



# The Effect of Size and Surface Treatment of Nucleating Agents on Polyamide 6 Morphology Studied by Flash Differential Scanning Calorimetry

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## Abstract:

The use of recycled polymer materials with low carbon footprint, which enables circular economy, is becoming increasingly interesting for commercial use in the world. The main reasons for this are the accumulation of waste after the use of polymer products and the warming of the atmosphere due to the overloading of the environment with greenhouse gases produced during the extraction of these materials. We were interested in the differences in properties between differently modified samples of thermoset waste in a thermoplastic matrix. Our main focus was on the influence of the modification of the interface on the stiffness and strength of the prepared material. The results of tensile and bending tests showed that with the right combination of compatibilizers, we were able to successfully increase the stiffness and strength of the composites. The simultaneous increase in stiffness and strength is a very good indicator that with the right combination of compatibilizers (polypropylene grafted maleic anhydride (PP-g-MA) and modified thermoplastic polyurethane (TPU) co-polymer) we were able to ensure good surface interaction between the fibres and the polymer matrix in these samples. Waste paper was added to the thermoplastic matrix as a reference. The difference in the performance of the composites can be explained by the better wettability of the waste paper with the SEBS-g-MA compatibilizer and the better interfacial interactions of the modified TPU copolymer with the thermoset and the PP-g-MA with the glass fibres in the case of the waste thermoset composite.

**Keywords:** waste thermoset, compatibilizer, thermoplastics, different fraction of waste thermosets, composites



## 1. Introduction

The recycling of plastic waste, especially post-consumer recycling (PCR), has begun and is still developing very slowly. Compared to other materials such as paper, glass and metals, recovery and recycling rates of plastics are generally low. Even in countries with advanced waste management systems and years of experience in recycling, the recycling rates of plastics are generally much lower than those of the other materials mentioned above. This is partly due to the wide variety of uses of plastics, types of additives and types of polymer composites. This diversity of uses is one of the main advantages of plastics, but it also poses a problem for recycling (Shen and Worrell, 2014).

The high melting point of polyamides is the result of strong hydrogen bonding in the crystals. This also enables stiffness above the glass transition temperature and almost up to the melting point. The effect is further enhanced when reinforcing materials such as glass fibres are added. The melting point is determined by the number of hydrogen bonds between the chains, which depends on the density of the amide groups. The melting temperature decreases with increasing length of the aliphatic chain between the amide groups. Polyamides crystallize quickly, which can be advantageous for small particles to further accelerate this process; this can shorten the cycle time and increase productivity. Nucleating agents are substances in the form of fine particles that accelerate the crystallization of polyamides. Nucleation increases tensile strength and stiffness, but makes the material more brittle (Wypych, 2016) (Palmer, 2000).

The glass transition temperature depends on the cold crystallization of polyamide 6 (PA6) and is influenced by the cooling conditions. The ratio between the rigid and the mobile amorphous fraction in PA6 is determined by the thermal history (Parodi et al., 2017).

Nucleating agents are additives used to increase the crystallization rate during processing by controlling the morphology of the polymers. For this reason, nucleating agents are often specific to a particular polymer or family of polymers and should be carefully selected depending on the desired change in physical properties. Nucleating agents at low cooling rates (10 K/min) cause a shift of crystallisation temperature ( $T_c$ ) to higher temperatures. The crystallization rate of PA6 is not affected by nucleating agents up to 90 °C, as the density of nuclei is very high at temperatures close to the glass transition temperature. In order to measure the effectiveness of nucleating agents, Flash differential scanning calorimetry (DSC) measurements must be carried out by rapidly cooling the melt to a crystallization temperature between the glass transition temperature ( $T_g$ ) and melting temperature ( $T_m$ ) (Mileva et al., 2012). The reported crystallization rate for PA6 is lower compared to PA66, which is due to the odd (PA6)/even (PA66) number of CH<sub>2</sub> groups (Poel, Istrate and Mathot, 2016).

With Flash DSC the fast-scan chip-calorimeter measurements in a broad temperature range were conducted, also in low temperature region. In the case of nylon, the crystallization rate showed a parabolic shape depending on the crystallisation temperature with a maximum between  $T_g$  and  $T_m$  (Li et al., 2020).

## 2. Methods & materials

Commercially available PA6 (Akulon K222-D) was purchased from DSM, Netherlands (PA6 in **Table 1**). Commercially available antioxidant (AT 10 – AO in **Table 1**) was purchased from AMIK ITALIA, Italy. Commercially available CaCO<sub>3</sub> was provided by Calcit, Slovenia. Trade names of CaCO<sub>3</sub> are Calplex Extra (NA<sub>01</sub> in **Table 1**), Calplex 5 (NA<sub>02</sub> in **Table 1**), Calplex 40 (NA<sub>03</sub> in **Table 1**), Polyplex Extra (NA<sub>11</sub> in **Table 1**), Polyplex 5 (NA<sub>12</sub> in **Table 1**) and Polyplex 40 (NA<sub>13</sub> in **Table 1**). Commercially available talc (Plustalc H15 – NA T in **Table 1**) was purchased from Elementis Minerals, Netherlands. Commercially available nucleating agent (Prisma AD PE Nucleant 91225 – NA AD in **Table 1**) was purchased from Frilvam, Italy and the only material in the pellet form.

The composition of the samples is listed in **Table 1**. For all tests, the materials were mixed separately and extruded on the Labtech LTE 20-44 twin screw extruder. The screws had a diameter of 20 mm, an L/D ratio of 44:1, a screw speed of 600 rpm and an increasing temperature profile from the hopper (245 °C) to the die (260 °C) (Pracella et al., 2010).



The first sample (Sample 0) was pure PA6, which we extruded and injection moulded. Then we produced a PA6 sample with 0.5 % antioxidant (Sample 1, with AO). Composites with 0.1 % calcium carbonate were produced in 6 different versions; in three the calcium carbonate was uncoated (Samples 2-4, with NA<sub>01</sub>, NA<sub>02</sub> and NA<sub>03</sub>, respectively) and in three it was coated with stearic acid (Samples 5-7, with NA<sub>11</sub>, NA<sub>12</sub> and NA<sub>13</sub>, respectively). We prepared two more versions: in one we added 0.1 % talc (Samples 8, with NA T) and in the last one we added 1 % nucleating agent based on polyethylene with 50 % talc (Samples 9, with NA AD). All composites contained 0.5 % antioxidant.

**Table 1.** Sample composition.

Sample	AO (%)	NA <sub>01</sub> (%)	NA <sub>02</sub> (%)	NA <sub>03</sub> (%)	NA <sub>11</sub> (%)	NA <sub>12</sub> (%)	NA <sub>13</sub> (%)	NA T (%)	NA AD (%)	PA6 (%)
0	0	0	0	0	0	0	0	0	0	100
1	0.5	0	0	0	0	0	0	0	0	99.5
2	0.5	0.1	0	0	0	0	0	0	0	99.4
3	0.5	0	0.1	0	0	0	0	0	0	99.4
4	0.5	0	0	0.1	0	0	0	0	0	99.4
5	0.5	0	0	0	0.1	0	0	0	0	99.4
6	0.5	0	0	0	0	0.1	0	0	0	99.4
7	0.5	0	0	0	0	0	0.1	0	0	99.4
8	0.5	0	0	0	0	0	0	0.1	0	99.4
9	0.5	0	0	0	0	0	0	0	1	98.5

The injection moulding was carried out on a Krauss Maffei 50-180 CX with a screw diameter of 30 mm. The temperature profile increased from the hopper (220 °C) to the nozzle (230 °C). The mould temperature was set to 80 °C and the cooling time to 20 s. Injection moulded pieces were shaped according to ISO 527 (for tensile tests and DMA) and ISO 179 (for impact test) (Jazani et al., 2011). The tensile tests were carried out with the Shimadzu AG-X plus in accordance with ISO 527-1 (Huang et al., 2013). Tensile modulus ( $E_t$ ), tensile strength ( $\sigma_m$ ), elongation at tensile strength ( $\epsilon_m$ ) and elongation at break ( $\epsilon_{tb}$ ) were evaluated in the tensile tests. Five measurements were carried out for each sample. The thermomechanical properties were analysed using a Perkin Elmer DMA 8000 (Wunderlich, 2005). The samples were heated at 2 °C/min from 25 °C to 210 °C in an air atmosphere. A frequency of 1 Hz and an amplitude of 20  $\mu$ m were used in dual cantilever mode (Jazani et al., 2011). Thermogravimetric analyses (TGA) were performed using a Mettler Toledo TGA/DSC 3+ thermal analyser. Analyses were carried out in a nitrogen atmosphere (20 mL/min) from 40 to 550 °C with a heating rate of 10 °C/min, followed by an isothermal segment in an oxygen atmosphere (20 mL/min) at 550 °C for 10 minutes using an Al crucible. Changes in the weight are evaluated as degradation in % and peak of the first derivative of the measured curve as degradation temperature. Thermal measurements were performed with a Differential Scanning Calorimeter (DSC 2, Mettler Toledo) in a nitrogen atmosphere (20 mL/min). The temperature of the samples was raised from 0 to 260 °C at a heating rate of 10 °C/min and held in the molten state for 5 minutes to clear the thermal history. After cooling at 10 °C/min, the samples were reheated at 10 °C/min to 260 °C.  $T_c$ , the crystallisation enthalpy ( $\Delta H_c$ ),  $T_m$  and the melting enthalpy ( $\Delta H_m$ ),  $T_g$  and the change in specific heat capacity ( $\Delta c_p$ ) were determined from the cooling and the second heating scan. The crystallinity ( $W_{c,h}$ ) was calculated from melting enthalpy, divided by the melting enthalpy of 100 % crystalline PA6 (Wunderlich, 2005).

Flash DSC 1 with Huber TC45 intercooler and nitrogen purge gas (50 mL/min) was used to preformed crystallization kinetics measurements. The test samples were heated (230 °C) and cooled (20 °C) three times at a high heating and cooling rate (2.000 °C/s) so that



most of the nuclei that formed during cooling from previous processing have melted. During the first heating, the sample melts and spreads over the entire sensor. During the second heating the thermal history is erased and during the third heating we obtain realistic results of the material. The sample is cooled to different temperatures and held there for 5 seconds. The temperatures at which the measurement stopped were between 55 °C and 180 °C in 5 °C increments. After the measurements were completed, we evaluated the rate of crystallization as an exothermic peak during this isothermal segment.

The impact tests were performed using the Dongguan Liyi Test Equipment pendulum, type LY-XJJD5 (Lin et al., 2011), according to ISO 179. The distance between the supports was 60 mm and a 5 J pendulum was used. Impact strength was determined as the quotient between the energy required to fracture the sample divided by the cross-section of the sample.

### 3. Results & Discussions

#### 3.1. Tensile tests

The results of the tensile tests are shown in **Table 2**. Sample 0 is the reference material, pure PA6, with a tensile modulus of 2.29 GPa, tensile strength of 70.0 MPa, elongation at tensile strength of 4.52 % and elongation at break of 114 %. In Sample 1, only the antioxidant was mixed into the matrix. The addition of the antioxidant reduced the tensile modulus  $E_t$  by 15.7 %, the tensile strength  $\sigma_m$  by 0.1 %, the elongation at break  $\epsilon_{tb}$  by 24.6 % and increased the elongation at tensile strength  $\epsilon_m$  by 3.5 % (**Table 2**).

Samples 2, 3 and 4 consisted of matrix, antioxidant and uncoated calcium carbonate, which differ in particle size. Sample 2 and 3 showed a decrease in tensile modulus  $E_t$  at tensile strength  $\sigma_m$  by 1.3 % and 6.1 %, respectively; Sample 4 showed an increase in tensile modulus  $E_t$  at tensile strength  $\sigma_m$  by 3.9 %. All three samples showed a decrease in tensile strength  $\sigma_m$  by 1.2 % for Sample 2, 2.7 % for sample 3 and 2.7 % for Sample 4. The elongation at tensile strength  $\epsilon_m$  did not change drastically. The elongation at break  $\epsilon_m$  decreased by 15.2 % for sample 2, 52.5 % for sample 3 and 42.1 % for sample 4 (**Table 2**).

Samples 5, 6 and 7 consist of a matrix, an antioxidant and coated calcium carbonate with different particle sizes. For all three samples, we observe a decrease in tensile modulus  $E_t$ , tensile strength  $\sigma_m$  and elongation at tensile strength  $\epsilon_m$  and an increase in elongation at break  $\epsilon_{tb}$ . Of these three samples, Sample 5, which contained Polyplex Extra with the smallest particles, achieved the highest stiffness, strength and toughness. It was as we expected: the smaller the particles, the better the effect of adding calcium carbonate (**Table 2**).

To improve the tensile modulus  $E_t$ , we need to use Calplex 40, which is uncoated and has larger particles than Calplex Extra and Calplex 5. Other calcium carbonates reduced the tensile modulus  $E_t$  (**Table 2**). All samples showed a reduction in tensile strength  $\sigma_m$  compared to neat PA6, but this was lowest for Calplex Extra, which is uncoated and contains the smallest particles. The Calplex additives are optimal for slight changes in tensile strength  $\sigma_m$ , which hardly change it at all; the Polyplex additives reduce it slightly (**Table 2**). Elongation at break  $\epsilon_{tb}$  increased the most with Polyplex 40, which contains larger particles and is coated.

Talc is added to Sample 8. Talc reduced the tensile modulus  $E_t$  by 3.5 % compared to pure PA6, while the tensile strength  $\sigma_m$  increased slightly (by 0.3 %). The elongation at tensile strength  $\epsilon_m$  decreased by 6.2 %; the elongation at break  $\epsilon_{tb}$  decreased by 71.6 % compared to the reference (**Table 2**).

A nucleating agent consisting of 50 % talc and 50 % LDPE reduced the tensile modulus  $E_t$  by 2.2 %, the tensile strength  $\sigma_m$  by 2.6 %, the elongation at tensile strength  $\epsilon_m$  by 1.6 % and the elongation at break  $\epsilon_{tb}$  by 33.1 % (**Table 2**).

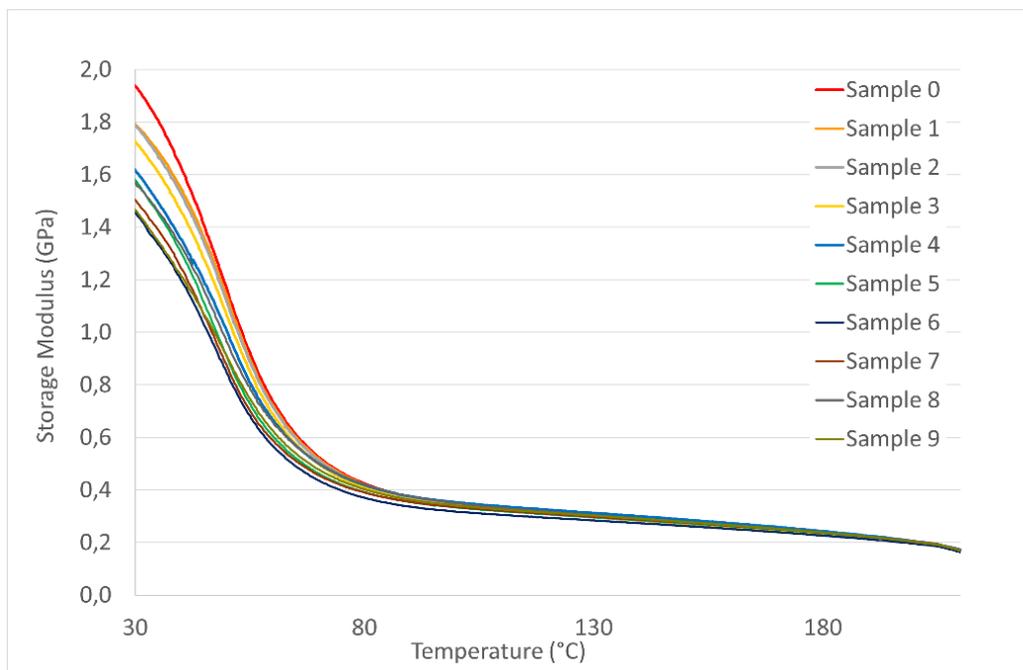


**Table 2.** Tensile tests results with recycled PP matrix.

Sample	E <sub>t</sub> (GPa)	σ <sub>m</sub> (MPa)	ε <sub>m</sub> (%)	ε <sub>tb</sub> (%)
0	2.29 ± 0.12	70.0 ± 1.1	4.52 ± 1.14	144 ± 36
1	1.93 ± 0.34	70.0 ± 0.5	4.68 ± 0.07	86 ± 57
2	2.26 ± 0.15	68.1 ± 0.2	4.52 ± 0.04	173 ± 46
3	2.15 ± 0.03	68.1 ± 0.6	4.52 ± 0.09	173 ± 41
4	2.38 ± 0.14	68.1 ± 0.6	4.51 ± 0.14	66 ± 42
5	2.25 ± 0.06	65.7 ± 0.4	4.39 ± 0.08	174 ± 52
6	2.01 ± 0.01	65.3 ± 0.5	4.47 ± 0.11	186 ± 0
7	2.02 ± 0.01	64.1 ± 1.2	4.46 ± 0.07	208 ± 37
8	2.21 ± 0.01	70.2 ± 0.4	4.24 ± 0.08	32 ± 9
9	2.24 ± 0.02	68.2 ± 0.5	4.45 ± 0.13	76 ± 22

3.2. Dynamic mechanical analysis tests

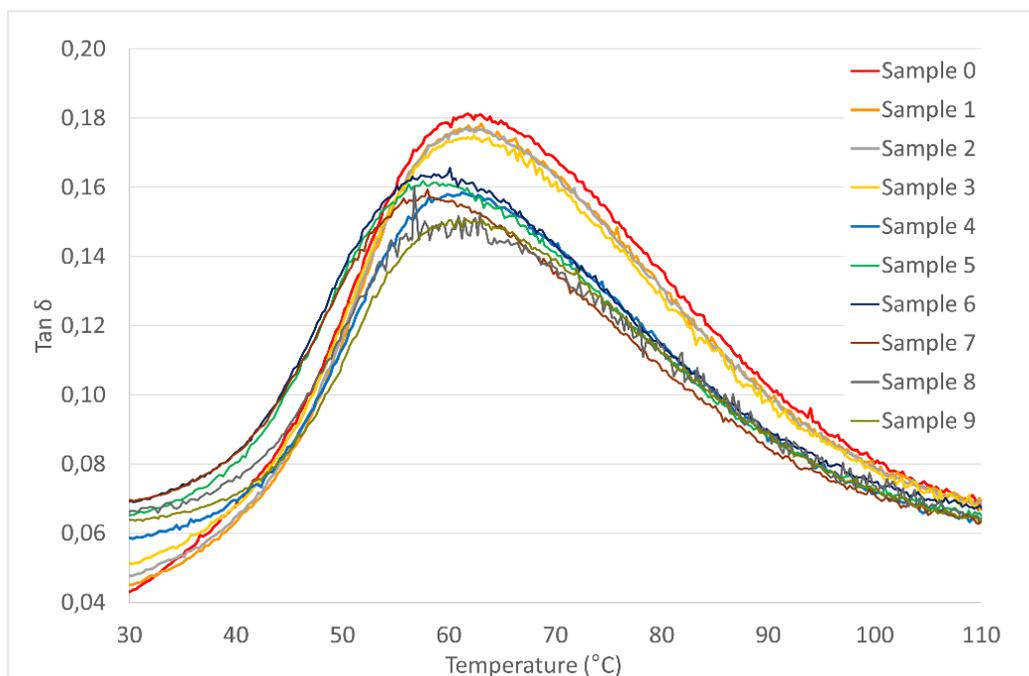
The storage modulus (**Figure 1**) of the samples agrees with the results of the tensile test, with the exception of Sample 4. Sample 0 has the highest storage modulus, while Sample 6 has the lowest storage modulus, measurement of stored energy representing the elastic proton. Uncoated calcium carbonate lowers the storage modulus, namely Calplex Extra by 7.7 %, Calplex 5 by 10.1 % and Calplex 40 by 16.6 %. Coated calcium carbonate reduces the storage modulus more, namely by 18.5 % for Polyplex Extra, 24.9 % for Polyplex 5 and 22.4 % for Polyplex 40. Talc lowers the storage modulus by 19.0 %, while the combination of talc and LDPE lowers it by 24.4 %. The curve is constantly decreasing, which means that we did not observe any cold crystallization during heating.



**Figure 1.** Storage modulus vs. temperature for the samples.



The peak of the loss factor curve (**Figure 2, Table 3**) indicates the glass transition. If the peak is lower, it means that the material has a higher elastic response, i.e. a higher degree of crystallinity. This can be observed in Sample 8, to which talc was added, and is consistent with the results of the tensile tests. The loss factor, quotient between viscous and elastic portion, is highest for Sample 0 and lowest for sample 9. Uncoated calcium carbonate lowers the loss factor. The smallest particle size (Calplex Extra) reduces the loss factor by 2.2 %, the medium size (Calplex 5) by 3.3 % and the largest size (Calplex 40) by 12.2 %. Coated calcium carbonate or Polyplex Extra, Polyplex 5 and Polyplex 40 reduce the loss factor by 10.5 %, 8.3 % and 12.2 % respectively. Talc reduces the loss factor by 11.6 % and the mixture of talc and LDPE by 16.6 %. From this we can conclude that the loss factor decreases with the addition of talc and calcium carbonate.



**Figure 2.** Loss factor (Tan  $\delta$  (-)) vs. temperature ( $^{\circ}\text{C}$ ) for the samples.

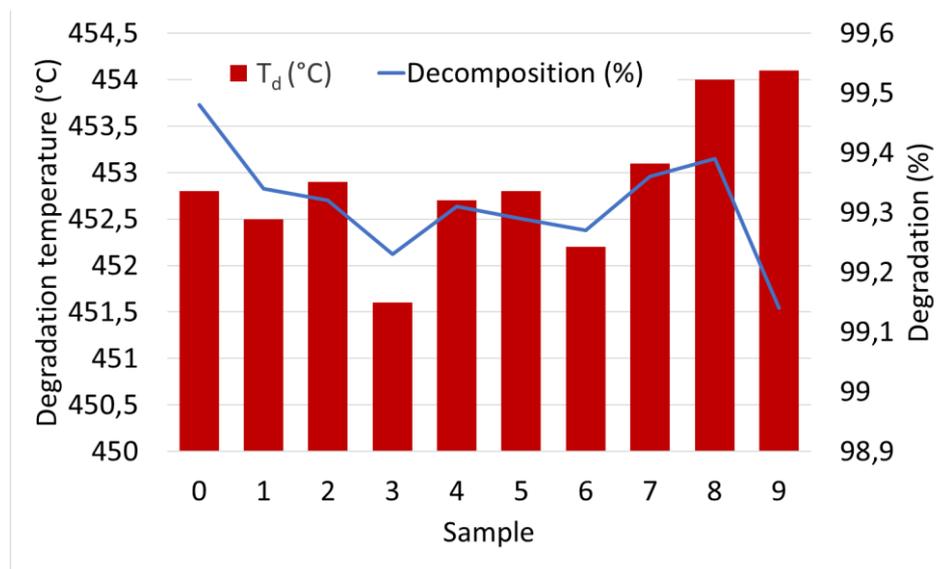
**Table 3.** Loss factor results at DMA measurements for the samples.

Sample	tan $\delta$ (-)	T <sub>g</sub> ( $^{\circ}\text{C}$ )
0	0.181	62.1
1	0.177	62.1
2	0.177	62.0
3	0.175	62.4
4	0.159	61.2
5	0.162	57.6
6	0.166	60.1
7	0.159	58.0
8	0.160	56.8
9	0.151	61.6



### 3.3. Thermogravimetric analysis tests

The degradation temperature of the samples varied within less than 0.3 % (**Figure 3**), which means that the additives we used did not drastically affect the degradation temperature. The inorganic filler content changed (**Figure 3**), which is completely logical as we have added inorganic fillers. Pure PA6 has 0.04 % inorganic fillers. The addition of an antioxidant increases the proportion of inorganic fillers to 0.08 %. In Samples 2 to 6, the inorganic fillers were 0.1 % - as much as we have added calcium carbonate. Sample 7 had only 0.04 % inorganic fillers. Sample 8 had 0.07 % inorganic fillers. Sample 9 had 0.34 % inorganic fillers, which means that some of the nucleating agent, which is low density polyethylene (LDPE), has degraded. Since the scattering of the results are smaller than the amount of inorganic residues in neat PA6 we can conclude that the distribution of the fillers is homogeneous and constant.



**Figure 3.** Degradation temperature and degradation amount at TGA measurements for the samples.

### 3.4. DSC tests

The results of the DSC analysis are shown in **Table 4**. The melting temperature  $T_m$  of sample 0 is at 220.1 °C. Most of the additives had no drastic influence on the melting temperature. The glass transition temperature  $T_g$  did not change drastically due to the additives (less than 2 % with the exception of Calplex 5, which lowered it by 3.7 %). In theory, it was stated that nucleating agents have no effect on the melting temperature (Poel, Istrate and Mathot, 2016), which is consistent with our results.

The melting enthalpy ( $\Delta H_m$ ) was most strongly affected by talc, which increased it by 5.2 % (**Table 4**, Sample 8). This was followed by Sample 9, where  $\Delta H_m$  was 4.4 % higher than that of pure PA6.  $\Delta H_m$  was reduced only by antioxidants (by 2.3 %) and Calplex 40 (by 2.6 %); all other additives increased it by between 0.8 % (Calplex 5) and 5.2 % (talc) (**Table 4**, Samples 3 and 8, respectively).

The degree of crystallinity of pure PA6 is 31.5 %. The addition of antioxidants reduced it by 1.8 %. Calplex Extra increased it by 4.2 %, Calplex 5 by 1.4 % and Calplex 40 reduced it by 2.1 % (**Table 4**). This indicates that smaller particles are more effective because they have a larger surface area in the case of uncoated calcium carbonate. The addition of Polyplex Extra increased the degree of crystallinity by 2.0 %, Polyplex 5 by 3.5 % and Polyplex 40 by 4.0 %. With coated calcium carbonate it is exactly the opposite as with uncoated calcium carbonate. Larger particles achieve a higher degree of crystallinity. The most optimal inorganic filler for increasing the degree of crystallinity is talc. It increases it by 5.9 %, while the combination of talc and LDPE increases it by 4.9 %.



The crystallization temperature is an indicator of heterogeneous crystallization, which is modelled by the addition of nucleating agents. Sample 8 has the highest crystallization temperature, followed by sample 2, which is consistent with the tensile tests and the DMA results.

**Table 4.** DSC tests results for the samples.

Sample	T <sub>g</sub> (°C)	T <sub>m</sub> (°C)	ΔH <sub>m</sub> (J/g)	W <sub>ch</sub> (%)	T <sub>c</sub> (°C)
0	55.5	220.1	72.5	31.5	191.7
1	54.9	220.8	70.8	31.0	191.4
2	54.5	220.0	75.1	32.9	192.1
3	53.5	220.7	73.1	32.0	191.5
4	54.3	220.4	70.6	30.9	191.8
5	54.6	220.3	73.5	32.1	191.8
6	55.3	220.7	74.6	32.6	191.5
7	55.1	220.6	75.0	32.8	191.5
8	54.4	220.5	76.3	33.4	193.0
9	54.6	220.0	75.7	33.1	191.9

### 3.5. Flash DSC tests

The Flash DSC analysis (**Figure 4**) shows the peak temperature of the crystallization and the crystallization rate. The highest is peak temperature, the fastest is the crystallization. The height of the graph represents the rate of crystallization. If the graph is higher, it means that the material crystallizes faster. All materials were tested under the same conditions. If the peak of the crystallization curve is shifted towards higher temperatures, this means that smaller crystals are formed (Poel, Istrate and Mathot, 2016). This gives the material greater transparency and toughness (Poel, Istrate and Mathot, 2016).

Sample 8, where talc was added into PA6 matrix, crystallized the fastest. From this we can conclude that Sample 8 had the highest degree of crystallinity, which was confirmed by our DSC analysis results (**Table 4**). Sample 2 crystallized fastest at a higher temperature, which means that smaller crystals were formed and it was more transparent after injection moulding. From this we can conclude that Calplex Extra provides nuclei for crystallization because it increases the crystallization temperature.

For calcium carbonate, Calplex Extra proved the best product because it is uncoated and contains the smallest particles. It increased the degree of crystallinity, the rate of crystallization when cooling PA6 and the temperature at which the material crystallizes fastest. Stiffness increased and strength was reduced. Coated calcium carbonate also increased the degree of crystallinity, but the other properties were not improved as much. With sufficiently small nucleating agents, no steric hindrances occurred, which is why the degree of crystallinity also increased.

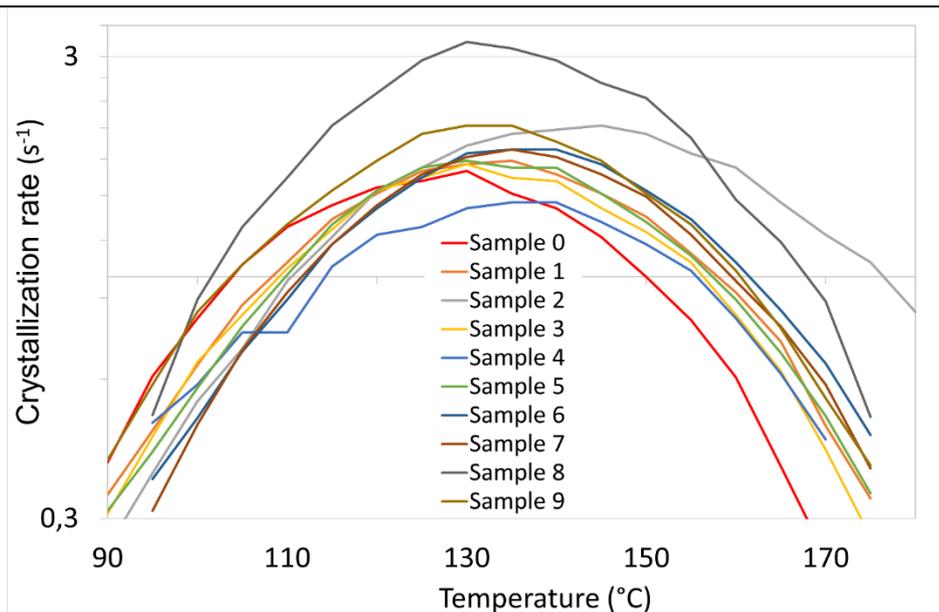


Figure 4. Flash DSC crystallization rate evaluation for the samples.

### 3.6. Impact tests

Figure 5 presents the impact strength of the samples. The impact strength of Sample 0 was 114 kJ/m<sup>2</sup>; the sample did not crack. The impact strength decreased with all additives except talc. Antioxidant decreased the impact strength by 5.5 %; Calplex Extra decreased it by 7.0 %; Calplex 5 decreased it by 3.3 %; Calplex 40 decreased it by 9.0 %. Polyplex Extra had the greatest effect on impact strength, reducing it by 17.1 %. Polyplex 5 reduced it by 5.4 % and Polyplex 40 by 2.0 %. Talc was the only agent that increased impact strength by 0.6 %. The mixture of talc and LDPE reduced the impact strength by 1.6 %. All additives that act as nucleating agents (except talc) reduced the impact strength.

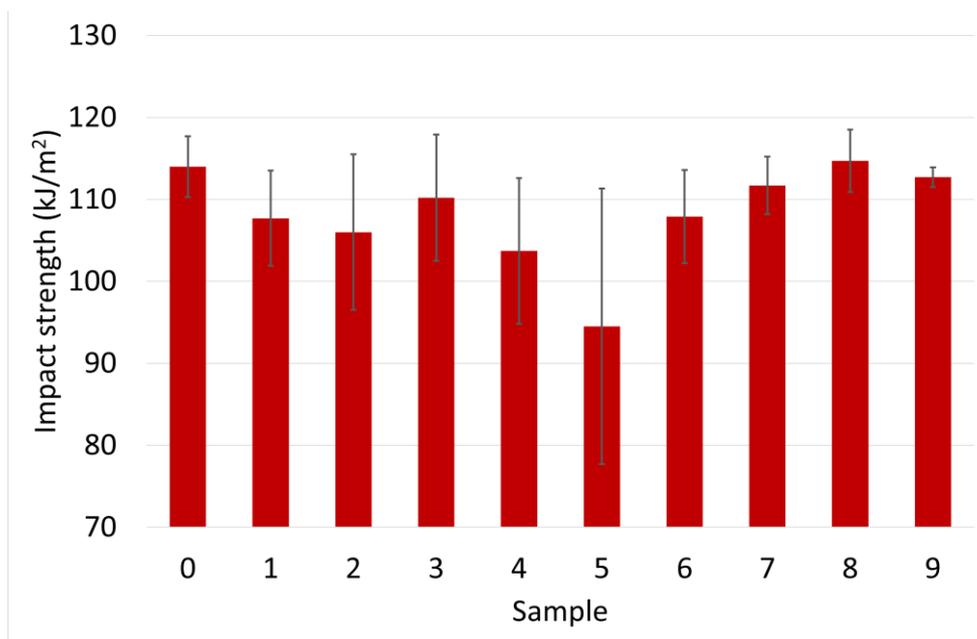


Figure 5. Impact strength for the samples.



#### 4. Conclusions

Generally, it was observed that the type, size, and surface treatment of the nucleating agents influenced the morphology behaviour of PA6. Among them, NA<sub>01</sub> was the best, as it increased the degree of crystallinity the most. The degree of crystallinity increased with all variants of added calcium carbonate, except for NA<sub>03</sub>. Uncoated particles larger than 90 µm already hinder crystallization. The highest crystallization temperature was achieved with Calplex Extra, which increased productivity. The difference between coated and uncoated calcium carbonate was large. Our results showed that it is better to use smaller particles as they have a larger specific surface area. The addition of calcium carbonate lowered the tensile modulus and tensile strength of PA6. The addition of coated calcium carbonate increased the elongation at break, while the addition of uncoated calcium carbonate decreased it. The impact toughness was reduced the most by Polyplex Extra and the least by Polyplex 40. The inorganic filler that increases the degree of crystallinity of PA6 the most was talc (Sample 8). It increased it by almost 6 % without changing the melting point of PA6. PA6 with added talc is also characterised by its crystallization rate, but crystallizes at a low temperature (Sample 8), compared to other tested samples. Talc lowered the tensile modulus and the elongation at break and slightly increased the tensile strength of PA6. Of the versions of calcium carbonate tested, Calplex Extra proved to be the most effective nucleating agent. Although talc increased the degree of crystallinity most successfully, Calplex Extra achieved the highest crystallization temperature. For further investigations, we would recommend determining the optimum concentration of calcium carbonate. The effect of the particle size and concentration of the nucleating agent should be further investigated. Based on the results of our study, we would suggest an investigation in the direction of even smaller particle size of nucleating agents. From the present study it is evident, that too high concentration has no positive effect on the degree of crystallinity. The initial concentration would be 0.1 %; it would be increased in steps of 0.1 %; the final concentration would be 1.0 %.

**Conflicts of Interest:** The authors declare no conflict of interest.

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