

## Effects of a Titanate Coupling Agent on the Mechanical and Thermo-Physical Properties of Talc-Reinforced Polyethylene Compounds

Adib Kalantar Mehrjerdi, Bemnet Abebe Mengistu, Dan Åkesson, Mikael Skrifvars

School of Engineering, University of Borås, SE-501 90, Borås, Sweden

Correspondence to: M. Skrifvars (E-mail: mikael.skrifvars@hb.se)

**ABSTRACT:** An experimental study was carried out to investigate the effects of a titanate coupling agent on the mechanical properties, moisture absorption, and thermal conductivity of talc-filled high-density polyethylene (HDPE). Talc (0–35 wt %) was used as reinforcement particulate filler in an HDPE matrix and samples were prepared in a micro-compounder and an injection molding machine. Isopropyl tri(dioctyl)phosphate titanate (0.5 wt %) was used as coupling agent. Composites with and without coupling agent were evaluated for changes in mechanical and thermo-physical properties, morphology, and void content. Addition of the titanate coupling agent most often resulted in an increase in stiffness and tensile strength. Furthermore, both the void content and the elongation at break of composites were reduced. Results also showed that the coupling agent had no effects on the thermal conductivity, thermal diffusivity, and specific heat capacity of the composites. In addition, it was observed that the coupling agent was more effective at low concentrations of filler. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 40449.

**KEYWORDS:** extrusion; polyolefins; thermal properties; blends

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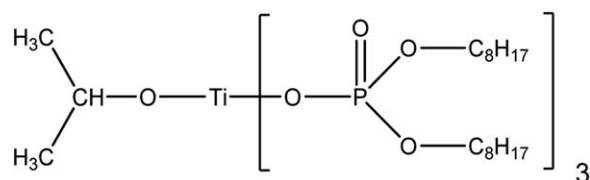
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### INTRODUCTION

The use of reinforcing agents, additives, and fillers can change the properties of polymers so that they can fulfil the material requirements of specific applications. Many fillers of different types and shapes can be blended with polymers, such as metals, carbon, glass fiber, and ceramics, each of which can contribute different qualities and properties. For example, carbon black has been used as an economical and highly effective additive in many thermoplastics and also in thermoset compounds for outdoor stabilization.<sup>1,2</sup> In the literature on composites, it has been shown that compounding polymers with fillers and particles can improve mechanical properties,<sup>3,4</sup> electrical conductivity, and thermal behavior<sup>5,6</sup> as well as protect against ultraviolet radiation.<sup>7</sup> Heat transfer properties are of particular interest in many polymer applications, as polymers are mainly thermal insulators. Electronic packaging, pipe networks, heat exchangers, and domestic appliances are applications in which an increase in the heat transfer properties would be an advantage.

In electronic applications such as computers, good thermal conductivity of the covering material is a vital property in order for the heat generated to be dissipated easily from the heat source to maintain the temperature of the device at a lower, desired

level. In other areas such as heat exchangers, higher heat exchange performance can be achieved with higher thermal conductivity. It is well known that the most commonly used polymers such as polyethylene (PE) and polypropylene (PP) can be considered to be thermal insulators with low thermal conductivity values ranging from 0.1 to 0.5 W m<sup>-1</sup> K<sup>-1</sup>. To improve the thermal conductivity of the polymers, several inorganic materials such as graphite and metallic powders have frequently been used as thermo-conductive fillers.<sup>5,8,9</sup> In a review by Bigg et al.,<sup>10</sup> it was shown that if the ratio of thermal conductivity of the filler to thermal conductivity of the matrix is >100, then the thermal conductivity of the compound is not increased. An important practical conclusion that can be drawn from this is that inorganic fillers with rather low thermal conductivity can be used equally effectively as metallic fillers to improve the thermal conductivity of polymer composites. In this case, the morphological characteristics of the composite, the size and shape the filler, and how the particles are in contact with each other in the matrix are more important factors than the relative thermal conductivity of the two-phase system. Another study showed that the thermal conductivity of copper-filled polypropylene did not improve in spite of the very high conductivity of pure copper, due to the poor interconnectivity of the copper particles.<sup>11</sup> The practical consequences of these studies are that



**Figure 1.** Chemical structure of titanate coupling agent.

inorganic fillers such as aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), magnesium oxide (MgO), and talcum can be used to produce thermally conductive composites.

Talc, a filler used in many polymer applications, is a mineral composed of hydrated magnesium silicate arranged in three disc-shaped layers. In the middle, there is a layer of magnesium-oxygen/hydroxyl octahedra, while the two outer layers are composed of silicon-oxygen tetrahedra. These layers are kept together by van der Waals forces, and the layers are able to slip over each other easily.<sup>12,13</sup> Thanks to its unique characteristics such as softness, chemical inertness, slipping, oil and grease absorption, whiteness, availability, and rather low price, talc has been used for many years as a popular filler in a wide range of industrial applications such as paper, pharmaceuticals, plastics, paint, lubricants, ceramics, and cosmetics.<sup>14</sup>

In recent years, there has been interest in investigating the effect of talc in polypropylene (PP) blends not only as a filler to reduce material costs but also because of its functional properties. Thus, there have been several studies on the influence of talc on the mechanical properties, crystallinity, thermal stability, and crystallite nucleation of PP/talc blends.<sup>15–19</sup> However, there have been fewer studies on the effects of talc on high-density PE (HDPE), particularly in terms of thermo-physical properties. From a previous study, we reported that adequate talc input enhanced the thermal conductivity of the HDPE/talc blends, while the mechanical properties of the composites remained almost unchanged.<sup>20</sup> In addition, it was shown that the adhesion between particles and matrix was weak. Thus, HDPE/talc composites should show a balance between good thermal conductivity and adequate mechanical properties while retaining ease of processing. It would, therefore, be advantageous to modify surface of the talc particle in order to increase adhesion of the particle to the matrix, which should further decrease the amount of micro-voids and further increase the thermal conductivity of the composites in addition to improving the mechanical properties.

In this article, we examine the formation and the properties of thermally conductive composites of HDPE and talc in the presence of a titanate coupling agent. The aim is to investigate the morphology and properties of HDPE composites based on talc filler and to compare the effects of the coupling agent on the thermal and mechanical properties. HDPE was blended in a micro-compounder up to a talc loading of 35 wt % and isopropyl tri(dioctyl)phosphato titanate was used as coupling agent. For characterization, specimens were produced by injection molding. The mechanical properties of the composites were

studied using tensile testing. Thermal conductivity of the specimens before and after long-term water absorption was evaluated using the transient plane source (TPS) method. The void content, morphology, and crystallinity of the composites were also measured and analyzed.

## EXPERIMENTAL

### Materials

High-density polyethylene supplied by Total Petrochemicals, Belgium, was used as a matrix material. The HDPE had a carbon black content of 2.5 wt %, a melt-flow index (MFI) of 0.3 g/10 min, and a density of 959 kg m<sup>-3</sup>. Commercial talc powder supplied by LUZENAC, France was used as filler. The density of the material was 2.78 g cm<sup>-3</sup>, and the median diameter (D50) was 10.5 μm. Isopropyl tri(dioctyl)phosphato titanate powder with a chemical structure as shown in Figure 1, was obtained from Kenrich Petrochemicals Inc. (CAPOW-KR 12/H) for use as a coupling agent. The specific gravity of the coupling agent was 1.04.

### Compounding of Composites

Both the HDPE and the coupling agent were dried in a vacuum oven at 70°C. The talc was dried overnight in an air-circulation oven at 100°C to expel moisture prior to blending. Composite samples were prepared by weighing the matrix, filler, and coupling agent as detailed in Table I. Then the weighed materials were mixed manually and loaded into a twin-screw micro-compounder (DSM Xplore, the Netherlands). The compounding was done at 50 rpm for 8 min, at 200°C, for the HDPE without coupling agent, and at 50 rpm for 8 min, at 180°C, for the HDPE with coupling agent. Argon gas was used to maintain an inert environment in the compounder. The melt was fed directly from the micro-compounder using a transfer cylinder into an injection molding machine (DSM Xplore) to produce test samples according to ISO 527-2-5A dog-bone shape for tensile testing.

### Characterization

The tensile properties were determined according to the ISO 527-1 standard test method with a universal testing machine

**Table I.** Composition of the Compounds Studied

Sample	HDPE (wt %)	Talc (wt %)	Titanate coupling agent <sup>a</sup> (wt %)
HT0	100	0	0
HT5	95	5	0
HT10	90	10	0
HT15	85	15	0
HT25	75	25	0
HT35	65	35	0
HTC5	94,35	4,98	0,5
HTC10	89,55	9,95	0,5
HTC15	84,58	14,93	0,5
HTC25	74,63	24,88	0,5
HTC35	64,68	34,83	0,5

<sup>a</sup> According to the supplier recommendation.

**Table II.** Mechanical Properties of the Different Composites (Uncertainties Represent the Standard Error)

Sample	Modulus (GPa)	Strength (MPa)	Elongation at break (%)
HT0	1.76 ± 0.07	57.5 ± 1.0	20.5 ± 1.8
HT5	2.34 ± 0.20	61.7 ± 0.4	14.7 ± 0.8
HT10	2.49 ± 0.12	63.2 ± 0.7	14.5 ± 0.5
HT15	2.24 ± 0.21	63.8 ± 1.2	14.7 ± 1.7
HT25	3.38 ± 0.08	65.0 ± 0.6	13.9 ± 0.7
HT35	4.22 ± 0.05	68.7 ± 0.7	13.8 ± 0.8
HTC5	2.39 ± 0.14	68.0 ± 0.9	14.2 ± 1.4
HTC10	3.38 ± 0.20	73.0 ± 1.7	10.4 ± 0.2
HTC15	3.08 ± 0.39	71.1 ± 0.9	9.7 ± 0.8
HTC25	4.07 ± 0.34	69.9 ± 0.5	8.2 ± 0.3
HTC35	4.73 ± 0.21	71.9 ± 0.3	9.4 ± 0.5

(H10KT; maximum capacity 10 kN; Tinius Olsen, Salford, UK). For each composite, 10 specimens were analyzed at a test speed of 10 mm/min at room temperature using a load cell of 5 kN. The sample dimensions were: length 75 mm, width 4 mm, and thickness 2 mm. A mechanical extensometer with a gauge length of 25 mm was used to measure the specimen elongation.

The transient plane source or hot disc method (TPS 2500 Hot Disk AB, Gothenburg, Sweden) was used to determine the thermal properties of the composites. This test follows the specification of ISO 22007-2, and simultaneously measures the thermal conductivity, thermal diffusivity, and volumetric specific heat of the composites at room temperature. The disk sensor (radius 2.001 mm) was sandwiched firmly between the two identical specimens (80 × 10 × 4 mm<sup>3</sup>). Each measurement was done at three different positions on the sample surface, and the average of values is reported. Because the thermal conductivity of materials might be affected by the moisture absorption, after the thermal conductivity test, all specimens were placed in distilled water and kept at room temperature until saturation. The specimens were weighed every 2 weeks until the increase in weight every 2 weeks became <1%. The thermo-physical properties were again determined for all samples.

The void content was calculated by comparing the measured density with the theoretical density. Measured density of all composites was determined by means of Archimedes' principle according to the ASTM D792 test method B (for testing of solid plastics in liquids other than water). Ethyl alcohol, which has a density of 0.7886 g cm<sup>-3</sup> at 21°C, was chosen as the fluid, and the theoretical densities of the samples were calculated according to the rule of mixtures.

The cross-sectional surfaces of cryo-fractured specimens were studied using scanning electron microscopy (SEM), with an S-4800 ultra-high resolution scanning electron microscope (Hitachi, Japan). To create a brittle fracture, the tensile bar specimens were immersed in liquid nitrogen and broken by hand.

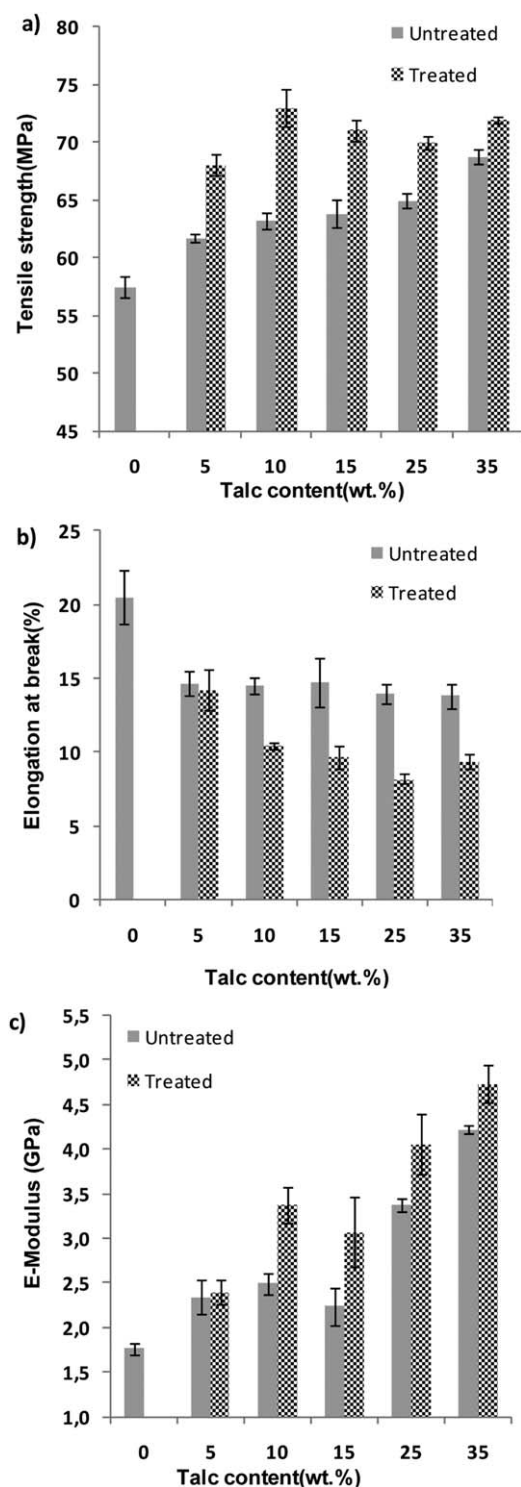
Specific heat capacity measurements were performed using a differential scanning calorimeter (DSC Q2000; TA Instruments). Measurements were carried out in the temperature range of 0–54°C and at a heating rate of 10°C min<sup>-1</sup>. The weight of the sample was in the 10–15 mg range. The instrument was calibrated for the same temperature region before measurements, using a sapphire with a well-known specific heat capacity as reference material. Average values for two replicates at 21°C are reported.

To test the effect of the filler modification on the properties, statistical comparisons based on one-way analysis of variance (ANOVA) at the 95% confidence level were performed. The data were analyzed using Minitab version 15.

## RESULTS AND DISCUSSION

The values obtained for the mechanical properties are presented in Table II. In the absence of the coupling agent (samples marked HT), the difference in mechanical properties between neat HDPE and its composites suggested that talc has a minor reinforcing effect on HDPE, based on the results of tensile strength tests (Table II). However, addition of talc to HDPE yields composites with an elastic modulus that is significantly higher than that of the neat HDPE. The elongation at break dropped sharply when 5 wt % talc was added to the HDPE, and it was observed that the elongation at break remained almost unchanged with higher loading of the talc. The tensile modulus increased with talc content, which was mainly attributed to the replacement of HDPE by the more rigid talc particles and also the talc filler restricting the mobility and deformability of the matrix.

Figure 2 shows the effect of titanate coupling agent on the mechanical properties of the composites. Average increases of 11% for tensile strength and 24% for tensile modulus were obtained with the coupling agent compared to untreated talc, although there was an average decrease of 28% of elongation at break when the coupling agent was added. The tensile strength for the treated composites showed that the treatment had the highest effect on composites with 5, 10, and 15 wt % talc content. For modulus of elasticity, the increase seems to be proportional to the concentration of the filler, with the exception for the sample with 10 wt % talc content. The improvements in the tensile strength and stiffness of the composites are thus partly explained by the better bonding between the matrix and the reinforcement. It is well known that adhesion between the polymer matrix and the reinforcement is one of the key factors that influence the properties of composite materials. The introduction of a coupling agent into the system promotes bonding of polymer to filler; so it improves the performance of the composite. The mechanism of reaction of titanate with the inorganic filler has been discussed in the literature.<sup>21</sup> The titanate coupling agent reacts with hydroxyl groups on the surface of the inorganic filler, resulting in the formation of a titanium monomolecular layer on the talc surface, which increases the compatibility of the filler/matrix interface. By replacing water of hydration present at the talc surface with the organo-functional titanate, the filler/matrix interface will become compatible and the dispersion of filler



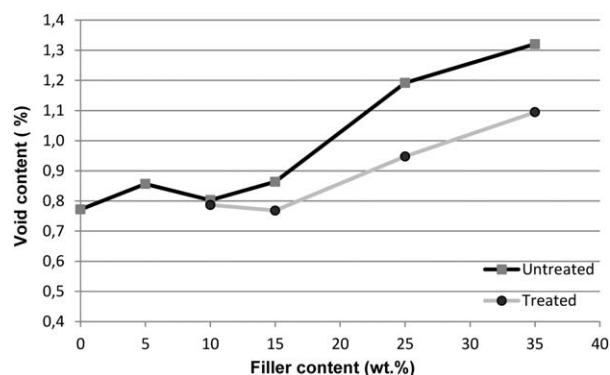
**Figure 2.** Mechanical properties of the composites as a function of filler and treatment (a) Tensile strength (b) Elongation at break (c) E-Modulus (uncertainties represent the standard error).

particles is enhanced. Consequently, this results in a more uniform dispersion and deagglomeration of the talc fillers in the matrix.

The incompatibility of the filler in the matrix causes the formation of micro-voids in the composites. The effect of random

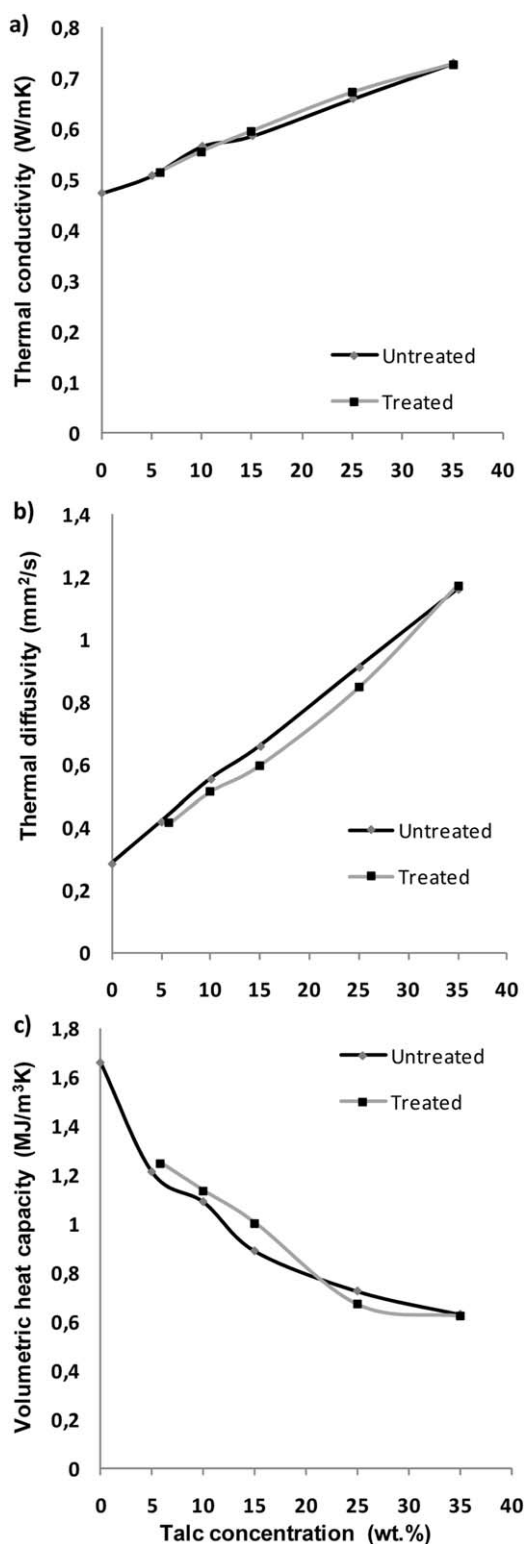
and uniform dispersion can be viewed by studying the void content. In Figure 3, it can be seen that the void content increased with filler loading when no coupling agent was present. These voids might be induced by the debonding of talc particles in the matrix due to poor adhesion. However, it is evident that in the presence of the titanate coupling agent, the void content decreased at all talc loadings for the compounds. Previous studies have shown an improvement in the mechanical properties of filled PP as a result of coating of the fillers with different types of coupling agents such as silane, titanate, and amine.<sup>22–24</sup> Li et al.<sup>25</sup> also examined the mechanical and morphological properties of HDPE/talc compounds and showed that the presence of silane coupling agents resulted in higher stiffness, lower strain at break, and almost unchanged tensile strength. In contrast, other researchers have observed that the tensile and flexural properties decreased while the impact and elongation properties improved for PP/talc composites with the addition of a titanate coupling agent.<sup>26</sup> Also, the results of the current study contradict past reports regarding reduction of the strength and improvement of the deformability of PP/talc and PP/CaCO<sub>3</sub> composites.<sup>23</sup> Moreover, Bajaj et al. showed that when mica was coated with a titanate coupling agent, the yield stress and yield strain increased compared to untreated mica and the tensile modulus decreased when a titanate coupling agent was added.<sup>21</sup>

The variations in the thermal conductivity, thermal diffusivity, and volumetric heat capacity with filler concentration are illustrated in Figure 4. The results show that the thermal conductivity and thermal diffusivity increased in an almost linear dependence with increased talc loading, while the volumetric heat capacity decreased over the talc loading range in the experiment. The thermal conductivity gradually increased with increasing filler content and all the data points formed a straight line, which is in accordance with the model proposed by Agari.<sup>8,27</sup> Most of the thermal characteristics of the untreated samples that were observed in the current experiment showed good agreement with the values for the treated samples, suggesting a negligible effect of the titanate coupling agent. It is known that the thermal conductivity of composites can be enhanced by maximizing the formation of percolated thermally conductive networks while minimizing the heat resistance along the heat channels. Berman described the mechanism of heat



**Figure 3.** Void content as function of filler treatment.





**Figure 4.** Thermo physical properties (a) Thermal conductivity (b) Thermal diffusivity (c) Volumetric heat capacity.

transfer in non-metals by the flow of phonons or lattice vibrational energy.<sup>28</sup> The thermal resistance is affected by various types of phonon-scattering processes, e.g., boundary scattering and interfacial phonon scattering. Thus, in order to maximize

the thermal conductivity in materials, these phonon-scattering processes must be minimized.<sup>29</sup> Although, in the current study, the untreated samples suffered from the presence of higher voids due to poor wetting as shown earlier, the effective thermal conductivity was as high as that for treated compounds. Lee et al.<sup>30</sup> reported that HDPE-containing surface-treated aluminum nitride fillers showed a slightly improved thermal conductivity than for HDPE-containing untreated fillers, which is contradictory to the results we report in this article.

It is known that most polymeric materials absorb moisture to some extent, resulting in dimensional changes, loss of mechanical and electrical properties, less resistance to heat, and increased weathering sensitivity. The moisture content also largely influences the thermal conductivity; for example, the thermal conductivity of a wet material is considerably greater than that of the dry material and water itself. This can be explained by enhancement of the convective heat transfer due to the capillary movement of the water within the wet material. For example the thermal conductivity of a dry clay brick, water, and a damp brick are 0.35, 0.6, and 1 W m<sup>-1</sup> K<sup>-1</sup> respectively.<sup>31</sup> Thermo-physical property measurements were therefore conducted again on the samples after 70 days of immersion in distilled water. Composites with absorbed moisture (for both groups, treated and untreated) had almost the same thermal conductivity, thermal diffusivity, and volumetric heat capacity values as their unabsorbed counterparts. The experimental results were verified statistically by ANOVA. Tukey's Studentized-range test at 5% significance level was used to compare the difference among treatment means. No statistically significant difference was observed between the thermal conductivity of dry composites and that of saturated samples ( $P = 0.0549$ ).

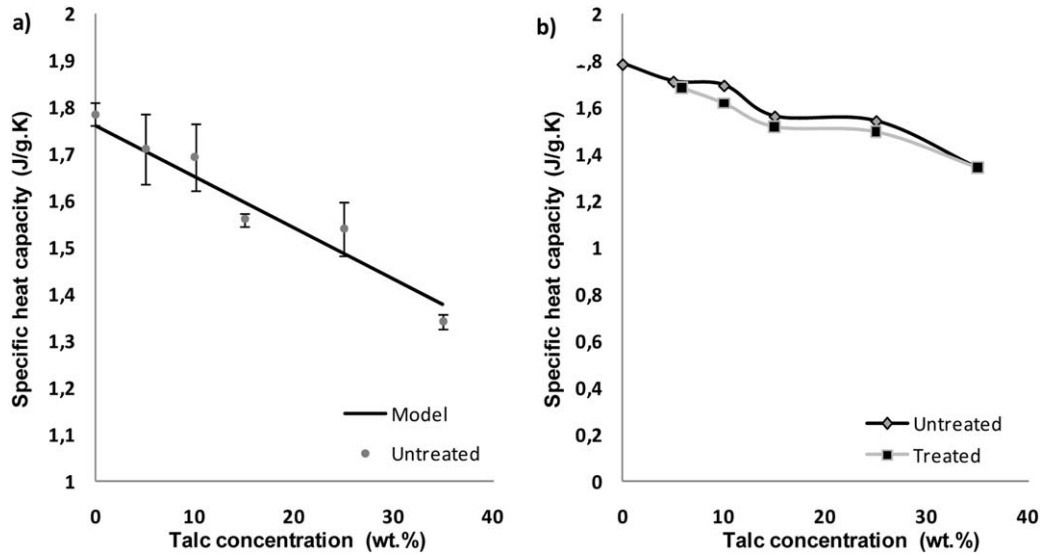
Figure 5(a) shows the specific heat capacities of the composites investigated, without coupling agent. A linear decrease in the specific heat capacity was observed when the filler content was increased from 5 to 35%. The degradation in heat capacity can be attributed to the lower specific heat capacity of the talc, which was measured to be 0.67 J g<sup>-1</sup> K<sup>-1</sup>—as compared to neat HDPE with the value 1.76 J g<sup>-1</sup> K<sup>-1</sup>. A general model can be introduced to explain the relationship, based on the rule of mixtures, see in eq. (1).

$$C_{pc} = C_{pf} \omega_f + C_{pr} \omega_r \quad (1)$$

Here  $C_{pc}$ ,  $C_{pf}$ , and  $C_{pr}$  are the specific heat capacities of composite, filler and resin, and  $\omega_f$  and  $\omega_r$  are weight proportions of filler and resin, respectively.

The effect of the titanate treatment on specific heat capacity is shown in Figure 5(b). Treatment of the composites with coupling agent does not significantly affect the specific heat capacity of the blends.

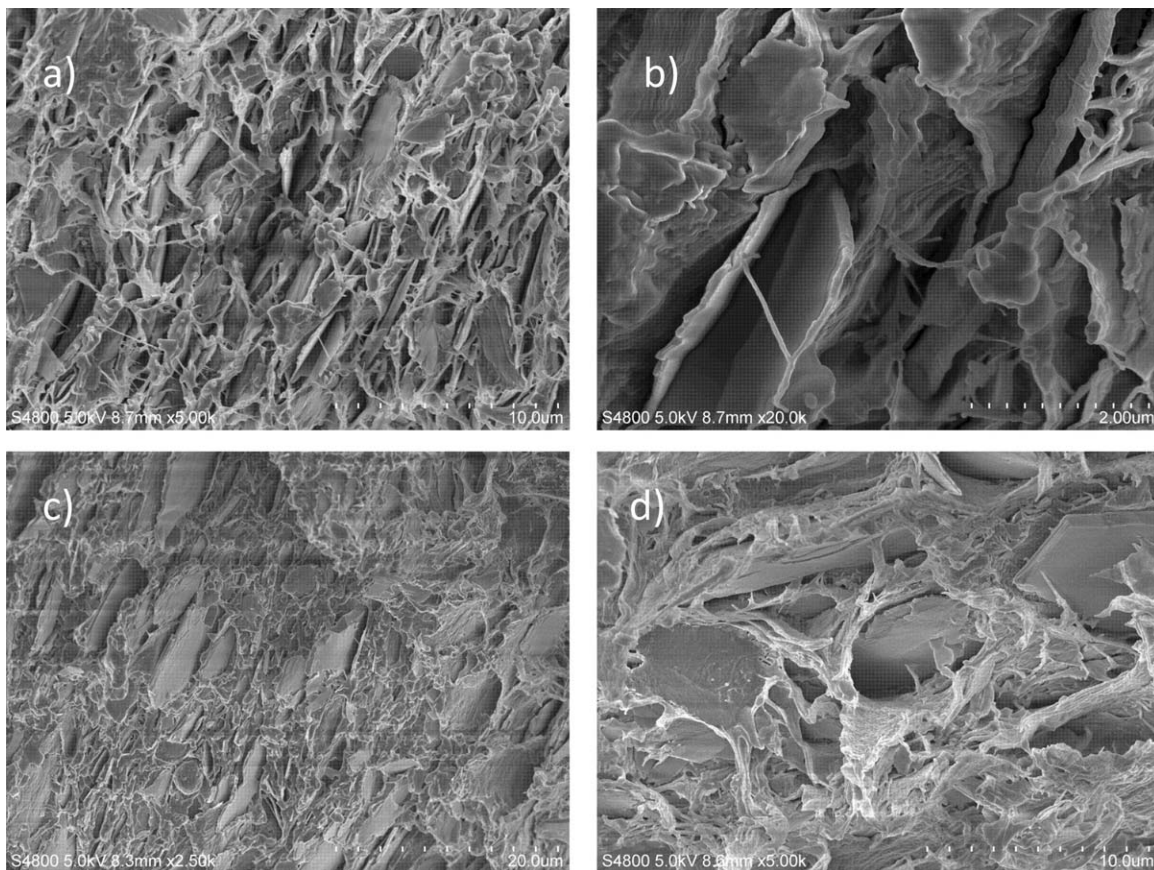
The deduction about reinforcing mechanisms could be confirmed by results on fracture surface morphology. Figure 6 illustrates cryo-fractured surface morphologies of untreated and treated HDPE/talc composites at 35 wt % and at different magnifications. As shown in Figure 6(a), talc particles are clearly visible and well dispersed in HDPE matrix. The



**Figure 5.** (a) Specific heat capacity as a function of filler content and uncertainties represent the standard error (b) Specific heat capacities with and without filler treatment.

characteristics in Figure 6(a,b) also indicate that, in the absence of coupling agent, the talc particles are mainly oriented along and well aligned with the direction of the injection molding; perpendicular to the fracture surface. No matrix residue on the talc surface is apparent, and pull-out of talc particles can be

seen in the imprints left on the surface. This can be interpreted as weak adhesion between the talc particles and the matrix. Bonding between talc and matrix is located at the edges of the talc; many cavities and micro-voids can be seen but no agglomerates are visible. However, treatment of the particle can to



**Figure 6.** SEM micrographs of the cryo-fractured surfaces of HDPE/talc at 35 wt-% (a and b) Untreated (c and d) Treated.

some extent affect the morphology of the HDPE/talc composites. As shown in Figure 6(c,d), the talc particle orientation is more random and many particles are even parallel to the fracture surface. Residues from the matrix on the particle surface can be seen; bonding between talc and matrix is located at both the surface and the edges of the particles, and fewer voids are apparent. Based on such evidences it can be inferred that adhesion between the particle and matrix had improved, as shown earlier.

It was found earlier (Figure 3) that there were fewer voids in the treated samples. Fewer voids might result in less phonon scattering and reduce thermal resistance, so the thermal conductivity would be expected to increase, which was not observed in the present study. Based on the SEM morphologies, this could be attributed to the more randomized orientation and uniform distribution of the particles in the presence of the titanate coupling agent resulting in fewer aligned heat-flow channels.

## CONCLUSIONS

The effects of the addition of a titanate coupling agent to talc filled high-density polyethylene compounds were investigated. All of the molded specimens showed an increase in tensile strength of up to 18% with the addition of the titanate coupling agent. The greatest improvement was seen in the stiffness, which could reach 82%, with treated particles. Elongation at break of composites decreased sharply when untreated talc at 5 wt % was introduced, but then no significant change was found with further increase in talc content. However, the treated composites showed significantly lower deformability than the ones with untreated filler. The coupling agent had a negligible effect on the thermo-physical properties such as thermal conductivity, thermal diffusivity, and specific heat capacity, although the void content decreased in the presence of the titanate coupling agent. Treatment of the HDPE/talc composites changed the morphology, distribution, and alignment of the particulates in the matrix.

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