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EVALUATION OF STRESS-STRAIN PROPERTIES OF SOME NEW POLYMER-CLAY NANOCOMPOSITES FOR AEROSPACE AND DEFENCE APPLICATIONS

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Abstract: *This paper presents the effects of three different types of layered silicates, namely bentonite (Btn), Montmorillonite K10 (K10) and Nanomer I.30P (I.30P), on the performance of some new hybrid materials based on a commercial polypropylene. The study evaluated the dependence of mechanical properties on the clay nature and particle size. Low volume additions ($\approx 5\%$) of clay nanoparticles have influenced differently the interfacial adhesion between matrix and filler, yielding in modified stress-strain properties. Experimental data indicated that Btn and I.30P acted as reinforcing fillers, whereas K10 behaved as a plasticizer.*

Keywords: *polymer-clay nanocomposites, mechanical properties, interfacial adhesion*

1. INTRODUCTION

Starting from the Toyota Research Group first polymer-clay nanocomposite, the improvement of properties of this new class of materials intended for structural applications was demonstrated. They rapidly enjoyed a spectacular success in military and commercial aircraft equipment, the automotive industry and even sporting goods and health care products.

Polymer-clay nanocomposites have been considered as matrices for fiber-reinforced composites for aerospace components, since the aerospace industry requires lightweight materials with high strength and stiffness, among other qualities. These enhanced polymer systems provide opportunities to address material limitations in advanced system concepts: impact resistance, control

over the coefficient of thermal expansion, enhanced fire retardancy, superior barrier properties against gas and vapor transmission (for military rations in defence industry, for barrier liners in storage tanks and fuel lines for cryogenic fuels in aerospace systems), suppression of microcracking and increased modulus, enhanced scratch resistance and ballistic performance, etc. [1-3]. The use of such composites as structural materials in both commercial and general aviation aircraft has been increasing, primarily because of the advantages composites offer over metals (*e.g.*, lower weight, better fatigue performance, corrosion resistance, tailorable mechanical properties, better design flexibility, lower assembly costs) [3]. While most of the Airbus A 380 fuselage is aluminum, composite materials comprise more than 20% of its airframe. Boeing 787 Dreamliner is 80%

composite by volume and each 787 contains approximately 35 short tons of carbon fiber reinforced plastics (CFRPs), made with 23 tons of carbon fiber.

Ongoing research and studies have shown that, in general, polymer-clay nanocomposites exhibit great improvement in mechanical properties, such as strength and stiffness, compared to pure polymers upon addition of minimal amount of nanosize clay particles. Since the use of nanoscale fillers in polymers has granted the possibility to design new materials with significantly improved performance and multifunctionality, numerous polymers have been investigated: polypropylene, polyethylene, polystyrene, polycaprolactone, polyimides, polycarbonates, poly(ethylene oxide) and epoxydic resins [2].

Polypropylene (PP) is one of the most widely used polyolefins and one of the most interesting thermoplastic materials due to its low cost, low density, high heat distortion temperature, processability and extraordinary versatility in terms of tailored properties and applications [4].

Huge consumption of polypropylene in hi-tech industries motivates the scientific community to find new approaches to improve mechanical properties of this polymer, so they don't negatively affect other required performance properties like impact resistance, controlled crystallinity, toughness and shrinkage. In order to improve polypropylene competitiveness in engineering applications, it is desirable to increase dimensional stability, heat distortion temperature, stiffness, strength, barrier properties, and impact resistance without sacrificing the processability.

Nowadays, clay nanoparticles play a key role in improving the mechanical, surface and barrier properties of PP. The most commonly used clays are from the smectite mineral group, such as monmorillonite (MMT), hectorite and saponite [5]. Montmorillonite belongs to the layered silicates nanoclays class, consisting of nanoparticles with anisotropic, plate-like, high aspect-ratio morphology, which leads to an improved permeation barrier. Using montmorillonite, the matrix polymer is expected to display increased dimensional stability at low reinforcement loading, characteristic that

makes it useful in aerospace and automotive industry. MMT has the widest acceptability for use in polymer-clay nanocomposites because of its availability, well known intercalation and/or exfoliation chemistry, high surface area and high surface reactivity. The layer aspect ratio can be in the range 1000 in well dispersed state without breaking of layers (although, during preparation process of nanocomposite, the breaking of clay layers into small plates occurs and the aspect ratio decreases to about 300-500) and surface area is about 750 m²/g [6].

Despite of its advantages, MMT is incompatible with polymers, since it is a hydrophilic compound, as all nanoclays from its class. Therefore, it is recommendable to use organically modified clays in order to improve the matrix-filler compatibility which strongly affects the interface adhesion properties of nanocomposites. Even more, a compatibilizing agent, such as maleic anhydride grafted polypropylene (PP-g-MA), in the case of PP-clay nanocomposites, may be used during preparation [7,8].

Melt intercalation by twin-screw extrusion is the main method to obtain polymer-clay nanocomposites. The success of the exfoliation by melt blending is associated with the presence of strong interactions between clay and polymer chains, as well as the diffusion of polymeric chains among clay layers. The process efficiency depends on the temperature, processing time, and screw shear profile. Moderate processing intervals, low process temperature, and medium shear rates were proven to be the best experimental conditions to obtain the dispersion of the clay in exfoliated and intercalated structures. Melt-mixing or compounding is the preferred processing method in the case of PP-clay nanocomposites because it is generally more attractive than solution preparation (or, in other cases, *in situ* polymerization), due to the better commercial feasibility and lower costs [9,10].

This paper presents a study on the variation of mechanical properties of a series of new polypropylene-clay hybrid nanocomposites obtained by the melt compounding technique, starting from a commercial grade PP and using three different types of nanoclays, namely:



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bentonite (Btn), a Romanian native clay from Valea Chioarului (Maramures), containing 70% MMT; Montmorillonite K10 (K10); Nanomer I.30P (I.30P), an organically modified MMT. The use of the Romanian native bentonite as filler for hybrid composites was investigated as a viable alternate option to other clays, because it is less expensive than commercial MMT. The comparative evaluation of these new nanocomposites was performed in order to estimate their competitiveness and to envisage the range of applications.

2. EXPERIMENTAL

2.1 Materials. The *polypropylene* used in experiments was an isotactic polypropylene (Malen-PF 401), a product supplied by Petrochemia Plock S.A. Poland. Polymer has a melt temperature of 170°C; decomposition temperature range 250–430°C, Vicat softening point 148°C; melt flow index (2.16 kg/230°C) 2.4–3.2 g/10min; volatile matter 0.3%wt.; isotactic index 95%.

Commercial additive agents, viz. Na-bentonite, Montmorillonite K10 and Nanomer I.30P were used as received.

Na-Bentonite was provided by S.C. Mateo S.R.L. It was collected at the site Valea Chioarului (Maramures County, Romania) [11]. Main characteristics of bentonite: powder, light cream colored, specific surface area = 25-30 m²/g; apparent specific weight = 260-300 g/L; particle size 9-20µm.

Montmorillonite K10 is a sodium montmorillonite (MMT) clay, with a high-purity grade, supplied by Sigma Aldrich Co. (USA). Main characteristics: specific surface area of 220–270 m²/g; white powder; bulk density = 300-370g/L; particle size 5-10 µm.

Nanomer I.30P is an organically modified montmorillonite, was donated by Nanocor Inc., USA. Main characteristics: natural montmorillonite (MMT) modified by octadecylamine (70-75% montmorillonite and 25-30% octadecylamine), white powder; density 1.7 g/cm³; mean dry particle size 10-25 µm, specific surface area of 9.51 m²/g and interlayer distance 2.10 nm.

2.2 Hybrid nanocomposites preparation

2.2.1 Melt processing. The hybrid organic-inorganic composite samples with different clay types, added in the same amount (5%), were prepared by melt mixing in a HAAKE RHEOCORD 9000 mixer (equipped with two internal roller mixers and a capacity of mixing chamber of 48.3 cm³; fill factor 0.8).

Before compounding, PP and clays were thoroughly dispersed in acetone for 1 h and dried, first in air and then at 80°C in a vacuum oven for 24 h to remove moisture and to enable uniform distribution of PP pellets and clay particles, which will subsequently influence the nanocomposites homogeneity. Samples composition is listed in Table 1.

Table 1.
Composition of the nanocomposite samples

Sample	Clay	Clay particle size, µm	Composition, %	
			PP	Clay
PP	–	–	100	–
PP-Btn	Bentonite	9-20	95	5
PP-MMT	Montmorillonite K10	5-10	95	5
PP-I.30P	Nanomer I.30P	15-25	95	5

Processing parameters were chosen as to minimize possible degradation of the matrix polymer and the inorganic filler. The mixing temperature was maintained at 190°C, the rotor speed was set at 100 rpm and the time was 7 min. After processing, samples were removed from the mixer and cooled down to room temperature.

2.2.2 Test samples preparation. For the mechanical tests, composites were cut into small chips or granules, which were then dried. In order to prepare films of desirable dimensions, the composite chips were pressed between Teflon sheets (12 MPa at 200°C for 5 min) in a laboratory heated press. The samples were then cooled in the press to room temperature. Films with thickness of about 1.0 mm were obtained. Specimens were finally cut in the usual dumb bell shape.

2.2.3 Mechanical testing. Tensile measurements, tensile strength, $\sigma = F/A_0$ vs. elongation $\varepsilon (\%) = (l - l_0)/l_0$, were performed at room temperature using a tensile test machine TIRA-TEST 2200 Germany, according to ASTM D-638-08. Measurements were made to a crosshead speed of 10 mm/min. Bone-shaped test specimens were cut from a single test bar using a hydraulic knife along the same direction. Five specimens of each sample were tested to establish testing reproducibility and average values are given with the standard deviations. The deviation of the data around mean values was less than 5%.

3. RESULTS AND DISCUSSION

3.1 Influence of the clay nature on the stress-strain relationship in nanocomposites. Mechanical properties of polymer-clay nanocomposites can be altered by various

Table 2. Mechanical properties of the nanocomposites samples

SAMPLE	Young modulus (MPa)	Yield strength (MPa)	Elongation at yield (%)	Tensile strength (MPa)	Elongation at break (%)
PP	1512	31.97	5.06	26.11	8.20
95PP/5 Btn	2198	30.35	3.96	27.83	5.06
95PP/5 K10	1159	18.21	8.69	18.00	10.77
95PP/5 I.30P	2146	28.27	3.61	26.43	4.29

inorganic particles are rigid and have a much higher stiffness than the thermoplastic matrix,

the composite modulus can be readily improved by adding either micro- or nanoparticles. In our case, the Young's modulus, determined from the slope of the initial elastic region, was 1512 MPa for neat PP, whereas its values for PP-Btn and PP-I.30P nanocomposites increased with 42-45% upon addition of 5% clay, as compared to pure

factors: properties of the polymer matrix, clay particle size and morphology (anisotropic, platelet-like, high aspect-ratio), particle loading and distribution, interfacial adhesion between clay and matrix, etc. Mechanical properties of hybrid composites are strongly related to the interfacial adhesion between clay particles and matrix, as known. The interactions in the polypropylene-clay systems are responsible for significant changes in mechanical properties. A strong interfacial adhesion leads to high tensile strength and low elongation, whereas a weak interfacial adhesion leads to low strength and relatively high elongation [12]. Table 2 shows experimental data obtained for some mechanical properties of the hybrid composites.

It was observed that the clay nature had critical effects on the mechanical properties of samples. Of primary importance for our study are Young's modulus, strength at the yield point and tensile strength.

The Young's modulus (elastic modulus) of a clay-polymer composite is generally determined by the elastic properties of its components (filler and matrix), particle loading and aspect ratio [13]. As known, the elastic modulus is a stiffness parameter which governs by the size and amount of the dispersed phase. In the light of the fact that the

polymer matrix. At the same time, it was observed that Young's modulus for samples with K10 decreased with approx. 25%.

This variation depends mainly on the microstructure and the interface morphology of the polymer-clay composites (including the interfacial bonding, the form, size and spatial distribution of the clay particles into the matrix, as well as the thickness of the interface) [14]. When there is a poor binding between the matrix and clay, the composite becomes brittle because the applied load may not be transferred to the clay [15]. Therefore, considering the nature of clays, it is obvious



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that I.30P had a beneficial effect on the nanocomposites elastic modulus than K10, due to the presence of the aliphatic segments (belonging to the organic modifier, octadecylammonium) in its structure which acts as a compatibilizing agent. An interesting behavior was recorded for the Romanian native bentonite: at the same clay content, the corresponding nanocomposite samples showed the highest value of Young's modulus. As Btn is not organically modified and despite the low polarity of PP, we presume that its strengthening effect, as result of adhesion forces at the interface, may be exclusively due to its particular composition and particle size, as well as particles distribution in the matrix as resulted from processing.

On the other hand, the yield strength of the PP-Btn nanocomposites decreased about 5%, 12% for PP-I.30P and 43% for PP-K10, respectively, compared with neat PP. Concerning the tensile strength, values slightly increased for PP-Btn (6.6%) and PP-I.30P (1.2%), but for PP-K10 the tensile strength registered a dramatic decrease of about 31%.

The strength of nanocomposites relies on the effectiveness of stress transfer between matrix and filler [13]. Factors like particles-matrix interfacial bonding strength and particle loading, as well as particle size, significantly affect the composite stress-strain behavior. For well-bonded particles, the applied stress can be efficiently transferred to the particles in the matrix, which clearly improves the strength (Btn and I.30P). However, for poorly bonded particles (K10), the strength decrease occurs upon adding particles because the adhesion is still weak at the interface, despite the increase of the interfacial layer volume on the expenses of the overall matrix volume. This explains data we obtained for all our PP-clay nanocomposites.

Nanoclay particles are stiff materials with no elongation properties; therefore, their addition can lower composites elongation [16]. The elongation at yield for PP-Btn and PP-I.30P decreased, as expected, with approx. 22% and 28.5%, respectively, whilst for PP-K10 increased with 55%. Concerning the elongation at break, samples PP-Btn and PP-I.30P displayed a decrease of 38% and 48%, respectively, but samples PP-K10 showed an increase of 31%. This behavior of hybrid composites, consistent with data for elastic modulus and upholding our hypothesis, was induced by the stiffness of clays and caused by the clay-polymer interface adhesion.

A geometric model to correlate the interfacial adhesion and the fracture mechanism (Figure 1) is already available [17] and it takes into consideration the voids formation and their evolution during stress.

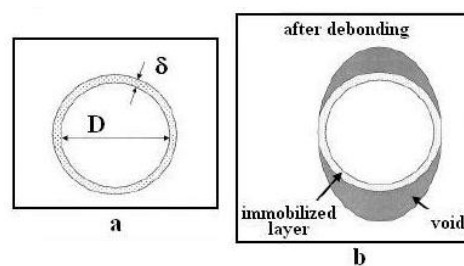


Figure 1. A geometric model that correlates the interfacial adhesion and the voids formation mechanism (D =particle diameter; δ =interfacial layer thickness)

It has been shown [17] that clay particles can facilitate the development of microvoids and activate dilatational yielding in the deformed zone close to the fracture surface. It is generally agreed that for toughening to occur in rigid filler systems, the particles must debond from the matrix, creating voids around the particles and allowing the interparticle ligaments to deform plastically [18]. The voids reduce the macroscopic plastic resistance of

the material, and thereby potentially increase the fracture strain and the overall toughness. Ideally, the voids are not likely to form immediately upon application of stress as this may reduce the elastic modulus.

A schematic illustration of the fracture mechanism in PP-clay nanocomposites is presented in Figure 2 [19]. For samples with poor interfacial adhesion (Figure 2a), the cracks start from the pole of the filler surface and form a huge number of microvoids between the filler and matrix.

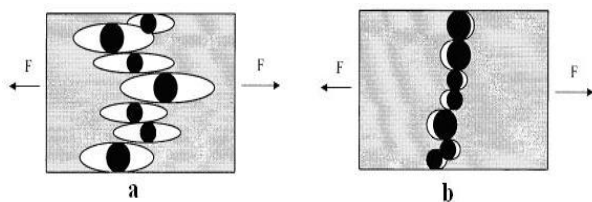


Figure 2. Schematic illustration of fracture mechanism in polymer-clay composites with (a) poor and (b) good interfacial adhesion

The microvoids are dragged along the direction of the stress. This fracture mechanism leads to a low tensile strength and relatively high elongation (PP-K10). On the other hand, in samples with good interfacial adhesion (Figure 2b), the cracks start from the equator of the filler and develop in the matrix or along the interface. Such fracture mechanism leads to a high tensile strength and low elongation (PP-Btn and PP-I.30P). Experimental data presented herein are consistent with the theoretical model, with a special mention for PP-Btn samples that proved to have the best mechanical properties in the series. Further confirmation will be obtained by comparative SEM analysis which is currently underway.

3.2 Effect of particle size on the stress-strain relationship in nanocomposites. The effect of particle size on the nanocomposites mechanical properties has recently attracted much attention. The particles restrict the mobility and deformation of the matrix by introducing a mechanical restraint. The restriction in polymeric molecular diffusion in the presence of solid particles occurs because of an effective attraction potential between segments of the chain and the repulsive potential that the polymer is subjected to when it is close to solid particles. The degree of the

particle restriction depends on the properties of the filler and matrix [20].

Interfacial interaction strongly influences properties of polymer composites. In PP-clay composites only van der Waals forces act between polymer and filler. They are, however, sufficient to attach the polymer to the filler. Adsorption of the polymer on the filler surface leads to the formation of an interphase in which molecules have decreased mobility. The existence of the interphase is proved by the increased composite viscosity with increasing specific surface area of the filler and by the similarly changing yield stress and strength (see Table 2). The effect of interaction depends on the size of the contacting surfaces (specific surface area of the filler) and the interaction strength. Smaller particles have larger surface area, leading to increased yield stress. Differences in the strength of the interaction are less significant than that of the specific surface area [18]. Two important structural phenomena must be considered: (1) small particles tend to form aggregates, which deteriorate properties; for particles having specific surface area over $5\text{m}^2/\text{g}$, the aggregation tendency becomes significant; (2) orientation of anisotropic particles determines their reinforcing effect.

Despite some assumptions of conventional micromechanics for rigid clay systems (*e.g.*, the zero thickness assumption for interfaces), numerous studies have offered mathematical support for models which include the interface effect by taking into account an interphase region between the matrix and particles [21], such as the self-consistent scheme based on a “particle-interphase-matrix” three-phase unit cell model [22].

As recorded in many other studies, the stress-strain relationship in nanocomposites significantly exceeds classical micromechanics predictions. Another possible explanation, apart from the contribution of the interphase, might be considered the natural trend of particles to aggregate. Therefore, in some systems, the primary nanoparticles aggregation (resulted from particles preparation prior to melt mixing in the presence of the matrix polymer) rather than polymer–nanoparticle interactions at the interface is mainly



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responsible for the observed reinforcement effect [23].

For the series of PP-clay nanocomposites we studied, the experimental data obtained for clays with different particle size, presented in Table 2, are consistent with theories available in the literature [24]. First observation is the opposite effect of K10 platelet-shape nanoparticles, compared with the other clays, despite its low value of particle size (5-10 μm) and narrow size distribution. The micrometric dimensions of particles are given by the primary agglomeration, but the smaller the particle size, the thicker the interphase layer on the expenses of the total volume of matrix, the component that actually contributes to energy adsorption. Therefore, the sample had a lower resistance and the deformation increased by 55% at yield and by 31% at break. For Btn (9-20 μm) and I.30P (15-25 μm) containing samples, the clay particles had a reinforcing effect, although their particle size is higher and have a wider size distribution. By this, the secondary aggregation (caused by composite preparation) occurs in a lower measure.

Both the larger (I.30P) and the smaller (K10) particles had a negative effect on the stress-strain relationship due to the presence of large individual particles (I.30P) or agglomerates (K10), which act as stress-concentrators that trigger the fracture mechanisms. The effect in the K10 containing samples was the result of particle-matrix debonding and plastic stretch of the interparticle ligaments [25]. Composites made with smaller particles would exhibit a larger reduction in the amount of deformable matrix because, at certain filler content, the interfacial area between filler and polymer is larger. This causes the composites made with K10 to exhibit lower fracture resistance (18 MPa) than those made with larger particles, I.30P (26.43

MPa), or medium size particles, Btn (27.83 MPa), regardless of the presence of the primary agglomerates. This effect depends on the compatibility at the filler-matrix interface, which determines the thickness and the mobility of the interphase layer.

3. CONCLUSIONS

This study is focused on the comparative evaluation of mechanical properties of a series of new PP-based nanocomposites, prepared by melt mixing method, using as fillers three different types of clay, in order to establish the level of competitiveness of a Romanian native bentonite to be used as an alternative to commercially available clays.

Experimental results proved that clay addition strongly affects the stress-strain dependence in the new composites. Thus, it was found that Young's modulus and tensile strength increased, whilst elongation at yield and break decreased for composites with Btn and I.30P. For composites with K10, Young's modulus and stress at break decreased, while elongation at yield and break increased. Therefore, K10 acted more as a plasticizer, in opposition with Btn and I.30P which effectively reinforced the matrix. The best results were obtained for samples containing Btn, the natural unmodified filler having an average particle size and size distribution.

For rigid clay systems, delamination of the clay layers occurs and results in a moderately increased toughness. Hence, it was suggested that extensive matrix shear yielding, activated by full debonding of the clay-matrix interfaces, leads to improvements in toughness. The relationship between the toughness of composites and the particle size becomes a competition of two effects: (1) a negative stress concentrating effect of particles

that increases with larger particles and agglomerates, and (2) a reduction in the amount of deformable polymer matrix that dominates in composites with smaller particles. However, due to the low polarity of PP, it is difficult to obtain nanocomposites with homogeneous dispersion of the silicates at the nanometer level inside the polymer. Organoclay containing layered silicates modified by nonpolar long alkyl groups are still relatively more polar than PP and may act as compatibilizing agents between matrix and clay particles.

Considering this study results, we have concluded that enhancement in mechanical properties might be achieved through two approaches used simultaneously: (a) optimization of processing parameters, the kinetic criterion being of major importance for clay layers debonding; (b) enhancement of the compatibility of the matrix-filler system by using both the organically modified clays and the modified PP, namely maleic anhydride grafted polypropylene (PP-g-MA), as matrix compatibilizing agent. This will be the subject of an upcoming study.

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