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MECHANICAL PROPERTIES OF NANO CALCIUM CARBONATE/POLYETHYLENE NANOCOMPOSITES

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ABSTRACT

This study is devoted to studying the mechanical properties of nanocomposites by introducing and increasing the proportion of nanocalcium carbonate (nano-$CaCO_3$) into polyethylene as an alternative to fillers. Polyethylene nanocomposites were obtained by incorporating industrially used 80 nm nanocalcium carbonate particles and laboratory-produced nanocalcium carbonate particles with particle sizes ranging from 100 nm to 900 nm into polyethylene. The size of nano-$CaCO_3$ particles was examined using SEM. The two types of nanocalcium carbonate particles used as fillers were introduced into polyethylene in proportions of 5%, 10%, and 15%, and the physical and mechanical properties of the samples were studied. When comparing the mechanical properties of the obtained samples with pure polyethylene, It was observed that the Young's modulus of samples obtained with industrially used calcium carbonate more than doubled with increasing nanocalcium carbonate content, the mechanical strength increased by up to 1.5 times, but the relative elongation decreased by up to 4 times. It was observed that the Young's modulus of the composites obtained in the presence of nanocalcium carbonate obtained under laboratory conditions increased by up to 2 times, the mechanical strength values remained almost unchanged, and the relative elongation decreased by almost 4 times. The mechanical properties were compared to see how the size of nanocalcium carbonate particles affected the mechanical properties of polyethylene, and it was found that the smaller the particle size, the more positive the mechanical properties.

Introduction

Calcium carbonate ($CaCO_3$) is one of the most abundant minerals in the earth's crust. It is available globally in a variety of particle sizes and purities, and can be used with or without organic coatings [1, 2]. The growing interest in polymer nanocomposites continues to drive the search for alternatives to the widely used clay-based nanofillers. One of these alternatives is calcium carbonate nanoparticles [2, 3].

Linear low-density polyethylene (PE) with high flexibility is widely used in the production of containers, pipes, cables, etc. [4, 5]. However, the application of PE is limited by its major disadvantages such as lower hardness, poor heat resistance, and low softening point. Therefore, initially, fillers were used to reduce the cost of polymers, but as the cost of polymers and the demands of modern products increase, more attention is being paid to increasing functionality [6–8]. Currently, "functional fillers" are most widely used in the polymer industry. The main condition for functional fillers is complete dispersion (disintegration of agglomerates into primary particles) and uniform spatial distribution in the polymer matrix, since agglomerates trap air and serve as a site for the onset of degradation, which leads to premature failure of the material [9, 10]. The introduction of fillers into the polymer composition primarily depends on the size of the filler being introduced, which has a significant impact on the thermal mechanical properties of the composite [11, 12]. So far, little attention has been paid to the influence of the chemical composition, composition, and stability of nano-$CaCO_3$ on the overall mechanical and thermal stability of PE. As a type of inorganic nanometer particles, nano-$CaCO_3$ has the advantages of fewer surface defects, more undivided atoms and large surface area, which has a great potential to combine physically or chemically with PE, and can affect the thermal decomposition of polyethylene due to the single particle effect.

In this study, the physical and mechanical properties of PE nanocomposites filled with nano-calcium carbonate (nano-$CaCO_3$) particles of two different sizes were investigated. The size of nano-$CaCO_3$ particles was examined using a scanning electron microscope (SEM). The polyethylene contains 5% to 15% nano-calcium carbonate. Samples of calcium carbonate-reinforced polyethylene composites with two different particle sizes and pure polyethylene were compared. Two different sizes of nano-$CaCO_3$ particles are discussed in terms of differences in mechanical strength, deformation behavior, and modulus of the incorporated material.

Computational Study

Materials

Low density linear PE (F-0320) with a melt flow index (MFI) = (2.16 kg, 210 ºC) of 3 g/10 min was provided by Uz-Kor Gas Chemical LLC JV. Nanocalcium carbonate (nano-$CaCO_3$) was purchased from Turkey with a size of 80 nm, which is commercially available. Nanocalcium carbonate (nano-$CaCO_3$) was obtained in the laboratory with a size of 100-900 nm. Formation of nanocomposites.

Nano-calcium carbonate synthesis.

Nano-$CaCO_3$ was synthesized by in situ precipitation. Calcium chloride (0.1 M, 100 mL) and sodium carbonate (0.1 M, 100 mL) solutions were prepared in distilled water. Citric acid (15 mol% relative to Ca^{2+}) was added to the $CaCl_2$ solution as a stabilizer. The Na_2CO_3 solution was added dropwise (2 mL/min) to the $CaCl_2$/citric acid solution under constant stirring (700 rpm), while maintaining pH 9.5 using NaOH. After complete addition, the suspension was stirred for 1 h and aged at room temperature for 12–24 h. The precipitate was collected by centrifugation (5000 rpm, 10 min), washed twice with distilled water and once with ethanol, and dried at 60 °C for 12–18 h.

Preparation of Nano-$CaCO_3$ /PE nanocomposites

The components were melted in a Brabender Plastograph (Germany). After first melting the PE in a plastograph at 180 ºC, nano-$CaCO_3$ was added and mixed for 8 min to ensure better mixing of the components. Then, the samples were injected at 180 ºC for mechanical studies in a Mercator 1971 (Poland) injection molding machine. Nanocomposites were obtained by adding two types of nano-$CaCO_3$ particles to the PE composition in the proportions of 5%, 10% and 15%.

Scanning Electron Microscope (SEM)

Morphological studies of the sample surface were carried out using a JSM-IT 210 scanning electron microscope (Jeol, Japan). During the measurements, an accelerating voltage (EHT - Extra High Tension) from 5 kV to 15 kV was used, the working distance (WD-working distance) was 10.8 mm. Images at different scales were obtained using the InTouch Scope software.

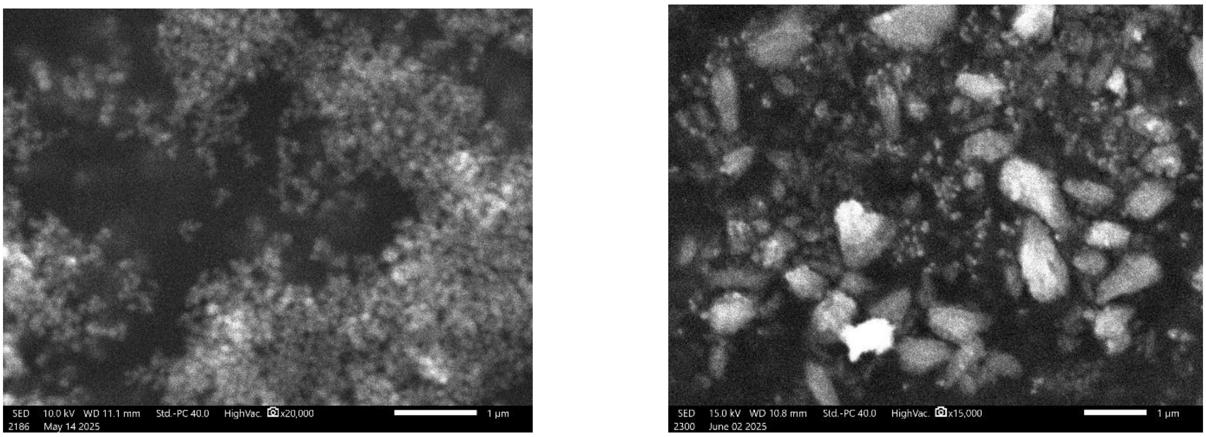
Physical-Mechanical Analysis

Tensile tests were conducted on a Shimadzu AG-X PLUS (Japan) in accordance with the international standard ASTMD 638. Tests were conducted at a speed of 1 mm/min up to 0.3% deformation to measure the elongation modulus (E), and then at a speed of 20 mm/min to measure the yield stress ($\boldsymbol{\sigma}$) and deformation ($\boldsymbol{\varepsilon}$).

Results and Discussion

Scanning electron microscope (SEM) analysis

As you know, Scanning Electron Microscopy (SEM) produces images of a sample by scanning the surface with an electron microscope beam. Through these images, we can obtain valuable information about the sample and sample particles.



a b

Figure 1. *SEM images of nano-$CaCO_3$ particles obtained in industry (a) and laboratory (b) at the same magnification*

Particle sizes were evaluated in SEM images. According to the results, the industrially used particle size is 80 nm, but it was also found that there were micro-sized particles that did not occupy even one twentieth of the entire image surface. Nano-$CaCO_3$ particles obtained in laboratory conditions vary in size, ranging from 100 nm to 900 nm. The size and polymorphic form of nano-$CaCO_3$ particles are important, and all these applications require the desired polymorphic forms and sizes. Calcium carbonate can crystallize in three anhydrous polymorphic forms. Calcite, (trigonal crystal system) rhombohedral, Aragonite, (orthorhombic) needle or rod-shaped, and Vaterite (hexagonal) concentric or flower-like spherical or ellipsoid [13-15].

It is appropriate to conduct analyses based on the results presented below to introduce calcium carbonate as a filler into polyethylene and obtain nanocomposites, and to evaluate the effect of particle size on mechanical properties. The mechanical properties results presented in Table 1 are composites obtained with the participation of industrially obtained nano-CaCO3 particles, with PE content of 5%, 10% and 15%, and the results of changes in mechanical properties were obtained.

Table 1

The effect of the proportion of calcium carbonate in polyethylene on the mechanical properties

№	PE(F-0320)/$CaCO_3$	σ [MPa]	ε [%]	E [MPa]
1.	PE	11.5±0.1	596.1±12.4	96.2±1.7
2.	95/5	13.4±0.2	605.2±26.2	165±6.6
3.	90/10	15.4±0.3	400.3±55	169±4.3
4.	85/15	17±0.4	155.7±8.5	215.4±8.8

As can be seen from the table, significant changes were observed in the mechanical properties of polyethylene with an increase in the calcium carbonate content. Initially, the yield stress (σ) increased from 11.5 MPa to 13.4 MPa with the addition of 5% $CaCO_3$, indicating an increase in strength. When the $CaCO_3$ content reached 10%, this stress increased to 15.4 MPa, but the elongation at break (ε) decreased significantly. The addition of 15% $CaCO_3$ reached the maximum strength (17 MPa), but the elongation at break decreased to 155.7% due to a significant decrease in deformation. The Young's modulus also increased to 215.4 MPa, indicating a stiff and strong composite.

The results show that the addition of calcium carbonate significantly changes the mechanical properties of polyethylene. Samples with 5% and 10% $CaCO_3$ addition maintained the optimal balance between strength and flexibility. However, when the $CaCO_3$ content was increased to 15%, although the strength increased, a decrease in elasticity was observed. These results are consistent with previous studies, for example, studies on high-density polyethylene and calcium carbonate-based composites also showed an increase in Young's modulus and a decrease in elasticity with increasing $CaCO_3$ content [3]. The results showed that the addition of $CaCO_3$ increases the hardness and strength of the material, but reduces the elasticity. Samples with 5-10% $CaCO_3$ addition showed optimal results, which suggests that this may be the most suitable range for practical applications.

Table 2

The effect of the proportion of calcium carbonate in polyethylene on the mechanical properties

№	PE(F-0320)/$CaCO_3$	σ [MPa]	ε [%]	E [MPa]
1.	PE	11.5±0.1	596.1±12.4	96.2±1.7
2.	95/5	10.9±0.4	362.7±36.6	137.8±3.1
3.	90/10	12.4±0.4	332.4±33.3	157.2±3.2
4.	85/15	10.8±0.3	167.3±17.5	188.4±4.9

As can be seen from Table 2, the addition of nano calcium carbonate obtained under laboratory conditions and with a size range of 100–900 nm resulted in changes in the mechanical properties of polyethylene as shown in Table 1. Initially, the yield stress (σ) decreased slightly from 11.5 MPa to 10.9 MPa when 5% $CaCO_3$ was added. This small decrease indicates that the nanoparticles were not fully dispersed in the polymer matrix due to the large particle size. At 10% $CaCO_3$, the strength increased to 12.4 MPa. However, when the $CaCO_3$ content reached 15%, the strength decreased again and was 10.8 MPa, indicating that the particles were not evenly distributed in the matrix due to the formation of particle agglomerations at high concentrations. The elongation at break (ε) decreased significantly in all samples. While for the original polyethylene, this indicator was 596.1%, it decreased to 362.7% when adding 5% $CaCO_3$, and to 167.3% at 15% loading. These changes indicate a decrease in the elasticity and flexibility of the composites. In particular, a significant decrease in plasticity was observed with increasing particle content, which indicates that the material became brittle. At the same time, the Young's modulus (E) always increased. While the initial value was 96.2 MPa, it increased to 137.8 MPa with the addition of 5% $CaCO_3$, to 157.2 MPa with 10%, and to 188.4 MPa with 15%. This confirms the role of particles in increasing the hardness. Although the particles increased stiffness in the polymer matrix, they did so at the expense of elasticity and stretchability.

The results obtained show that the addition of nanoparticle $CaCO_3$ in the size range of 100–900 nm increases the hardness and strength properties of PE-based nanocomposites to a certain extent, but these changes occur at the expense of plasticity. Particles of this size are not able to transmit stress effectively due to their dispersion properties, relatively small surface area, and tendency to agglomerate. Therefore, these composites are suitable for applications where strength is required but low elasticity is required.

Conclusions

Analysis of the two tables above shows that the mechanical properties of polyethylene-based nanocomposites are directly dependent on the amount and size of $CaCO_3$ particles added to the composition. Nanoparticles with a size of approximately 80 nm used in industrial conditions interact well with the polyethylene matrix, significantly increasing the strength (σ) and hardness (E) indicators, while almost maintaining elasticity (ε). In particular, optimal composites were obtained by adding 5–10% $CaCO_3$, providing a balance of "strength-flexibility". Under the influence of nanoparticles with a size of 100–900 nm, obtained in laboratory conditions, the composites had relatively low strength, and elasticity was sharply reduced. Although Young's modulus increased in all cases, these indicators led to embrittlement of the composite. This is explained by the uneven distribution of larger particles in the polymer matrix, agglomeration, and inability to transmit stress efficiently enough. In general, particle size and quantity play a crucial role in controlling the mechanical properties of nanocomposites. From a practical point of view, adding 5–10% of 80 nm $CaCO_3$ can produce polyethylene composites with high strength and sufficient elasticity. This allows them to be effectively used in various packaging materials, film products, or other industrial sectors that require strong but flexible elements.

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