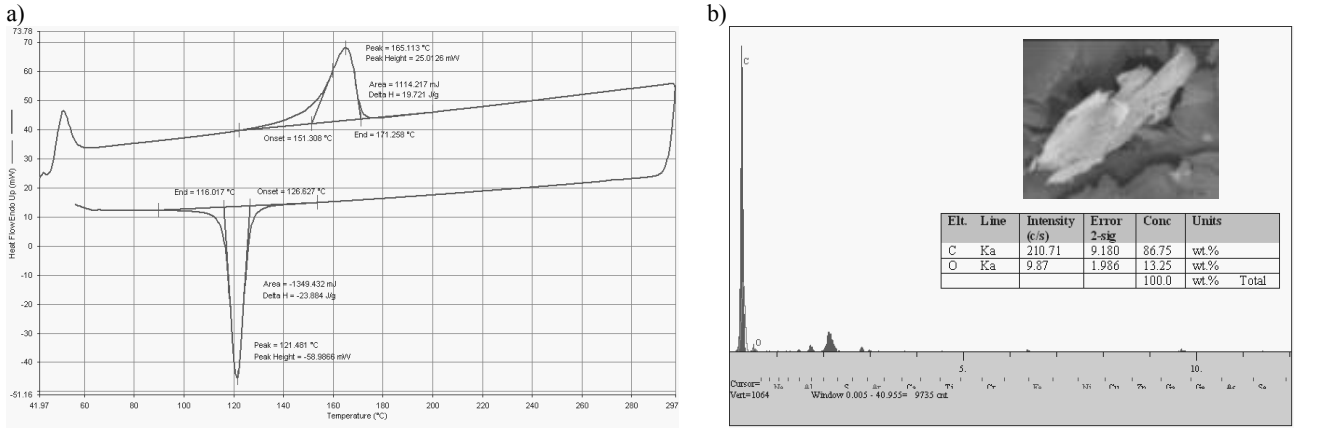

Fig. 2. DSC thermogram (a) and EDS analysis (b) of iPP composite included 50%wt. graphite

As it can be seen in Figure 1-c and Figure 1-d, intensities of peaks of polymer decreases with increased the amount of graphite content. The graphite peaks have been dominant with increasing graphite content of composite. It is possible to claim that this affect mechanical and physical properties of composite. DSC thermogram and EDS analysis of iPP composite including 50%wt. graphite filler is given in Figure 2.

Fractured surfaces of PMC materials, including 10%, 30 % and 50% of graphite compositions, attained as a result of tensile strength test are analyzed by SEM method (Figure 3). The light areas reflect graphites and the dark areas show iPP. In Figure 3-a and Figure 3-b, iPP deforms in shape of fibrous nature. The photograph in Figure 3-b shows the left upper corner of the photograph in Figure 3-a. In Figure 3-c, the particle is not removed from surface. These can be attributed to existence of the bond between particle and matrix. In Figure 3-d, it can be seen that graphite grain which has a size 3x5 μm are not spilled from iPP matrix.

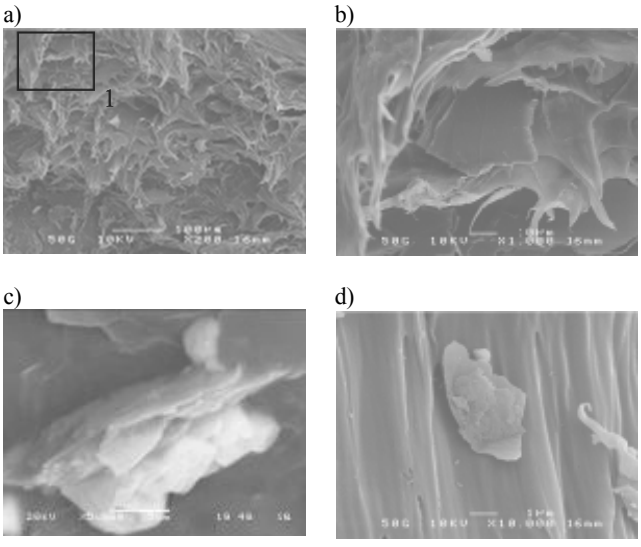

Fig. 3. The microstructure of the fractured surfaces of the composite, a) 200X, b) 1000X, c) 5000X, d) 10000X

4. Conclusions

In present study, it was found that, the content of graphite filler affected structural integrity and mechanical properties of composites. Graphite can be added to the polymers up to 30-50 wt. % to increase the elastic modulus and density. With increasing the amount of the graphite addition to the iPP results in a decrease in elongation at break values. XRD investigations showed that the crystallization ratio changes in the iPP composites. It is thought that the nucleating effect of the graphite leads to an increased rate in orientation of the polymer. From the DSC studies the crystallization was increased by increasing the graphite content. Graphite addition was observed to be an alternative additive to the iPP. If suitable coupling agents could be added to graphite and iPP mixtures, the mechanical properties can possibly be increased.

References

[1] H. Suzuki Y. Ishigure, S. Iijima, H. Ito, T. Ota, H. Unuma, M. Takahashi, Y. Hikichi, Electrical and elastic properties of conductor-polymer composites, Journal of Materials Science 34 (1999) 2979-2985.

[2] S. Nagasawa, A. Fujimori, T. Masuko, M. Iguchi, Crystallization of polypropylene containing nucleators, Polymer 46 (2005) 5241-5250.

[3] I.A. Tchmutin, A.T. Ponomarenko, E.P. Krinichnayab, G.I. Kozubb, O.N. Efimov, Electrical properties of composites based on conjugated polymers and conductive fillers, Carbon 41 (2003) 1391-1395.

[4] A.S. Luyt, V.G. Geethamma, Effect of oxidized paraffin wax on the thermal and mechanical properties of linear low-density polyethylene–layered silicate nanocomposites, Polymer Testing 26/4 (2007) 461-470.

[5] P. Mareri, S. Bastide, N. Binda, A. Crespy, Mechanical behaviour of polypropylene composites containing fine mineral filler: Effect of filler surface treatment, Composites Science and Technology 58/5 (1998) 747-752.

[6] B. Pukanszky, G. Voros, Stress distribution around inclusions, interaction, and mechanical properties of particulate-filled composites, Polymer Composites 17/3 (1996) 384-392.

[7] M. Micusik, M. Omastova, Z. Nogellova, P. Fedorko, K. Olejnikova, M. Trchova, I. Chodak, Effect of crosslinking on the properties of composites based on LDPE and conducting organic filler, European Polymer Journal 42/10 (2006) 2379-2388.

[8] A.O. Maged, A. Ayman, W.S. Ulrich, Influence of excessive filler coating on the tensile properties of LDPE–calcium carbonate composites, Polymer 45/4 (2004) 1177-1183.

[9] J. Jancar, Advances in polymer science. Mineral fillers in thermoplastics I, vol. 139, Springer, Berlin/Heidelberg, 1999.

[10] T. Labour, C. Gauthier, R. Seguela, G. Vigier, Y. Bomal, G. Orange, Influence of the β crystalline phase on the mechanical properties of unfilled and $CaCO_3$-filled polypropylene. I. Structural and mechanical characterisation, Polymer 42/16 (2001) 7127-7135.

[11] J.I. Velasco, C. Morhain, A.B. Martinez, M.A. Rodriguez-Perez, J.A. de Saja, The effect of filler type, morphology and coating on the anisotropy and microstructure heterogeneity of injection-moulded discs of polypropylene filled with aluminium and magnesium hydroxides. Part 1. A wide-angle X-ray diffraction study, Polymer 43/25 (2002) 6805-6811.

[12] M. Alonso, J.I. Velasco, J.A. de Saja, Constrained crystallization and activity of filler in surface modified talc polypropylene composites, European Polymer Journal 33/3 (1997) 255-262.

[13] F.W. Billmeyer, Textbook of Polymer Science, John Wiley and Sons, 1984.

[14] A.S. Luyt, J.A. Molefi, H. Krump, Thermal, mechanical and electrical properties of copper powder filled low-density and linear low-density polyethylene composites, Polymer Degradation and Stability 91/7 (2006) 1629-1636.

[15] J. Maity, C. Jacob, C.K. Das, S. Alam, R.P. Singh, Homocomposites of chopped fluorinated polyethylene fiber with low-density polyethylene matrix, Materials Science and Engineering A 479/1-2 (2008) 125-135.

[16] M. Kracalik, L. Pospisil, M. Slouf, J. Mikesova, A. Sikora, J. Simonik, I. Fortelny, Recycled poly(ethylene therepthalate) reinforced with basalt fibers: rheology, structure, and utility properties, Polymer Composites 29/4 (2008) 437-442.

[17] M.E. Tawfik, S.B. Eskander, Polymer concrete from marble wastes and recycled poly(ethylene therephthalate), Journal of Elastomers and Plastics 38/1 (2006) 65-79.