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Original scientific paper

# The Use of Untreated Neuburg Siliceous Earth as Filler for High-Density Polyethylene

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**Abstract:** This paper reports the results of a study on twin-screw direct extrusion run at a screw speed of 120 rev/min to produce a composite pellet product which was used for an injection molding process and after that subjected to static tensile testing as well as hardness and specimen morphology examination. As filler, we used untreated Neuburg siliceous earth (NSE) marketed under trade names Silitin Z86 and Silitin V88 ranging from 10 to 60 wt% in a matrix made of high-density polyethylene (PE-HD) Hostalen GD 7255. The results demonstrate that Young's modulus and maximum tensile stress of the polyethylene composite with untreated Neuburg siliceous earth first decrease and then increase with increasing the filler's content. The addition of Silitin Z86 causes an increase in tensile stress at break while the addition of Silitin V88 leads to its decrease. Strain at maximum tensile stress of the tested molded pieces first increases with increasing the filler's content, but then starts to decrease. The tested composite molded pieces have lower strain at break as opposed to those without the filler. An increase in the filler's content leads to an increase in hardness of the tested specimens. The results of specimen morphology demonstrate that the filler is unevenly distributed in the polymer and reveals a strong tendency to agglomerate.

**Keywords:** mechanical properties; material testing; polymer composites; twin-screw direct extrusion

## 1 INTRODUCTION

Technological development led to a higher demand for new materials with improved strength properties. This, in turn, led to extensive research on modifying various polymers including polyolefines. From an economic point of view, modification of popular polymers is much more cost effective than development of new materials [1, 2, 3].

Composite polymers have more and more applications. Structural composites, which are the most widely used in the industry, must meet more and more stringent strength requirements. The popularity of composite materials is due to their functional properties which greatly depend on type of filler [4, 5]. The effect of filler on properties of a composite material, in turn, mainly depends on the filler's chemical composition and surface properties enabling formation of adhesive bonds between the polymer and the filler [6, 5]. To ensure that fillers which are introduced directly into polymers during processing have adequate polymer matrix adhesion, suitable proadhesive preparations are used [1, 4]. Fillers which have a significant impact on strength are known as active fillers [5], and they usually have good adhesion properties. Active fillers do not cause any significant changes in strength and have lower polymer matrix adhesion.

New constructional composite materials are more and more often produced by innovative processing methods including extrusion and reactive extrusion [5, 6, 7].

A particular group of fillers are powder fillers including Neuburg siliceous earth (NSE), a natural combination of kaolinite and silica. This filler is a mixture of cryptocristalline and amorphous silica (quartz) and lamellar kaolinite. The two totally different minerals form a loose structure. As a result of long-term deposition in crust of the earth, silica pellets have rounded but not sharp edges. Neuburg siliceous earth has been manufactured for over 100 years by Hoffmann Mineral (Neuburg, Germany). Their products include two product groups: Silitin and Aktisil [8, 9]. The first group includes a natural siliceous earth, the kinds of which differ with respect to particle size distribution and color neutrality. The other group includes siliceous earth treated with silanes, where alkoxy group hydrolysis leads to formation of a chemical bond on the surface of the filler. Siliceous earth is treated to improve functional properties of products containing the additives [10, 11].

The objective of this study is to determine and estimate a selection of mechanical properties and cross-sectional morphology of produced composite materials which have a high-density polyethylene matrix with different contents of Neuburg siliceous earth. Another goal of this work is to determine the suitability of untreated Neuburg siliceous earth as filler for polyethylene polymers.

## 2 EXPERIMENTAL

### 2.1 Test Stand

The experiments were performed on a twin-screw direct extruder ZSK 18 MEGAlab manufactured by Coperion Werner and Pfleiderer GmbH (Stuttgart, Germany) and mounted in the Institut für Produktionstechnik of Westsächsische Hochschule Zwickau Germany. The extruder has cylindrical screws with a diameter of 18mm and a length to diameter ratio of 24. The screws are double-coiled. The barrel is made of segments and consists of six segments, each with a length of 72 mm. The extruder's drive system is equipped with an asynchronous alternating-current engine with a power of 10 kW and a torque of 38 Nm as well as variable-speed transmission. The polymer is fed into the hopper via a screw feeder driven by an engine with a power of 250 W and a variable-speed drive ranging from 0 to 10 rev/min. The extruder is part of an extrusion line for cold pelletizing consisting of a water-cooling bath and an SP 30 Pure pelletizer manufactured by Pell-Tec Pelletizing Technology GmbH (Stuttgart, Germany).

In the tests we used a Krauss Maffei 50-55CX injection molding machine equipped with a twin-cavity mold for producing strength specimens in compliance with ISO 294-1:2002. The injection molding machine has a screw with a diameter of 18mm and a mold closing force of 500 000 N, while its maximum injection pressure is 250 MPa. The machine has a cooled feed zone, three heating zones in the plasticizing system, a heated nozzle and a thermostated injection mold.

Other devices used in the tests included a testing machine and a Shore hardness tester. Tensile strength tests for produced composite injection molds were performed using a standard testing machine from Zwick Roell (Ulm, Germany), Z010 AllroundLine. The Z010 testing machine can be operated at the maximum tensile force of up to 10kN and a tensile rate of up to 2000mm/min. Hardness was measured by the Shore method using a hardness tester from Heinrich Bareiss Prüfgerätebau GmbH (Oberdischingen, Germany). The hardness tester has a unique digital system for processing gauge head data into digital signal; it also has a lighted read-out of the Shore scale and automatic recognition of the connected gauge head with hardness scale selection.

The morphology of specimen cross sections was examined with a Nikon Eclipse LV100ND microscope, equipped with a DS-U3 camera and NIS-Elements AR 4.20.00 software.

## 2.2 Materials

The tests were conducted using high-density polyethylene with the trade name Hostalen GD 7255 manufactured by Orlen Polyolefines (Plock, Poland) and mainly used for injection molding purposes. This polymer exhibits high density, good fluidity, high rigidity and impact strength, and has a processing temperature ranging from 180 to 220 °C. The properties of the tested polymer are listed in Tab. 1.

As filler we used untreated Neuburg siliceous earth with the trade names Silitin Z86 and Silitin V88. Basic properties of these fillers are listed in Tab. 2. According to the data provided by the manufacturer, NSE is characterized by high ease and rate of mixing, minimal sedimentation, high surface activity, low compressibility, high abrasion resistance and extensive specific surface area.

**Table 1** Basic properties of the tested polymer

| Property | Value |
|---|---|
| Density, kg/m$^3$ | 955 |
| Melt flow rate (190 °C; 2.16 kg), g/10 min | 4 |
| Melt flow rate (230 °C; 5.0 kg), g/10 min | 11 |
| Tensile Modulus, MPa | 1180 |
| Tensile Stress at Yield, MPa | 27 |
| Tensile Strain at Yield, % | 8 |
| Charpy notched impact strength (23 °C, Type 1, Notch A, kJ/m$^2$) | 10 |
| Ball indentation hardness (H 132/30) MPa | 52 |
| Shore hardness (°Sh D) | 60 |
| Vicat softening temperature B50 (50 °C/h, 50 N), °C | 70 |

**Table 2** Selected properties of untreated NSE fillers

| Property | Neuburg siliceous earth | |
|---|---|---|
| | Silitin Z86 | Silitin V88 |
| Density, kg/m$^3$ | 2600 | |
| Electrical conductivity, 80 µS/cm | 80 | |
| Melting point, °C | 1600 | |
| pH Value | 6-8 | |
| Particle size, µm | 2-9 | 4-18 |
| Specific surface area, m$^2$/g | 11 | 8 |
| Oil absorption, g/100 g | 55 | 47 |
| Refractive index | 1.55 | |

Silitin has a very wide spectrum of application in hardenable materials processing, for instance in the production of rubber products, automotive parts, machinery components, cable coatings and electric devices. It can also be used for production of sports and industrial polymer floors, polymer casts, paint coatings, etc.

## 2.3 Investigated Parameters

Given the objective of the study, we devised a set of key parameters describing the investigated processes, i.e. twin-screw direct extrusion, injection molding and specimen tensile testing. The following parameters were investigated in the experimental tests:

- Young's modulus $E$, MPa,
- maximum tensile stress $\sigma_z$, MPa,
- tensile stress at break $\sigma_r$, MPa,
- strain at maximum stress $\varepsilon_z$, %,
- strain at break $\varepsilon_r$, %,
- hardness $H$, °ShD.

The variables included two filler types: Silitin Z86 and Silitin V88, their content in the composite being: $\zeta$ = 10; 20; 30; 40; 50 and 60 %.

As for constant factors, the screw rotational speed of the extruder, $v$, was set to 120 rev/min while the tangential speed of knife-edges of the pelletizer was 15 m/min. The processed material was high-density polyethylene (PE-HD) with the trade name Hostalen GD 7255. Other constant factors included geometrical elements of the plasticizing unit, extruder head, tooling and injection mold, as well as the water-cooling bath and pelletizer. In addition to this, the temperature of the injection mold was 19 °C; the temperature along the extruder's plasticizing unit was set to 130, 140, 150, 160, 170 and 180 °C, respectively; the polymer pressure in the extruder head was 8 bar, the temperature along the extruder's plasticizing unit was set to 180, 200 and 220 °C, respectively; the injection pressure was equal to 1050 bar; the holding pressure was 600 bar; the holding time was 6s; and the injection cycle time was 35 s, while the cooling time was set to 20 s.

The disturbing factors included variations in electric voltage, relative humidity and ambient temperature. It is estimated that the disturbing parameters had a negligible effect on the investigated process and can thus be omitted in the result discussion.

## 2.4 Experimental Details

Prior to starting the extruder, we prepared a mixture of high-density polyethylene and Neuburg siliceous earth in a scheduled ratio. The filler and the polymer were mixed mechanically. The polymeric composition was filled with 0.4 wt% of adhesion promoter in the form of carbofunctional silane with the trade name aminopropylotriethoxysilane [12], thereby modifying the surface of the inactive high-density polymer. Next, we ran extrusion with cold pelletizing. The extrusion process was continued until the plasticizing unit of the extruding machine was completely empty. Once the extruder's plasticizing unit was empty, another dose of the prepared mixture (with a different filler content) was fed into the hopper. After that, the produced polymer mixtures were subjected to injection molding in order to produce specimens for tensile testing.

Static tensile tests on a testing machine were conducted in compliance with ISO 527-1:1998 [13], while hardness measurements were performed according to the procedure described in ISO 868:2005 [14].

## 3 RESULTS AND DISCUSSION

### 3.1 Strength Tests of NSE Composites

Based on the results of strength tests, we created diagrams illustrating the dependence of Young's modulus, maximum tensile stress and stress at break as well as strain at maximum stress on the filler's content in produced injection molds. The above relationships are illustrated in Figs. 1 to 5.

With changing the content of untreated Neuburg siliceous earth in the test specimen, we first observe a decrease and then an increase in Young's modulus (Fig. 1). The addition of a small amount (10%) of the filler leads to decreasing the Young's modulus of the produced composite by 68 MPa for Silitin Z86 and by 77 MPa for Silitin V88,which corresponds to a decrease by 9.44% and 10.69% in the initial value. On increasing the NSE content in the tested composite by up to 60%, the Young‘s modulus increases by 15 MPa and 26 MPa compared to that of the unfilled polymer. This is probably caused by the fact that high-density polyethylene is a linear polymer with no branching, where partial spatial ordering of chains is possible [15]. This has a positive effect on Young's modulus because it enables better spatial ordering of chains and close proximity of macromolecules, which leads to formation of strong bonds between chains [16]. Lower values of Young's modulus are observed for bigger particles of Silitin V88, smaller specific surface area and, in effect, smaller difference in interfacial surface energy.

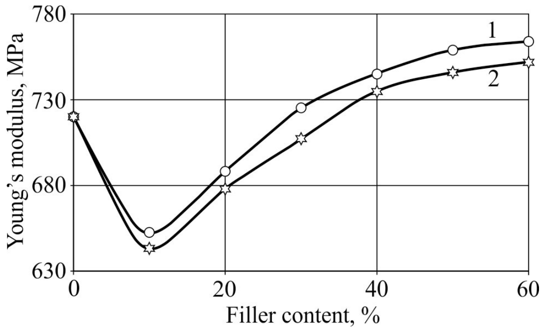

**Figure 1** Young's modulus versus filler content for NSE-filled polymer composites produced at rotational speed 120 rev/min: 1 – Silitin Z86, 2 – Silitin V88

Polyethylene composites with Neuburg siliceous earth Silitin Z86 and Silitin V88 exhibit a lower tensile strength compared to that of unfilled polymer (Fig. 2) in the entire range of variability in filler content. Irrespective of the NSE kind, the addition of even a small content of the filler (10%) leads to a decrease in tensile strength, ranging from 6.2% for Silitin Z86 to 10.0% for Silitin V88 compared to the initial value. One can observe that a further increase in the filler's content in the composite leads to an increase in tensile strength. A higher increase in the tensile strength of produced injection molds is observed for Silitin Z86. This is probably due to the size of pellets of this Neuburg siliceous earth. The pellet size of Silitin Z86 ranges from 2 to 9 μm, and it is almost two times smaller than that of Silitin V88, which plays a significant role on the interface of polymer matrix and the surface of filler pellets, as mentioned above. Mechanical properties of composites depend on the filler's capacity of integration into the polymer's microstructure [17]. The presence of smaller pellets also leads to higher strength of a composite. The results of a literature survey demonstrate that composite materials with higher mechanical properties can be produced by, among others, optimization of the amount, size, distribution and shape of filler pellets [18].

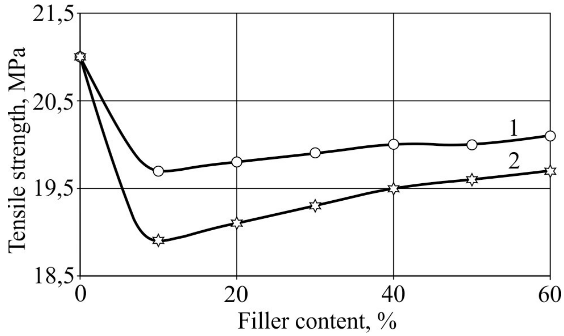

**Figure 2** Tensile strength versus filler content for produced NSE-filled polymer composites produced at rotational speed 120 rev/min: 1 – Silitin Z86, 2 – Silitin V88

The nature of changes in stress at break versus NSE content in the produced polymer composites is twofold (Fig. 3). The addition of 10% of both NSE types leads to a decrease in the stress at break in the tested material by about 2 MPa. A further increase in the content of Silitin Z86 in high-density polyethylene results in a further decrease and then in a sudden increase in stress at break amounting to over 6.6MPa. On the other hand, a further increase in the content of Silitin V88 causes a decrease in stress at break to a value of 6.1 MPa, which means it decreased by 46.96% compared to the initial value which was 11.5 MPa. This can be due to the loss of polymer matrix adhesion by small particles of Neuburg siliceous earth. Adhesion on the polymer-filler interface is a significant factor and often has a decisive effect on properties of composite materials. Polymer adhesion depends on the conditions of its adsorption on the surface of the filler, wettability of the filler's pellets by the melted polymer and the formation of bonds between the filler and the polymer [19].

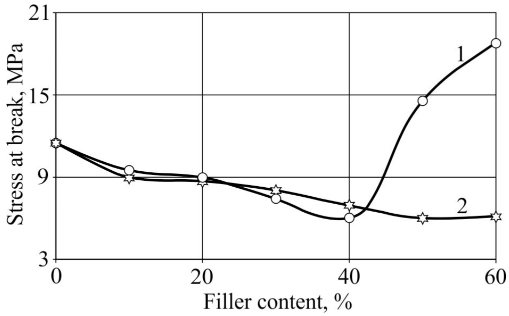

**Figure 3** Stress at break versus filler content for the NSE-filled polymer composites produced at rotational speed 120 rev/min: 1 – Silitin Z86, 2 – Silitin V88

On increasing the filler's content (whatever its type), strain at maximum tensile stress of the examined injection molds initially increases and then starts to decrease (Fig. 4). The highest strain of the produced injection molds can be observed for 10 wt% Silitin Z86 and Silitin V88; for both filler types it increases to 0.2 MPa, which is 2.17% of the initial value.

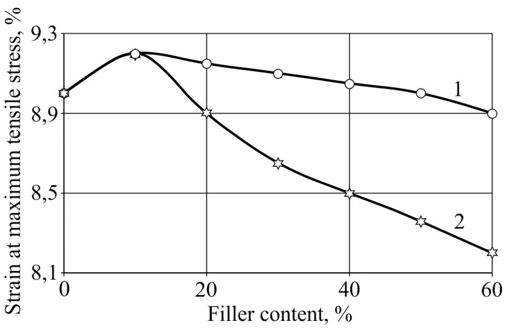

**Figure 4** Strain at maximum tensile stress versus filler content for the NSE-filled polymer composites produced at rotational speed 120 rev/min: 1 – Silitin Z86, 2 – Silitin V88

With increasing the filler content in polyethylene, the strain at maximum tensile stress decreases. It is 0.8 MPa for Silitin V88 and 0.1 MPa for Silitin Z86. A higher strain at maximum tensile stress was observed only on the addition of Silitin Z86, which can be explained by a much better polyethylene adhesion of Silitin Z86 resulting from the properties of this filler, as proved by the results of Young's modulus and tensile strength, also reported in other studies [20, 21].

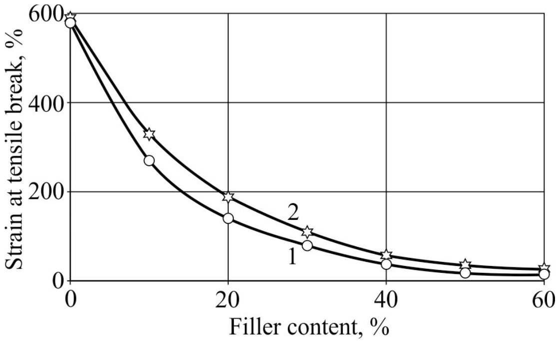

**Figure 5** Strain at tensile break versus filler content for the NSE-filled polymer composites produced at rotational speed 120 rev/min: 1 – Silitin Z86, 2 – Silitin V88

At the screw rotational speed set to 120 rev/min and the percentage of Neuburg siliceous earth changed in the range from 0 to 60%, the examined injection molds have a smaller strain at break compared to that of the unfilled polymer (Fig. 5). Polyethylene composites filled with NSE, Silitin Z86 and Silitin V88, reveal a very high decrease in strain at break, by 574 MPa and 564 MPa, respectively, which is 97.6% and 95.6% of the strain at break for the unfilled polymer. This effect is probably caused by bad adsorption on the surface of the filler and low filler pellets wettability by the melted polymer. The wettability of fillers and their dispergation in the polymer matrix depend, among others, on the polymer's surface energy and its polar and dispersive components, as well as on dipole moment and polarizability. The value of surface energy can be changed by the addition of surface-active substances. Introduced in the polymer composite, these substances adsorb on the interface and decrease the surface tension of the fillers, playing the role of structural plasticizers [19].

On a more developed surface of contact between Silitin Z86 and the polyethylene, during increase in loading we can observe a higher strain in the produced adhesive joint than is the case with Silitin V88, which has a smaller specific surface area of pellets, as also reported in other studies [20].

### 3.2 Hardness Tests of NSE-Filled Composites

The investigation of the effect of filler content on hardness of the produced injection molds was performed by Shore-D hardness scale measurements. The results are illustrated in Fig. 6 as a relationship between Shore-D hardness and different filler contents.

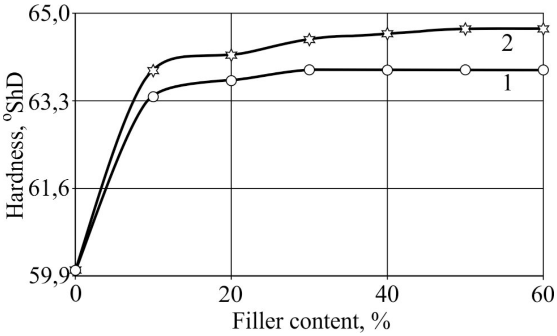

**Figure 6** Hardness of injection molds versus filler content for NSE-filled polymer composites produced at rotational speed 120 rev/min: 1 – Silitin Z86, 2 – Silitin V88

When added to high-density polyethylene, even in a small amount, Silitin Z86 and Silitin V88 cause an increase in hardness of the produced composite. A further increase in the filler's content in the composite also leads to a small increase in hardness. The increase in hardness can be attributed to the lamellar structure of kaolinite which is further reinforced by silica pellets. The produced molds have the highest hardness when the content of both filler types is the highest. This phenomenon was previously observed and described in the literature [21, 22]. The highest increase in hardness can be observed for Silitin V88 and it amounts to 7.75%, which corresponds to an increase in hardness by 4.65 °Sh D. This can be caused by the presence of hard quartz pellets, the size of which is bigger than that of Silitin V88.

### 3.3 Morphology of Produced Polymer Composites

As part of the experiments, we performed microscopic examination of cross sections of both the unfilled polymer and produced polymer composites containing 20, 40, 60 wt% Silitin Z86 and Silitin V88. The photographs of cross-sectional morphology of the examined injection molds are shown in Fig. 7.

All microscopic images of cross sections of both the produced composite injection molds and untreated high-density polyethylene show the presence of parallel scratches, which means that the cross-sectional surface of the specimens was scratched by a knife during their preparation. In the photographs, the white regions indicate polyethylene; the dark regions are the kaolinite lamellas, while the dark spots indicate quartz pellets. Each photograph reveals – to a smaller or higher degree – the structure's non-homogeneity, which demonstrates that the filler was insufficiently mixed with the polymer matrix. The presence of polyethylene agglomerates also points to insufficient surface wettability of the polymer matrix.

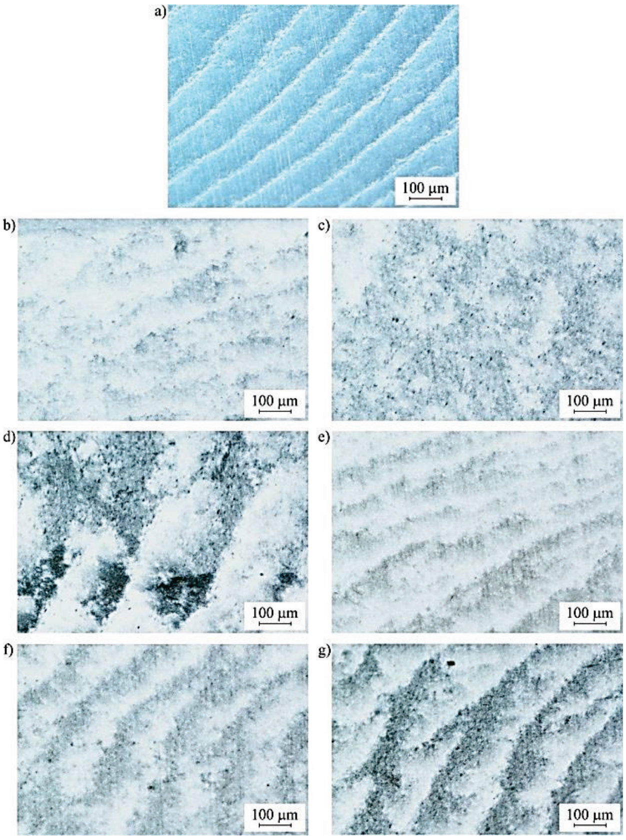

**Figure 7** Cross section of unfilled high-density polyethylene and NSE-filled polymer composites produced at rotational speed 120 rev/min: a) 100% high-density polyethylene b) PE-HD + 20% Silitin Z86; c) PE-HD + 40% Silitin Z86; d) PE-HD + 60% Silitin Z86; e) PE-HD + 20% Silitin V88; f) PE-HD + 40% Silitin V88: g) PE-HD + 60% Silitin V88

The cross sections of specimens with 20 wt% Silitin Z86 exhibit the highest yet still insufficient homogeneity of the filler in the polymer matrix (Fig. 7). The examination of specimen morphology reveals a non-uniform filler distribution for 40 and 60 wt% Silitin Z86 and Silitin V88 (Figs. 7d, 7e, 7f, and 7g). The micrometric particles of Neuburg siliceous earth (NSE) with a higher surface energy than that of high-density polyethylene also reveal a tendency to agglomerate in this place (Figs. 7d and 7g). This is undesired, as it leads to decreasing the contact surface between the filler and the polymer, thus causing lower adhesion.

## 4 CONCLUSIONS

The modification of polymers by a wide variety of fillers causes numerous changes in processing, mechanical properties and morphology of product structure. Examination of properties of thereby treated polymers is vital in terms of application of a given polymeric composition.

The results of a polyethylene composite filled with untreated Neuburg Siliceous Earth demonstrate that its mechanical properties are not uniform: Young's modulus and tensile strength of the composite first decrease and then increase with increasing the filler's content.

The addition of 60 wt% Silitin Z86 leads to an increase in stress at break compared to its initial value, when the filler was not added. As for Silitin V88, an increase in the filler's content leads to decreasing the stress at break of the examined composite material.

The strain at maximum tensile stress of the examined injection molds increases when the filler content is increased to 10%, and then it begins to decrease. The examined injection molds have a smaller strain at break compared to that of the unfilled polymer.

Injection molds with different NSE contents have a higher hardness than the unfilled polymer. The highest hardness was observed for the specimens with 60 wt% of the tested filler types.

The optical microscope results of specimen cross-sectional morphology reveal an uneven distribution of the filler in the polymer. The produced polymer composites with 40 and 60 wt% Silitin Z86 and Silitin V88 show a strong tendency to agglomerate. The wide use of NSE as filler for polyethylene polymers must involve modification of the polyethylene's surface in order to increase surface energy, decrease agglomerate size and improve the filler's dispersion and distribution in the polymer matrix. It is also recommended that the mixing during production of a composite pellet product be made more intensive, e.g. by equipping the screw with elements for intensive mixing and shearing or by elongation of the plasticizing unit.

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