



Reprocessing of High-Density Polyethylene Reinforced with Carbon Nanotubes

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Abstract

High-density polyethylene (HDPE) was compounded with 3 wt% carbon nanotubes (CNTs). In order to simulate mechanical recycling, both the nanocomposite and neat HDPE were repeatedly extruded and subsequently analysed by tensile tests, Charpy impact strength, differential scanning calorimetry (DSC), oxidation induction time (OIT), Gel Performance Chromatography (GPC), Fourier Transform Infrared Spectroscopy (FTIR) and TEM. After 10 cycles of extrusion, thermal, mechanical, and rheological tests did not reveal any significant degradation. In order to better study the effect of the CNTs, a large number of cycles were simulated by processing the materials for up to 200 min. After 200 min of processing, the neat HDPE was significantly degraded whereas the nanocomposite was almost unaffected.

Keywords Nanocomposite · Recycling · Repeated processing · Carbon nanotubes · HDPE

Introduction

High-density polyethylene (HDPE) is one of the most commonly used plastics, with a wide range of applications. Mechanical recycling of HDPE has therefore been studied in detail [1–6]. The number of times that HDPE can be recycled can vary depending on the processing conditions and the stabilization of the polymer, but, generally speaking, HDPE is relatively stable to degradation during repeated processing. For example, Oblak et al. processed HDPE through 100 extrusion cycles [4]. When the recycled polymer was characterised with nanoindentation, the hardness was almost unaffected after 10 extrusion cycles, and after 100 cycles the hardness was reduced to about 20% of its initial value. Repeated processing of HDPE has also been studied for HDPE filled with natural fibres [7], wood flour [8], and glass fibres [9].

Carbon nanotubes (CNTs) have been extensively investigated over the past few decades. CNTs were first reported by Iijima [10] and can be conceptually understood as a sheet of graphene rolled into a tube. CNTs have low density, high aspect ratio and have very strong mechanical properties

[11]. CNTs are therefore excellent candidates to reinforce plastics. CNTs are also electrical conductive and may be used for antistatic packaging. HDPE has been reinforced with CNTs in several studies. Adewunmi et al. studied the rheological properties of HDPE reinforced with up to 7 wt% CNTs [12]. Tang, Santare and Advani reinforced HDPE with multiwall CNTs (MWCNTs) [13]. Films were produced with up to 5 wt% CNTs were produced. Increased mechanical properties were observed with increasing loading of the CNTs. Kanagarai et al. Studied the mechanical properties of HDPE reinforced with CNTs up to loadings of 0.44 vol% [14]. The Young's modulus increased up to 22%. Zou et al. studied the dispersion of CNTs in HDPE by extrusion [15]. A better dispersion was found at higher screw speeds.

Considering the extensive research that has been carried out in the field of CNTs, it is reasonable to assume that consumer goods produced from nanocomposites will start to enter the market. Consequently, the stream of nanocomposite waste will increase and the importance of studying recycling of these materials is therefore vital. In addition, CNTs are still relatively expensive materials and there is an incentive to recycle them. Mechanical recycling of nanocomposites has not been studied thoroughly, and previous research has mainly focused on the recycling of polymers reinforced with nanoclay [16–19]. La Mantia et al. studied the reprocessing of low-density polyethylene (LDPE) reinforced with an organically modified nanoclay [20]. They found a relatively

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complex degradation pattern depending on the processing conditions, but that the nanocomposite could still be recycled and used relatively well even after four rounds of reprocessing. The mechanical recycling of polypropylene (PP) reinforced with CNTs has also been studied [21]. Repeated injection moulding was studied for PP compounded with 3 wt% MWCNTs. The nanocomposite was recycled 20 times, and after the last cycle tensile tests did not reveal any significant changes. However, the melt flow index increased for both neat PP and PP filled with MWCNTs.

The purpose of this work was to study the repeated processing of HDPE reinforced with CNTs. Mechanical recycling of plastics reinforced with CNTs has not been studied extensively, and to our knowledge recycling of HDPE/CNT has not been studied previously. HDPE was compounded with 3 wt.% CNT and the resulting nanocomposite was first extruded and ground 10 times. In order to simulate a large number of rounds of recycling, the nanocomposite was processed continuously for up to 200 min. The effect of the reprocessing was evaluated using mechanical, rheological, and spectroscopic tests.

Experimental

Materials

Masterbatch (Plasticyl HDPE1501), consisting of 15 wt% nanotubes compounded with HDPE, was kindly provided by Belgium Nanocyl SA. The masterbatch contains MWCNTs (NANOCYL® NC7000) produced via the Catalytic Chemical Vapor Deposition (CCVD) process with an average diameter of 9.5 nm. HDPE (Purell GA7760) was obtained from LyondellBasell. This is an injection moulding grade with a melt flow rate of 18 g/10 min (190 °C/2.16 kg).

Processing

Nanocomposite with 3 wt% MWCNT was prepared by compounding HDPE with the masterbatch using a 15-ml twin-screw microcompounder (DSM Netherlands). The compounding was done at 200 °C, and at 70 rpm for 5 min.

The repeated processing of the prepared nanocomposite was studied on the same instrument. The material was extruded at a temperature of 170 °C, and at 4 rpm. Material was taken out after the extrusion, and the rest of the material was ground and extruded again.

Test bodies were prepared from the material after the extrusion by injection moulding on the same instrument. The material was first processed at 170 °C, and at 70 rpm for

2 min, and then transferred to a laboratory injection moulder from the same company with a mould temperature of 20 °C.

A large number of reprocessing cycles were simulated by processing the material for a long time. The nanocomposite was injected into the microcompounder and processed (170 °C, 70 prm) for 20 min, 100 min, and 200 min, simulating 10, 50, and 100 extrusions, respectively. After the continuous processing, the material was injection moulded as described above.

Characterisation

The injection moulded test bodies were characterised using tensile tests according to ISO 527. A tensile tester from Tinius Olsen was used and at least five test bodies, each 75 mm long, were tested. The modulus was determined at 1 mm/min using a load cell of 250 N and the tensile strength was determined at 20 mm/min using a load cell of 5000 N.

Charpy impact tests were performed using a QC-639L instrument from Comotech Testing Machines Co. Ltd. (Taiwan). Notched samples were tested edge-wise with a pendulum of 1 J.

The thermal properties were analysed using DSC (Q2000; TA Instruments, USA). Samples were heated – 40 to 200 °C in sealed aluminium pans. Tests were performed in an environment of nitrogen gas at a heating rate of 10 mm/min. Heat of fusion (ΔH) and the peak of the melting (T_M) were recorded. Three samples were analysed, the values averaged, and the standard deviation calculated. Oxidation induction temperature (OIT) was determined on the same instrument. Samples were heated from 30 to 200 °C at a heating rate of 20 °C/min and then kept isothermal. When 200 °C had been reached, the gas flow of 50 ml/min nitrogen was switched to 50 ml/min of oxygen gas. Samples weighed 3.0–4.5 mg. Each sample was replicated 3 times.

Samples were also characterised by Fourier transform infrared spectroscopy (FTIR) using a Nicolote equipped with an ATR. They were measured with 64 scans.

Gel performance chromatography (GPC) was performed by an external laboratory. The tests were performed using Agilent PL GPC220 using 1,2,4-trichlorobenzene as a solvent. The characterisation by GPC was carried out at a temperature of 160 °C using a column from Agilent Technologies (PLgel olexis guard with 13 μm particle size). Refractive index was used as detector. The GPC instrument was calibrated with polystyrene as a reference.

TEM micrographs were prepared by an external company, using a 100-kV transmission electron microscope from Zeiss. Samples were analysed after 0 min and 200 min of processing.

Results

The results of the tensile testing are shown in Table 1. The neat HDPE had a tensile strength of 27 MPa and addition of CNTs to the polymer increased the tensile strength to 35 MPa, showing the reinforcing effect of the CNTs. This corresponds to an increase of 30%. As expected, due to the stiff nature of the MWCNTs, there was a significant reduction in the elongation when the CNTs were added to the polymer matrix.

After 10 cycles of extrusion, there was no significant sign of polymer degradation for the neat HDPE. Polyethylene is generally relatively stable against repeated processing [4] so this result is expected. However, although not significant, a minor increase in tensile strength was noticed which could possibly indicate on a better alignment of the polymer chains as a function of increased chain mobility during processing. The properties of the nanocomposite were also stable over the 10 cycles. Tensile strength and modulus were both roughly constant. This is in agreement with the findings of Zhang et al., who studied reprocessing of polypropylene (PP) filled with 3 wt% CNTs [21]. The tensile properties were relatively unaffected after 20 cycles of repeated injection moulding.

However, a small reduction of the elongation after 10 cycles can be discerned for the nanocomposite. A simple one-factor ANOVA with a significance level of 0.05 results in a p-value of 0.00072 indicate a decrease in elongation after 10 cycles. Although the ANOVA test for the neat HDPE also indicate on a difference between the population means (p-value = 0.037) the large standard deviations obtained provide little support for a change in elongation after 10 cycles. The reduction in elongation for the nanocomposite could be related to the reduced chain length with fewer entanglements of the polymer chains [21].

The stability of the nanocomposite tensile strength and modulus can be due to two competing mechanisms. First, the polymer is, as already stated, relatively stable against repeated processing and only a very minor reduction of mechanical properties would be expected under the chosen

experimental conditions. Secondly, an increase in tensile strength and modulus with repeated processing would be expected due to a slightly better dispersion of the nanotubes and increased adsorption of the polymer molecules on the surface of the nanotubes. However, at loadings of above 1% it has been shown that carbon nanotubes tend to agglomerate forming points of high stress concentration and loss of strength [22]. Consequently, the material shows little change in mechanical properties with repeated processing.

The results of the Charpy impact strength tests are shown in Fig. 1. Addition of CNTs to the polymer did not improve the Charpy impact strength. Ghoshal et al. reinforced PP with MWCNTs [23]. At a low concentration of MWCNTs (0.1%), there was a slight increase in impact strength, but at 1% there was a reduction in impact strength. However, when the interphase between the MWCNTs and the matrix was improved by functionalising the MWCNTs, the impact strength was significantly improved (152%). Thus, it is likely that the adhesion between the polymer matrix and the CNTs in this study was too low to be able see an improvement in the impact strength. Due to the low adhesion and the relatively high amounts of CNTs used, it is most likely that points of stress concentration have been created where agglomerates of CNTs are formed. This can explain the decrease in impact strength achieved.

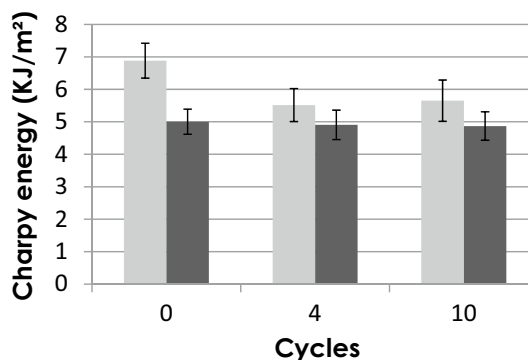


Fig. 1 Charpy impact strength for neat HDPE (light grey) and for HDPE/CNT (dark grey) as a function of processing cycle

Table 1 Tensile properties of the neat HDPE and HDPE reinforced with CNTs

Cycle	Tensile strength (MPa)	Elongation (%)	Modulus (GPa)	Tensile strength (MPa)	Elongation (%)	Modulus (GPa)
	Neat HDPE			HDPE/CNT		
0	27.3 (0.2)	792.3 (86.1)	1.1 (0.3)	34.6 (0.3)	38.2 (8.0)	1.7 (0.1)
2	27.8 (0.5)	914.2 (215.9)	1.7 (0.3)	33.7 (2.6)	50.4 (12.6)	1.8 (0.1)
4	27.6 (0.1)	770.9 (18.5)	1.2 (0.1)	35.7 (0.5)	37.0 (7.8)	1.6 (0.1)
6	27.8 (0.2)	863.8 (189.0)	1.2 (0.3)	35.6 (0.5)	42.5 (2.6)	1.5 (0.1)
8	27.9 (0.3)	785.2 (131.2)	1.4 (0.1)	35.5 (0.3)	41.3 (2.9)	1.4 (0.1)
10	28.9 (0.5)	545.9 (309.8)	1.4 (0.0)	36.0 (0.4)	29.0 (3.1)	1.5 (0.1)

For the neat HDPE, a minor reduction in the Charpy impact strength could be discerned during the reprocessing. The nanocomposite showed insignificant changes in impact strength after 10 cycles which, similar to above, can be a function of the competition between dispersion and agglomeration of the CNTs.

The results of the characterisation by DSC are given in Table 2. After 10 cycles of processing, no significant changes could be detected. Both the heat of fusion and the T_M were stable, which indicates that very little degradation had occurred after the 10 cycles. This is in agreement with the findings of Abad et al., who studied the reprocessing of both HDPE and low-density polyethylene [1]. After five cycles of extrusion, there was no significant change in crystallinity.

The viscosity was also characterised by GPC, and the results are shown in Table 3. Addition of CNTs to the polymer matrix increased the viscosity somewhat, but, and as can be seen, there was no significant change in the viscosity after 10 cycles of extrusion. Mn, Mw, and the polydispersity were roughly constant after 10 cycles, and this applies to both the neat HDPE and the nanocomposite. This is in line with the mechanical and thermal characterisation.

The results from both DSC and GPC indicate that very little degradation of the polymer chains have occurred. This is in agreement with previous studies. Furthermore, the short residence times (2 min) and the relatively low processing temperature do not result in harsh enough conditions for detectable degradation to occur. In the case for the nanocomposite, and as discussed below, an additional protective effect from the presence of the nanotubes can also be discerned and is more evident when longer residence times are used.

In summary, the first part of the study, where real processing cycles were performed, did not reveal any significant polymer degradation. The thermal, rheological, and mechanical properties were relatively unaffected by the 10 cycles of processing. This shows that, from an industrial point of view, it should be possible to recover industrial waste from a nanocomposite by mechanical recycling multiple times. From an academic point of view, however, it is interesting

Table 2 Characterisation by DSC. Standard deviations are given within parentheses

Material	Cycle	T_M (°C)	ΔH (J/g)
Neat HDPE	0	132.9 (± 0.4)	218.0 (± 4.2)
	4	133.5 (± 0.3)	214.8 (± 2.1)
	10	133.7 (± 0.2)	216.7 (± 4.5)
HDPE/CNT	0	133.6 (± 0.2)	208.6 (± 4.9)
	4	133.8 (± 0.2)	206.6 (± 5.2)
	10	133.5 (± 0.4)	214.1 (± 1.1)

Table 3 Characterisation by GPC

Sample	Cycle	Mw (g/mol)	Mn (g/mol)	Polydispersity
Neat HDPE	0	61,950	8870	7.0
	4	61,950	9125	6.8
	10	62,200	8700	7.2
HDPE/CNT	0	67,200	8835	7.6
	4	63,350	8525	7.4
	10	64,900	8405	7.7

to study how and when the CNTs affect the properties when the nanocomposite is recycled several times. Thus, in order to better understand the effect of the CNTs, a larger number of processing cycles were simulated in the second step of this study. This was done by processing the material continuously for up to 200 min. With a processing time of 2 min for one cycle, this would correspond to 100 processing cycles. The results of the characterisation with GPC are shown in Fig. 2. Neat HDPE is relatively stable after 10 min of processing which is in agreement with the results from the first part of this study. After 100 min of processing, the viscosity is clearly reduced and after 200 min of processing, the viscosity is reduced by roughly 50%. This shows clearly that the polymer was degraded. As for the nanocomposite, even after 200 min of continuous processing, the viscosity of the nanocomposites was not affected. This shows the remarkable protective effect of the CNTs. Studies on the thermal stability of CNT nanocomposites have previously focused on thermogravimetric analysis (TGA). For example,

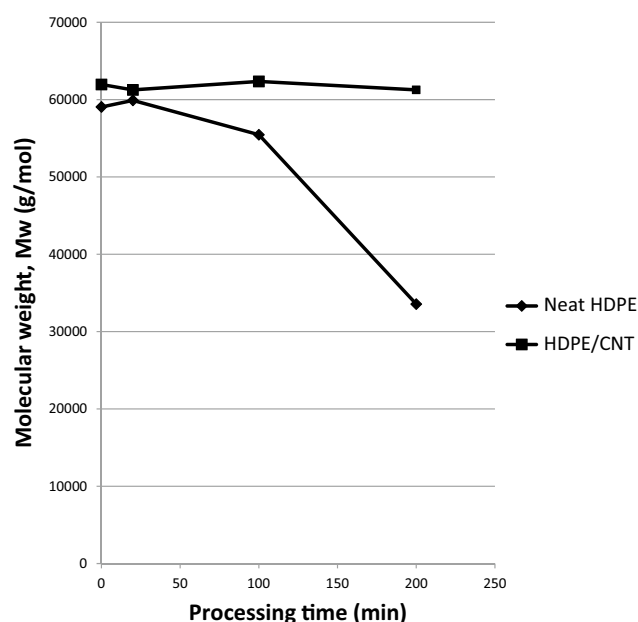


Fig. 2 Molecular weight, Mw, determined by GPC as a function of the processing time

Bikiaris et al. reinforced PP with 2.5 wt% MWCNT [24]. By analysing the materials with thermogravimetric analysis in an atmosphere of oxygen, a clear difference could be seen between the neat PP and the nanocomposite. Addition of the CNTs to the matrix increased the maximum degradation temperature by almost 20 °C. It has been suggested that the increased thermal stability is caused by well-dispersed nanotubes that hinder transportation of degradation products [25]. In another study, Yang et al. reinforced atactic PP with MWCNTs. When they analysed the thermal stability with TGA in an atmosphere of nitrogen, the maximum degradation temperature increased by 70 °C when 5 wt% CNTs were added to the polymer matrix. These authors suggested that the increased thermal stability was caused by physical adsorption of the polymer molecules on the surface of the nanotubes [26].

In previous studies [27] it has also been shown that inclusion of nanotubes and the resulting physical adsorption of the polymer chains lead to a more structured polymer matrix in the vicinity of the nanotubes. Although not crystalline in its nature, this ordered structure with its decreased mobility and lowered permeability of both radicals as well as oxygen and water could be one reason for the increased stability that was obtained during processing.

The neat HDPE and HDPE reinforced with CNTs were characterised by DSC and the results are summarised in Table 4. Despite the fact that the material was processed for a long time, there were only minor differences in heat of fusion and melting point. For example, the neat HDPE had a T_M of 132.9 °C, which was reduced to 131.4 °C after 200 min of processing. There were very minor changes for the nanocomposite also. Characterisation was also done by OIT, and the results are shown in Fig. 3. OIT is a common method of evaluating the long-term stability of polyolefins. It is a relatively straight forward method that is used to evaluate the antioxidant performance of PE [28]. The neat HDPE had an OIT of 24 min and after 200 min of processing, the OIT was reduced to 19 min. Concerning the nanocomposite,

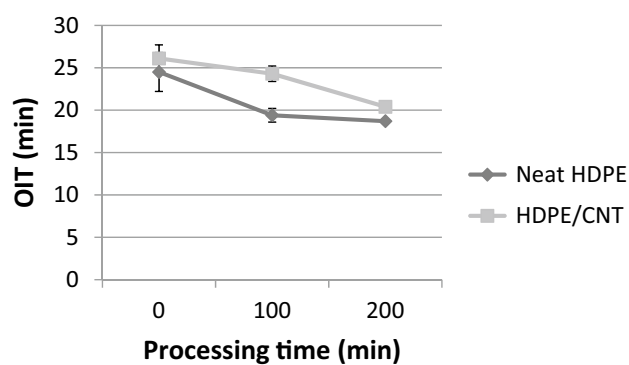


Fig. 3 OIT as a function of the processing time

the OIT started at 26 min, and after 200 min of processing, the OIT was reduced to 20 min. The OIT showed that the addition of the CNTs to the polymer matrix improved the thermal stability.

FTIR was also used in order to characterise the materials before and after 200 min of processing (Fig. 4). For the neat HDPE, a relatively large peak was formed at about 1700 cm^{-1} after 200 min of processing. This is a clear sign of oxidation, as carbonyl groups have been formed. Peaks could also be seen at 1120 cm^{-1} . Abad et al. studied repeated processing of both low-density polyethylene and HDPE [1]. After five cycles of extrusion, oxidation could be detected as peaks at 1760 cm^{-1} and 1120 cm^{-1} . The latter peak was attributed to stretching of the C–O–C of the ester group, which would indicate chain scission. In comparison, the nanocomposite was significantly more stable and there were no signs of oxidation. This is in agreement with the results of the GPC test.

Figure 5 shows the TEM images after 0 min and 200 min of processing. With processing of the material for a long time, one could possibly expect better dispersion and a shorter aspect ratio of the nanotubes. As can be seen in Fig. 5, there were nanotubes that were well-dispersed and also agglomerations. The images before and after processing were similar, which would indicate that there were no major changes. One possible explanation for the lack of increased dispersion would be that the nanotubes re-agglomerated during the processing.

Conclusions

Repeated processing of HDPE reinforced with MWCNTs was studied. In the first part of the study, the nanocomposite was extruded repeatedly 10 times. Very little change in thermal and mechanical properties could be seen. As for the mechanical tests, the tensile strength did not change significantly while a certain reduction in Charpy impact strength

Table 4 Thermal characterisation by DSC after the continuous processing

Material	Processing time (min)	T_M (°C)	ΔH (J/g)
Neat HDPE	0	132.9 (0.4)	218.0 (4.2)
	20	133.1 (0.1)	230.9 (9.3)
	100	133.0 (0.5)	214.9 (3.3)
	200	131.4 (0.0)	220.2 (6.6)
HDPE/CNT	0	133.6 (0.2)	209.6 (4.9)
	20	132.9 (0.3)	216.3 (5.8)
	100	133.1 (0.6)	202.9 (10.1)
	200	132.6 (0.5)	210.4 (7.1)

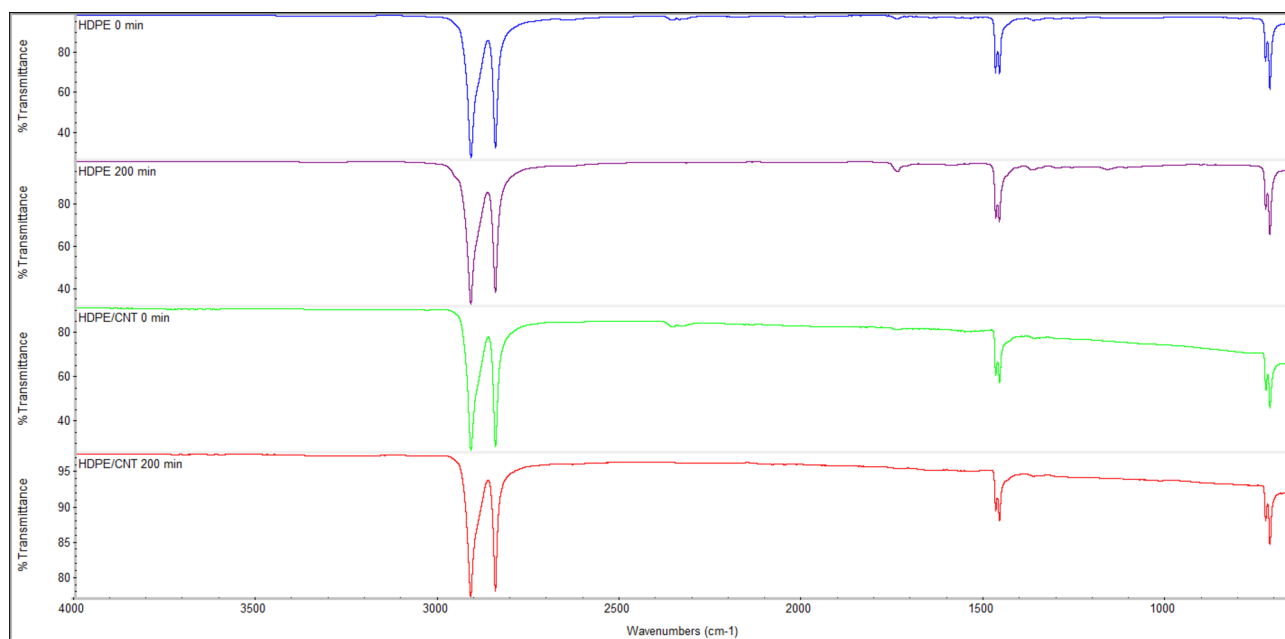


Fig. 4 Characterisation by FTIR after the continuous processing. From the top: neat HDPE after 0 min, neat HDPE after 200 min, HDPE reinforced with CNT after 0 min, and HDPE reinforced with CNT after 200 min

Fig. 5 TEM micrographs after processing for 0 min (left) and 200 min (right)



could be seen for the neat HDPE. Further characterisation by DSC did not reveal any significant change. This shows that, based on these tests, it should be possible to recover HDPE reinforced by CNTs by mechanical recycling.

Tests were also done where the material was continuously processed for up to 200 min. This showed that the neat HDPE could sustain this relatively long processing with no significant reduction of ΔH and T_M . Characterisations by OIT tests were also done. A certain reduction in OIT was recorded for both the neat HDPE as well as for the nanocomposite. However, when the materials were characterised by GPC, a clear difference between the neat and the reinforced polymer could be seen. A clear reduction in molecular weight could be seen

for neat HDPE. However, when the CNTs were added to the polymer matrix, the molecular weight was almost unaffected after 200 min of processing. This demonstrates the quite remarkable protective effect of CNTs against thermo-oxidative degradation.

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