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APPLICATION NOTE

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Producing high-performance polymer-composites by embedding nanoparticles using twin screw extrusion

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Key words
Compounding, Carbon nanotubes, Composites, Twin screw extrusion

Introduction
Carbon nanotubes are graphite sheets rolled into seamless tubes, with diameters of just a few nanometers and lengths up to centimeters. Nanotubes have received much attention because of their unique mechanical, electrical and thermal properties.

There are a large number of potential applications for CNTs, especially in the field of polymer compounds, where they are used to improve mechanical and electrical properties. Polymer nanocomposites are frequently used in the automotive and aviation industries, as well as in construction materials for windmill blades.

Key to unleashing the unique properties of the polymer nanocomposites is dispersion of the CNTs thoroughly in the polymer matrix. Only when the CNT particles are dispersed homogeneously within the polymer and the formation of larger clusters is avoided, can the desired property improvements be achieved. The improved mechanical properties of the final compound can be tested by means of dynamic mechanical thermal analysis (DMTA) which can be performed, for instance, with a rotational rheometer [1].

One approach that can lead to a homogeneous distribution of the CNT particles within the polymer matrix is the use of CNT suspensions for the extrusion process. For this the CNTs are functionalized first (i.e. by amination) and dispersed afterwards in a carrier liquid like ethanol by means of high shear mixing or ultra sonic treatment. The obtained CNT suspension is then feed into the extrusion process. Using CNT suspensions in the extrusion process also avoids the formation of CNT dust in the laboratory environment.

The aim of this report is to demonstrate that CNT suspensions can be used to produce polymer nanocomposites by means of twin screw extrusion. With the described procedure a homogeneous distribution of the CNTs in the polymer matrix can be achieved in order to obtain the desired property improvements for the polymer nanocomposite.

Material and methods
Test material
- Base Polymer: Polypropylene Metocene HM562S (LyondellBasell)
- Two CNT-Ethanol suspensions with different functionalization (Rescoll/France)

Test equipment
- Torque rheometer system Thermo Scientific™ HAAKE™ PolyLab OS System
- Co-rotating twin screw extruder Thermo Scientific™ HAAKE™ Rheomex PTW16 OS System (L/D = 40)
- Gravimetric RotoTube feeder for pellets
- Liquid feeding pump for the suspensions
- Vacuum pump
- Strand line with Varicut pelletizer

Test conditions
- Screw speed: 250 rpm
- Temperature profile: 20°/230°/250°/250°/230°/220°/220°/200°/200°
- Feed rate PP: 0.919 kg/h
- Feed rate CNT-suspension: 0.114 kg/h (equivalent to 0.5 % CNT in PP)

Test procedure
The complete extruder and screw configuration is presented in Fig. 1. In the first stage (zone 1) the polypropylene was added and molten in the first mixing section (zone 2). The CNT suspension was dosed into the second feeding port (zone 3) into the polypropylene melt by means of a liquid feeding pump. The ethanol of the suspension was removed from the extruder using an atmospheric venting port in zone 4 and a vacuum venting in zone 9.

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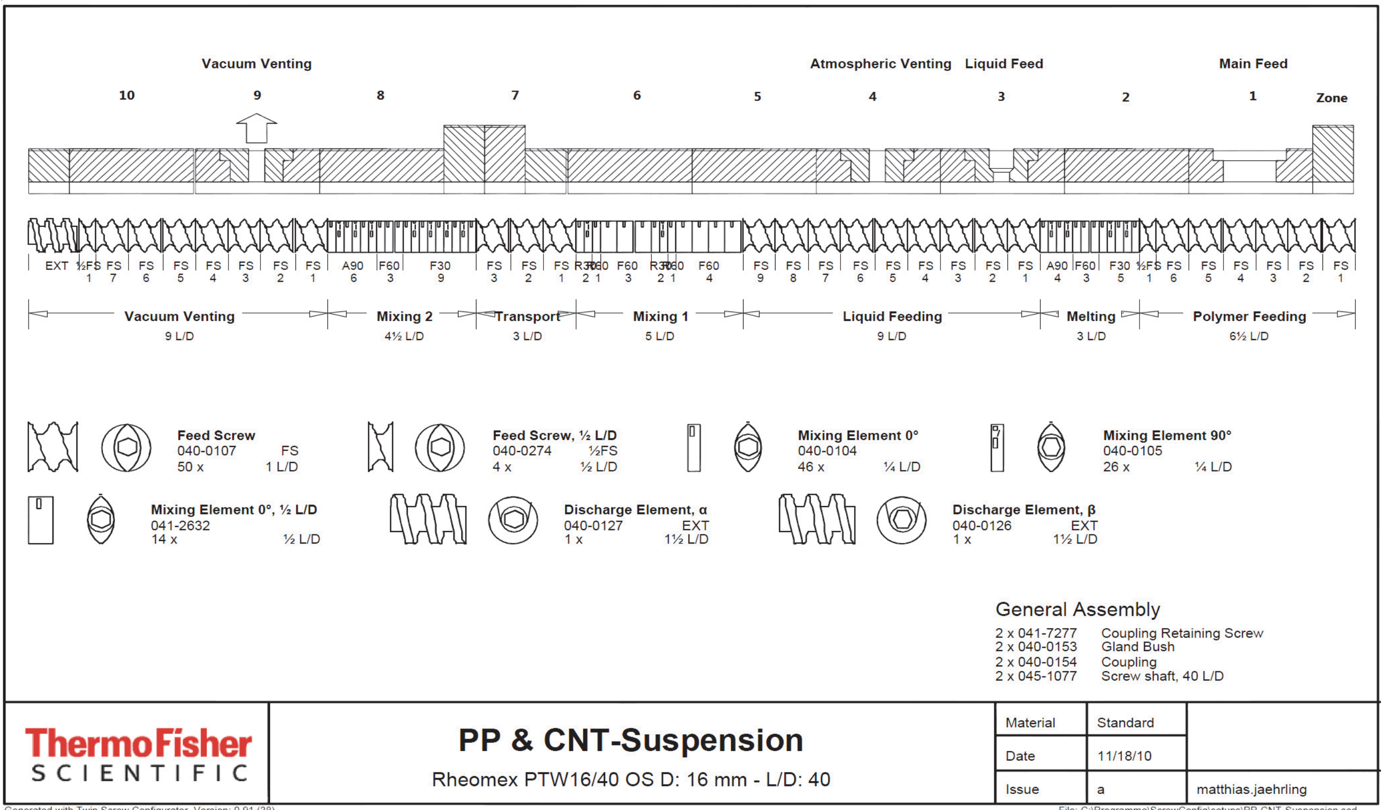

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Fig. 1: Extruder- and screw configuration.

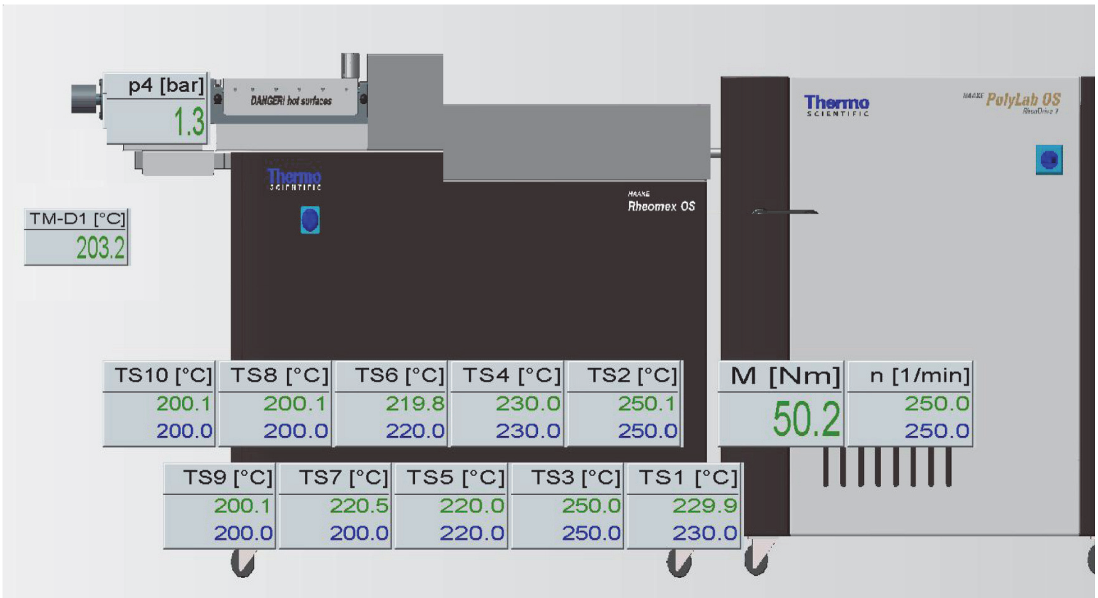

Fig. 2: Extrusion conditions.

The CNTs and the polypropylene were thoroughly mixed and sheared in two mixing sections in zones 5/6 and zone 8.

Results

During the test, the melt pressure at the die was measured. Fig. 3 shows this melt pressure as an overlay of three different extrusion tests. One test was done with the pure polypropylene, one test with the addition of CNT suspension "1" and one with CNT suspension "4".

It can be clearly seen that the pressure increased when a CNT suspension was added. The pressure difference between the two different suspen-sions itself was not significant. The extruded material was then formed into a strand, which was cooled down in a water bath and cut into pellets by a pelletizer.

Using our mini injection moulding machine, the Thermo Scientific™ HAAKE™ MiniJet System those pellets were injection moulded into specimens like disks and DMTA bars for further investigation.

Fig. 4 shows a microscopic picture taken from specimens made from the PP compound containing 0.5% CNT from suspension "1".

In this picture no agglomeration can be seen and the CNTs seem to be evenly distributed in the polymer matrix. Fig. 5 shows a microscopic picture taken from the PP compound containing 0.5% CNT from suspension "4". This picture shows a large amount of agglomerates. The dispersion seems to be much worse than the result we got from suspension "1".

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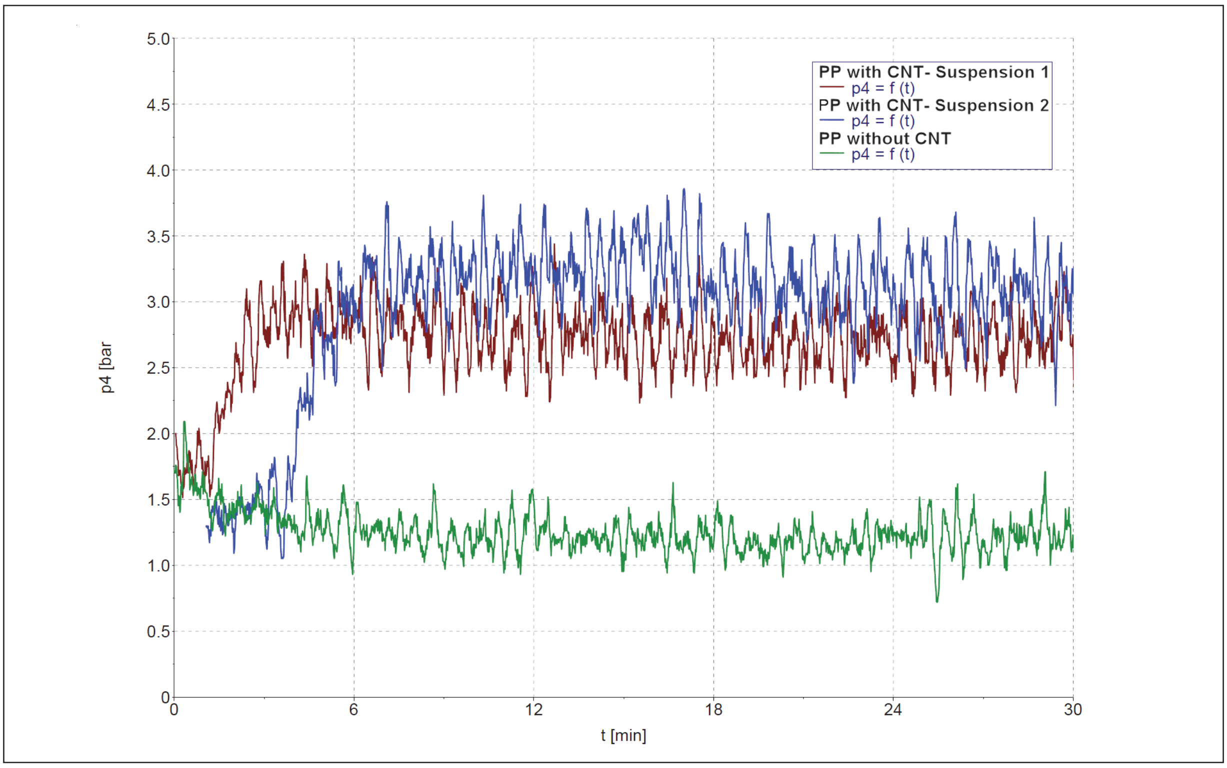

Fig. 3: Pressure at Die-Had.

Fig. 4: PP with 0.5% of CNT “1“.

Fig. 5: PP with 0.5% of CNT “2“.

Conclusion

The PolyLab System with the lab scale twin screw compounder, Rheomex PTW16, can be used to prepare compounds from polymers and CNTs using CNT suspensions.

The result of these tests shows significant differences between the compounds made with the differently functionalized CNTs.

Acknowledgement

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Reference

[1] Thermo Scientific Application note V241 “Dynamic mechanical thermal analysis (DMTA) on polymer nanocomposites“ Fabian Meyer, Klaus Oldörp and Frits de Jong

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