

PROPRIETĂȚILE MECANICE ȘI MORFOLOGICE ALE COMPOZITELOR PE BAZĂ DE HDPE RANFORSAȚE CU NANOPULBERI DE SiO₂, ZnO, Mg(OH)₂ ȘI CaCO₃

THE MECHANICAL AND MORPHOLOGICAL PROPERTIES OF HDPE COMPOSITES FILLED WITH SiO₂, ZnO, Mg(OH)₂ and CaCO₃ NANO POWDER

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Thermoplastic resin-mineral filler composites have been widely used in molded products due to effective cost reduction. Generally, the addition of mineral fillers will have an embrittling effect on polymers although the rigidity of the polymers can be increased. Much effort has been devoted to improving the properties of polymers by the addition of inorganic fillers, such as SiO₂, ZnO, CaCO₃ and Mg(OH)₂.

In this study, mechanical properties of the high density polyethylene (HDPE) composites filled with silicon dioxide (SiO₂), zinc oxide (ZnO) magnesium hydroxide [Mg(OH)₂] and calcium carbonate (CaCO₃) were investigated. The structure and properties of the composites are characterized using a scanning electron microscopy (SEM) and Energy dispersive X-ray spectroscopy (EDS). Furthermore, HDPE composites were subjected to examinations to obtain their tensile strengths, yield strengths, elasticity modulus, % elongation, Izod impact strength, hardness and melt flow index (MFI).

Compozitele de tip polimer termoplastic-umplură minerală sunt utilizate pe scară largă pentru obținerea de produse turnate, datorită costului de producție scăzut. În general, adăugarea de umpluturi minerale va conduce la creșterea fragilității polimerilor deși rigiditatea acestora poate fi îmbunătățită. Un efort considerabil a fost acordat îmbunătățirii proprietăților polimerilor prin adăugarea de materiale de umplură anorganice, cum ar fi SiO₂, ZnO, CaCO₃ și Mg(OH)₂.

În acest studiu au fost investigate proprietățile mecanice ale materialelor compozite pe bază de polietilenă cu densitate mare (HDPE) ranforsată cu dioxid de siliciu (SiO₂), oxid de zinc (ZnO), hidroxid de magneziu [Mg(OH)₂] și carbonat de calciu (CaCO₃). Structura și proprietățile compozitelor sunt caracterizate cu ajutorul microscopiei electronice de baleiaj (SEM) și a spectroscopiei de raze X dispersive în energie (EDS). De asemenea, materialele compozite au fost analizate din punct de vedere al rezistenței la tracțiune și al limitei de elasticitate, al modulului de elasticitate, alungirii la rupere, a rezistenței la impact Izod, a durtății și al indicelui de curgere (IFM).

Keywords: silicon dioxide, zinc oxide, magnesium hydroxide, calcium carbonate

1. Introduction

Inexpensive inorganic substances are widely used as fillers to improve mechanical and thermal properties of polymers in the plastic industry. These mainly include fillers, such as calcium carbonate (CaCO₃), mica, wollastonite, glass fiber, glass beads, jute, silica (SiO₂), etc [1-3]. In recent years micro- and nano-fillers have attracted great interest, both in industry and in academia, because they often exhibit remarkable improvement in materials properties when compared to conventional composites made by using macro-fillers. These micro-/nano-composites can be made with very low loading of micro-/nano-fillers as compared with macro particle sized fillers [4]. High density polyethylene (HDPE) is widely used as a commodity polymer with high-tonnage production due to its distinctive mechanical and physical properties. Because of its low toughness, weather resistance, and environmental stress cracking resistance as compared to engineering polymers,

its application in many areas has been limited. To improve these disadvantages, HDPE has been reinforced with fillers [5-7]. Polyolefin composites based on silica have aroused great interest among researchers [8-9].

Silica can be more effective in improving the mechanical strength and abrasion resistance of polypropylene (PP) and high density polyethylene (HDPE) than do ordinary fillers because of its small size and the surface-interface effect [10-17]. ZnO as functional inorganic filler has been widely used in functional devices, catalysts, pigments, optical materials, cosmetics, and ultraviolet (UV) absorbers [18-21]. In addition, after Sawai and coworkers [22-23] found that ZnO powder had antibacterial activity against some bacteria strains in 1995; more and more researchers have embarked on studies of ZnO as an antibacterial agent. Therefore, the introduction of ZnO into polymers can not only enhance the mechanical properties of the polymers because of a strong interfacial interaction between polymers and

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nanoparticles but also endow polymers with some other functional capabilities, such as photo stabilization [24-25] and antibacterial activity [24]. Work has been reported on many aspects of Mg(OH)₂ filled polymer composites, including its flame retardancy, mechanical properties, preparation and processing, crystallization and morphology, thermal oxidative degradation, rheology, interface and microstructure, and functional modification, but there are a few reports on the effects of particle size. Zhang et al. reported that effect of particle size on the properties of Mg(OH)₂ filled rubber composites [25]. Mishra et al. investigated the effect of nano- Mg(OH)₂ on the mechanical and flame retarding properties of PP composites [28].

Calcium carbonate is the second most widely used mineral filler after talc. The use of CaCO₃ with a suitable hardness (Mohr hardness ~3) does not cause short-term failure in processing machinery [29]. The adhesion between the filler and the matrix is very important for polymer composites. If the adhesion is strong in the interface, the modulus, rigidity, strength, and toughness of a particulate composite will be increased. Many mineral fillers and CaCO₃ are based on inorganic materials that do not exhibit good adhesion to thermoplastic matrices. Therefore, the surface modification of filler particles with suitable coupling agents is often recommended to enhance filler particle dispersion into polymer matrices to improve strong bonding with the matrix and molding [30]. HDPE filled with mineral particles also improves dimensional stability, opacity, and barrier properties. CaCO₃ is the largest volume mineral used in the polymer industry because of its low cost and abundance. It is available globally in a variety of particle shapes, purities, and sizes (micro and nano). However, because of its higher polar nature and higher surface areas, CaCO₃ is difficult to disperse and stabilize in a polymer matrix [31-34].

The effect of addition of nanosized calcium carbonate with polyethylene on tensile properties, viscosity, and dimensional stability has been investigated by some researchers. Lazzeria et al.[35] fabricated PE nanocomposite with 70% improved Young's modulus compared to virgin PE. The creep behavior of PE nanocomposites reinforced with different nanosized calcium carbonate depends strongly on the calcium carbonate content. One study showed that the best creep resistance of PE nanocomposites can be achieved at 10% of calcium carbonate incorporation in the composite [36]. Zebarjad et al. reported that nanosized calcium carbonate has a significant effect on crystallinity, melting point, and heat of melting of HDPE [37].

In this article, 5, 10, 15 and 20 wt % of SiO₂, ZnO, Mg(OH)₂, CaCO₃ powder, as fillers and HDPE as polymer matrix, were used to prepare the polymer composites. The effect of all particles on the mechanical and morphological properties of HDPE was investigated.

2. Experimental

2.1. Compositions and Materials

Seventeen different polymer composites (given in Table 1) were prepared by using the following materials: HDPE, known as Petilen YY (I 668 UV), was obtained from Petkim A.Ş., Turkey. Silicon dioxide (SiO₂), known as CAB-O-SIL TS-610, was obtained from CABOT, Billerica, MA-USA. Its specific gravity was 2.2 g/cm³ and average particle length of 0.2-0.3 micron. Zinc oxide powder (MKN-ZnO-050) was supplied by MKnano (Ontario-Canada). Particle size (TEM) was ≤50 nm and its purity was 99.9 %. Magnesium hydroxide powder (MKN-Mg(OH)₂-050) was supplied by MKnano (Ontario - Canada). Its purity was 99 % and particle size of 50 nm. Calcium carbonate (calcite- CaCO₃) nanopowder

Table 1

Composition of the different polymer composite formulations
Compoziția procentuală a compozitelor pe bază de polimer

Groups Cod	HDPE (wt %)(% grav.)	SiO ₂ (wt %)(% grav.)	ZnO (wt %)(% grav.)	Mg(OH) ₂ (wt %)(% grav.)	CaCO ₃ (wt %)(% grav.)
1	100	-	-	-	-
2	95	5	-	-	-
3	90	10	-	-	-
4	85	15	-	-	-
5	80	20	-	-	-
6	95	-	5	-	-
7	90	-	10	-	-
8	85	-	15	-	-
9	80	-	20	-	-
10	95	-	-	5	-
11	90	-	-	10	-
12	85	-	-	15	-
13	80	-	-	20	-
14	95	-	-	-	5
15	90	-	-	-	10
16	85	-	-	-	15
17	80	-	-	-	20

(Calprec PR) was supplied by Cales de Llierca (Girona-Spain). Its purity was 98 % and particle diameter of 50 nm. SiO₂, ZnO, Mg(OH)₂ and CaCO₃ were dried in a VacuCell VD 55 vacuum oven (MMM Medcenter Einrichtungen GmbH, München- Germany) at 105 °C for 24 hours before being blended with HDPE. Mechanical premixing of solid compositions was done using a LB-5601 liquid-solids blender (The Patterson-Kelley Co., Inc. east Stroudsburg, PA - USA) brand batch blender for 15 min. Samples with various proportions of HDPE/SiO₂, HDPE/ZnO, HDPE/Mg(OH)₂ and HDPE/CaCO₃ polymer composites (presented in Table 1) were produced between 180-220 °C at 20-30 bar pressure, and a rotation rate of 30 rpm, with a Microsan co-rotating twin-screw extruder (Microsan Instrument Inc. Kocaeli - Turkey). L/D ratio is 30, Ø: 25 mm. To prepare the samples for mechanical test, the following injection conditions were used: injection temperature was 180-220 °C, injection pressure was 110-130 bar, dwelling time in mold was 10 s, and screw rotation was 25 rpm, Polymer composites were also dried in vacuum oven at 105 °C for 4 hours after extrusion.

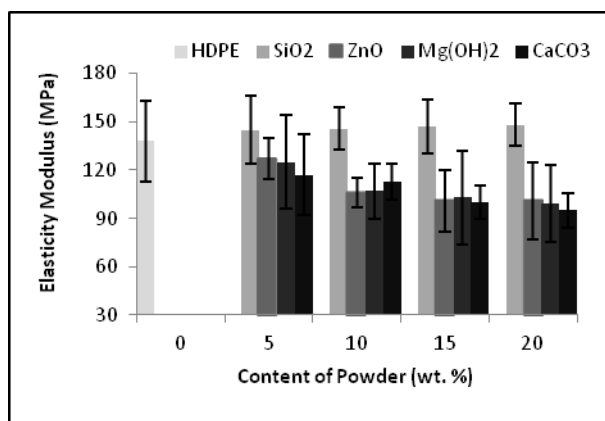
2.2. Test procedure

Composite specimens were conditioned at 23°C and 50% humidity for 24 h before testing

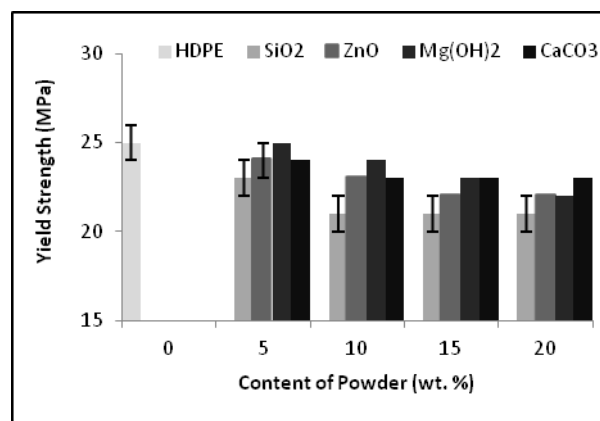
(ASTM D618). Tensile tests were prepared according to the ASTM D638 standards by using a Zwick Z010 (Ulm-Germany) testing machine with a load cell capacity of 10 kN at a cross-head speed of 50 mm/min. The hardness test was done according to the ASTM D2240 method with Zwick hardness measurement equipment. To investigate fracture behavior, Izod impact test (notched) was done at room temperature according to the ASTM D256 method with Zwick B5113 impact test device (Ulm, Germany). The fractured surfaces of the composites were coated to an approximate thickness of 10 nm of a gold (Au) (80%)/palladium (Pd) (20%) alloys to prevent electrical charging by Polaron SC 7620 (Bad Schwalbach-Germany). The surfaces of the prepared samples were observed by the JEOL-JSM 5910 LV (Tokyo, Japan) scanning electron microscopy (SEM) at an acceleration voltage of 20 kV and magnification is 5000. Elemental analysis was done using Energy dispersive X-ray spectroscopy (EDS) (Oxford Instruments, England). Seven samples were tested in each set and the average value was reported.

3. Results and discussion

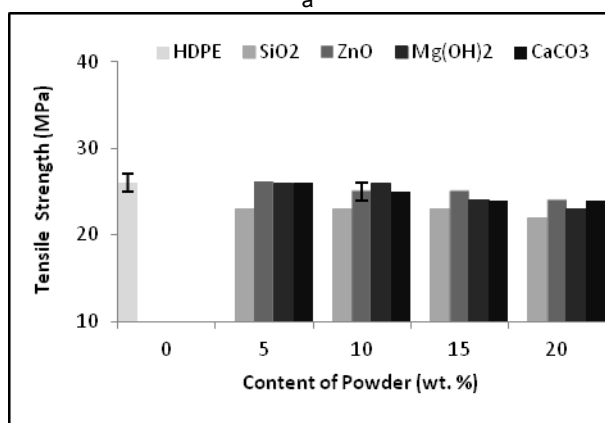
Mechanical properties of the HDPE/SiO₂, HDPE/ZnO, HDPE/Mg(OH)₂ and HDPE/CaCO₃ polymer composites are given in Figure 1.



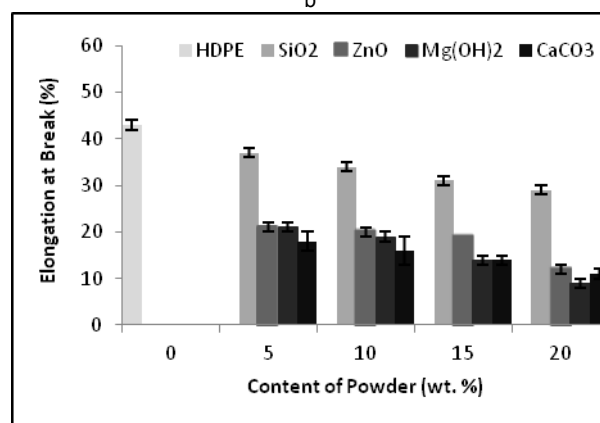
a



b



c



d

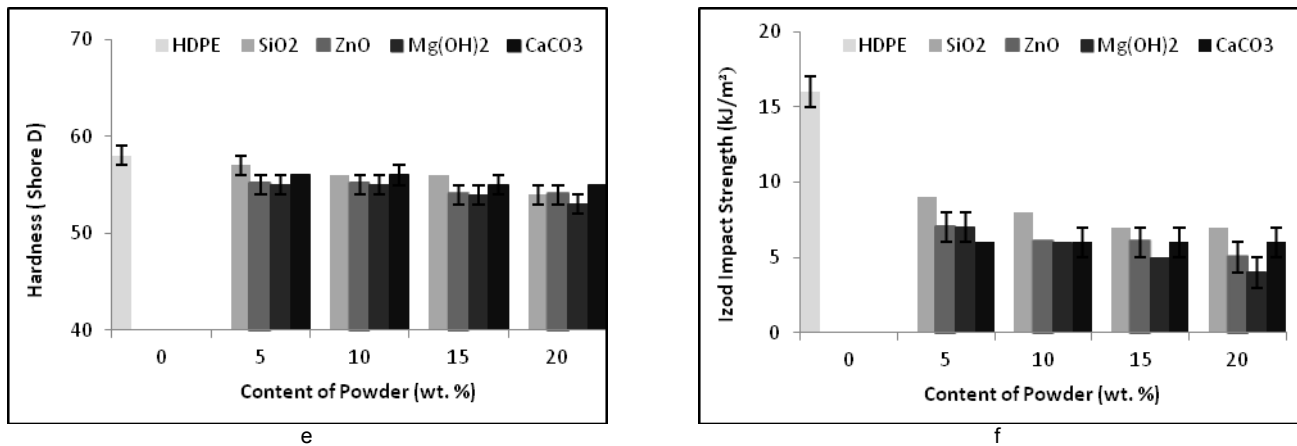


Fig. 1 - Mechanical properties of the HDPE/SiO₂, ZnO, Mg(OH)₂ and CaCO₃ polymer composites: a. Elasticity modulus; b. Yield Strength; c. Tensile Strength; d. Elongation at Break; e. Hardness; f. Izod Impact Strength / *Proprietățile mecanice ale compozitelor polimerice the HDPE/SiO₂, ZnO, Mg(OH)₂ și CaCO₃: a. Modulul de elasticitate; b. Limita de curgere; c. Rezistența la încovoiere; d. Alungirea la rupere; e. Duritatea; f. Rezistența la impact Izod.*

Elasticity modulus is the stiffness (the ratio between stress and strain) of a material at the elastic stage of a tensile. The effects of particle loading on the elastic modulus of HDPE/SiO₂, HDPE/ZnO, HDPE/Mg(OH)₂ and HDPE/CaCO₃ composites are given in Figure 1-A. It is seen that the modulus is affected by particle loading in the range studied. The elasticity modulus of HDPE/SiO₂ composites increased as the filler concentration increases from 0 to 20 wt %. But, the elasticity modulus of HDPE/ZnO, HDPE/Mg(OH)₂ and HDPE/CaCO₃ composites decreased as the filler concentration increases from 0 to 20 wt%. This decrease was attributed to a possible agglomeration of the particles. The minimum elasticity modulus is observed at the 20 wt% CaCO₃ concentration for HDPE. The effect of particle loading on the yield strength and tensile strength of HDPE/SiO₂, HDPE/ZnO, HDPE/Mg(OH)₂ and HDPE/CaCO₃ composites are shown in Fig. 1-b and Fig. 1-c respectively. With increased loading, the yield strength of composites filled with SiO₂, ZnO, Mg(OH)₂ and CaCO₃ are decreased. This decrease was due to the poor adhesion between untreated filler and matrix. On the other hand with increased loading, tensile strength of composites filled with Mg(OH)₂ and CaCO₃ are decreased but filled with SiO₂ and ZnO did not significant change. The elongation at break of SiO₂, ZnO, Mg(OH)₂ and CaCO₃ filled composites was measured, as shown in Figure 1-d. With increased loading, the elongation at break of all composites filled with SiO₂, ZnO, Mg(OH)₂ and CaCO₃ are decreased for all. The minimum elongation at break is observed at the 20 wt % Mg (OH)₂ concentration for HDPE. In comparison with the elongation at break of virgin HDPE, the elongation at break decreased by 79 % for the composites with a 20 wt % Mg (OH)₂ concentration. The relationship between the filler content and the hardness of the polymer composites is shown in Figure 1-e. The hardness

just a little decreased as the particle concentration increased from 0 to 20 wt%. This was due to the low hardness of used particles. Figure 1-f illustrates the effect of filler on the Izod impact strength (notched) of HDPE composites. The impact strength decreased as the particle concentration increased from 0 to 20 wt %. In comparison with the Izod impact strength of virgin HDPE, the Izod impact strength decreased by 75 % for the composites with a 20 wt % Mg (OH)₂ concentration. It is possible that particles agglomerated easily into large particles, which could be sites for crack propagation, and acted as a micro-crack initiator. However, at a larger filler concentration, the value of the impact strength was not much reduced.

Melt flow index (MFI) values of the HDPE/SiO₂, HDPE/ZnO, HDPE/Mg(OH)₂ and HDPE/CaCO₃ polymer composites are given in Figure 2.

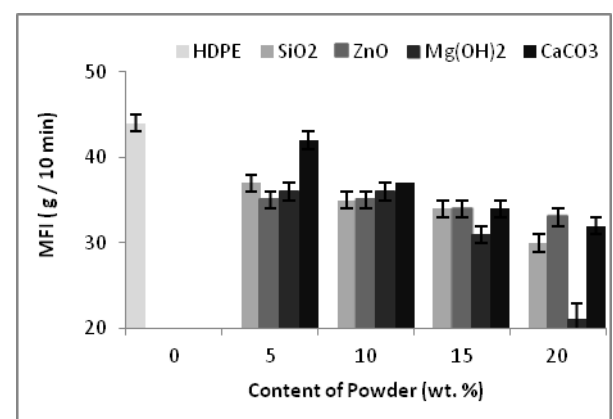


Fig. 2 - MFI values of the HDPE polymer composites / *Valorile MFI ale compozitelor de bază de HDPE.*

The relationship between the wt % of the filler and melt flow index of HDPE composites is shown in Figure 2. Melt flow index of HDPE composites shows a decreased as the all fillers

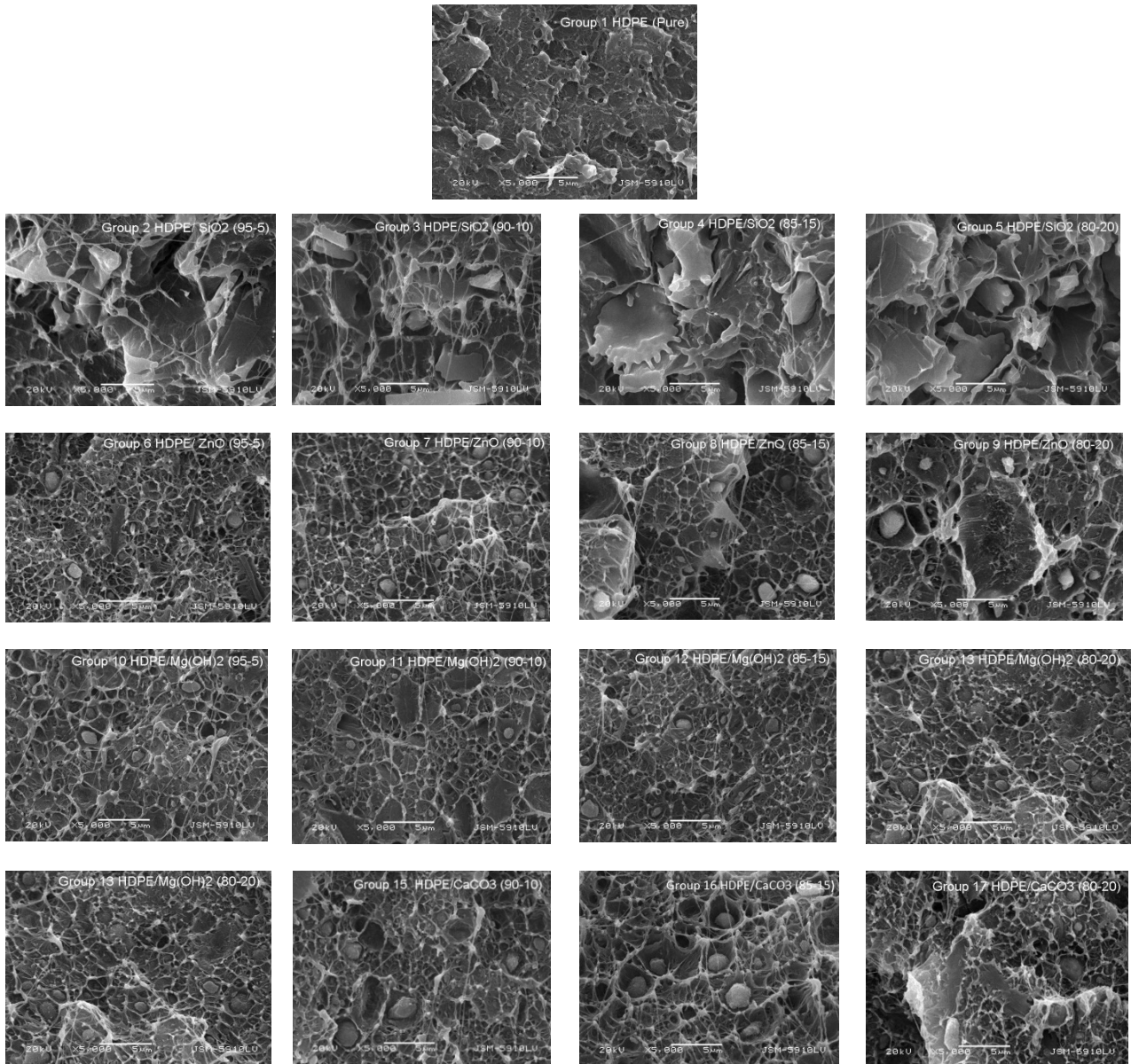


Fig 3 - SEM micrographs of the HDPE polymer composites / *Micrografii SEM ale compozitelor pe bază de HDPE.*

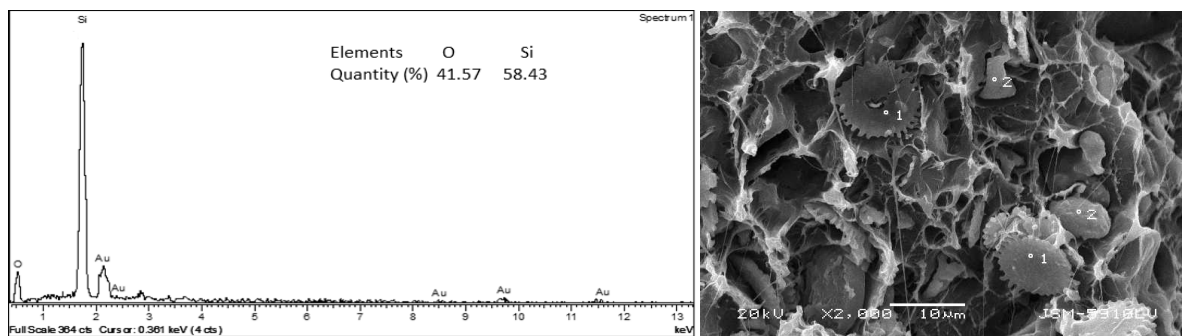


Fig. 4 - Energy dispersive X-ray spectroscopy (EDS) spectra of the HDPE polymer composite. *Spectrele de spectroscopie de raze X dispersive în energie (EDS) ale compozitelor pe bază de HDPE.*

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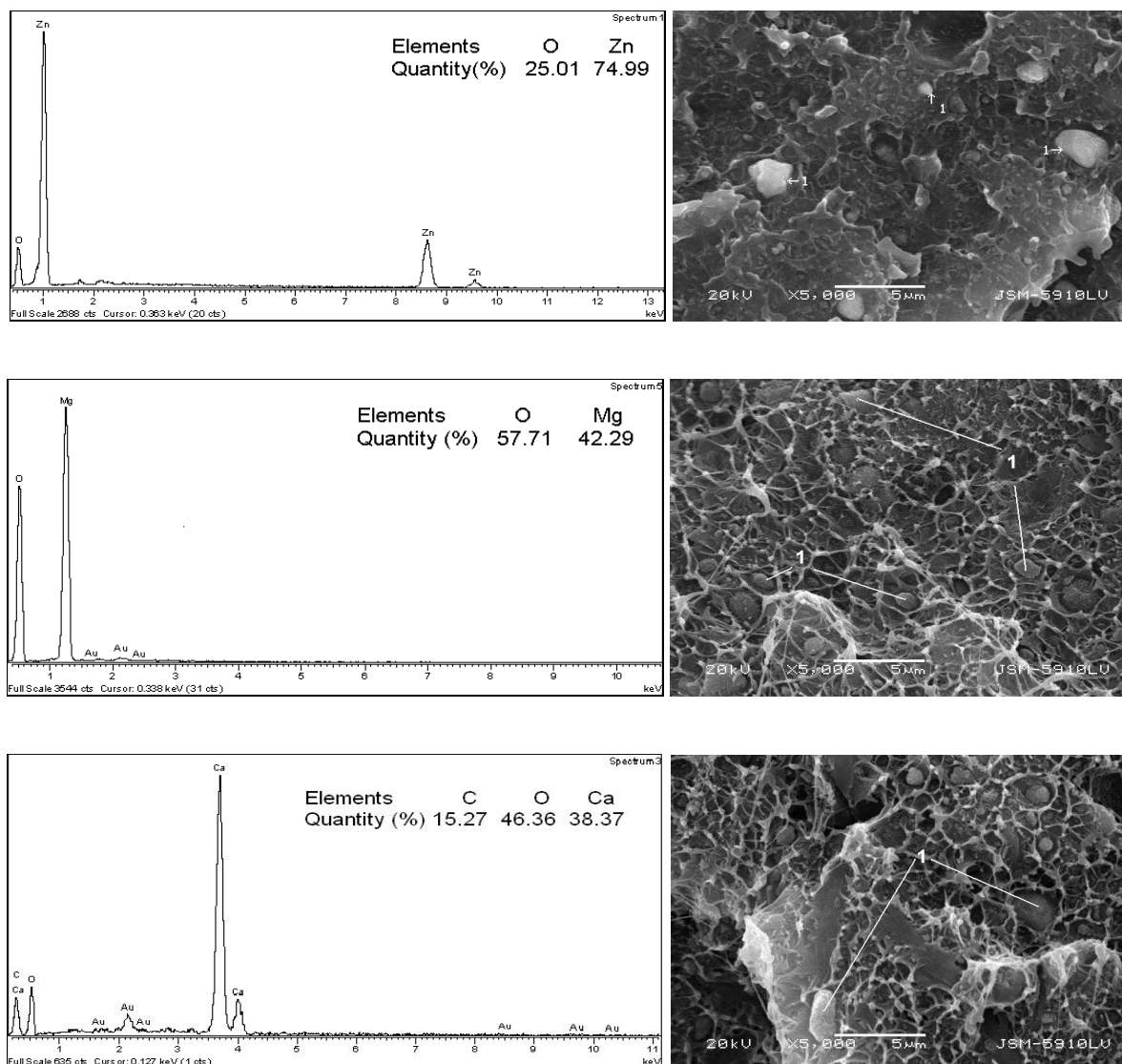


Fig. 4

concentration increases from 0 to 20 wt %. In comparison with the melt flow index of virgin HDPE, the melt flow index decreased by 52% for the composites with a 20 wt % Mg(OH)₂ filler concentration. This decrease was attributed to a possible agglomeration of the particles. Probably agglomerated particles locked the flow. As a result, composites with higher filler contents showed lower MFI values.

The micrographs taken from the fracture surface of HDPE, HDPE/SiO₂, HDPE/ZnO, HDPE/Mg(OH)₂ and HDPE/CaCO₃ composite samples are shown in Figure 3 and energy dispersive X-ray spectroscopy spectrums of the polymer composite are shown in Figure 4. The boundaries and the contrast can be obviously seen between the two phases of SiO₂, ZnO, Mg(OH)₂ and CaCO₃ particles and HDPE matrix.

4. Conclusion

The effects of SiO₂, ZnO, Mg(OH)₂ and CaCO₃ on the mechanical properties, such as the

yield and tensile strengths, elastic modulus, hardness, Izod impact strength and MFI, of HDPE composites were investigated. Mechanical and morphological investigations have demonstrated that the introduction of SiO₂, ZnO, Mg(OH)₂ and CaCO₃ in HDPE leads to marked modifications of physical and mechanical properties of the material.

The following results were obtained:

1. With increased loading, elasticity modulus, % elongation, and hardness of composites filled with SiO₂, ZnO, Mg(OH)₂ and CaCO₃ are decreased. This was due to agglomeration of particles at higher percentage and weak adhesion between fillers and the HDPE matrix. Also, it is clear that particle loading has an important effect on the composite toughness, which can be improved or reduced with changing particle loading.

2. With increased loading Izod impact strength of composites filled with SiO₂, ZnO, Mg(OH)₂ and CaCO₃ are decreased. Fracture propagation was more pronounced with the

addition of the particles. It is possible that all type of fillers agglomerated easily into large particles, which could be sites for crack propagation, and acted as a micro-crack initiator.

3. MFI values of composites decreased with increasing particle loading. Higher filler content showed lower MFI value.

4. The micrographs indicate that the SiO₂, ZnO, Mg(OH)₂ and CaCO₃ particulates are homogeneously dispersed on the fractured surfaces of polymer matrix. But the bond between the polymer and particles is weak and, addition of particles resulted in porous structure. The SEM micrograph also shows the agglomeration of SiO₂, ZnO, Mg(OH)₂ and CaCO₃ particles. It is known that this type of particles have a great tendency to form agglomerates, in fact, agglomeration is a well-known phenomenon, and its probability increases with decreasing particle size.

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3rd RILEM Int. Symposium of the TC-TDK Testing methods for determination of double-K criterion for crack propagation in concrete From 10 April 2014 to 13 April 2014, Hangzhou, China

The double-K criterion has been developed in the nineties of last century. The double-K constitute two material parameters ,the first is called Kc ini which defines the onset of cracking,andthe second is called Kc un which defines the onset of unstable cracking or failure. Both are derived from linear-elastic fracture mechanics principles. The two material parameters characterize the mechanical behaviour of concrete.It has been shown that the double-K does not depend on the size of the specimens. Many comparative tests have been performed with specimen sizes varying in a large range and no size effect could be found.

The double-K criterion has been used to assess the safety of large concrete structures like dams . The Chinese standard number DL/T5332-2005 defines testing methods for the determination of double-K. It has been introduced in the People's Republic of China in 2005. The testing of double-K can be done on every universal testing equipment ,even without displacement control. The aim of TC is to make the knowledge of double-K internationally available by preparing a RILEM recommendation.

This is the third symposium of RILEM TC-TDK Technical Committee. The first symposium was organized by professor Shilang Xu in Hangzhou ,China in April,2012 . The second symposium of RILEM TC-TDK Technical Committee in April, 2013 in Hangzhou, China, also organized by professor Shilang Xu.

In the first symposium, the round-robin testing for double-K criterion testing method was proposed. The specific issues concerned with the testing method for double-K criterion were further discussed in the second symposium, besides that, data processing and final results related to the round-robin test were also covered in the second symposium. The main purpose of the third symposium is to discuss the draft recommendation of testing methods for determination of double-K criterion.

Important dates: Submission Test results: December 20th 2013

Registration deadline: February 1st 2014
