Application Note
LR-68

Compounding of Carbon Nanotube (CNT) Suspensions with Polypropylene

Matthias Jährling and Dirk Hauch, Thermo Fisher Scientific, Process Instruments, Karlsruhe, Germany

Carbon nanotubes are graphite sheets rolled into seamless tubes, with diameters of just some nanometers and lengths up to centimeters. Nanotubes have received much attention because of their unique properties such as high modulus and electrical/thermal conductivity. This has resulted in a large number of potential applications for CNTs, also in the field of polymer compounds, where they can be used to improve their mechanical and electrical properties.
One key to unleash these unique properties is to disperse (exfoliate) the CNTs thoroughly in the polymer matrix.
Because the CNT particles are under a cloud of being potentially hazardous to health, the safe handling and avoidance of CNT dust is more than important. One approach adressing both of these challenges is to have the CNTs pre-dispersed in a suspension. For this the CNTs are functionalized (i.e. by amination) and dispersed in a carrier liquid like ethanol by means of high shear stirring, or ultra sonic. This CNT suspension is fed into the extrusion process.

Test Objective

The aim of the described test is to prove the feasibility of CNT-suspensions, as an option for safe handling of CNTs in a lab environment and for use with small scale twin screw compounders.

Test Material

- Base Polymer: Polypropylene Metocene HM562S (LyondellBassell)
- Two CNT-Ethanol suspensions with different functionalization (Rescoll/France)

Test Equipment

- Torque rheometer system Thermo Scientific HAAKE PolyLab OS
- Co-rotating twin screw extruder Thermo Scientific HAAKE Rheomex PTW16 OS (L/D=40)
- Gravimetric roto tube 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 °C
- Feed rate PP: 0.919 kg/h
- Feed rate CNT-suspension: 0.114 kg/h (equivalent to 0.5% CNT in PP)

Test Procedure

In the first stage (zone 1) the Polypropylene was fed into the main feed port and melted in the first mixing section (zone 2). The CNT suspension was dosed into the second feeding port (zone 3) into the PP melt by means of a liquid feeding pump. The ethanol from the suspension was then vented in two steps, using an atmospheric venting port in zone 4 and a vacuum venting in zone 9.
The CNTs and the PP were thoroughly mixed and sheared in two mixing sections in zone 6 and zone 8 (Fig. 1).

Test 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.

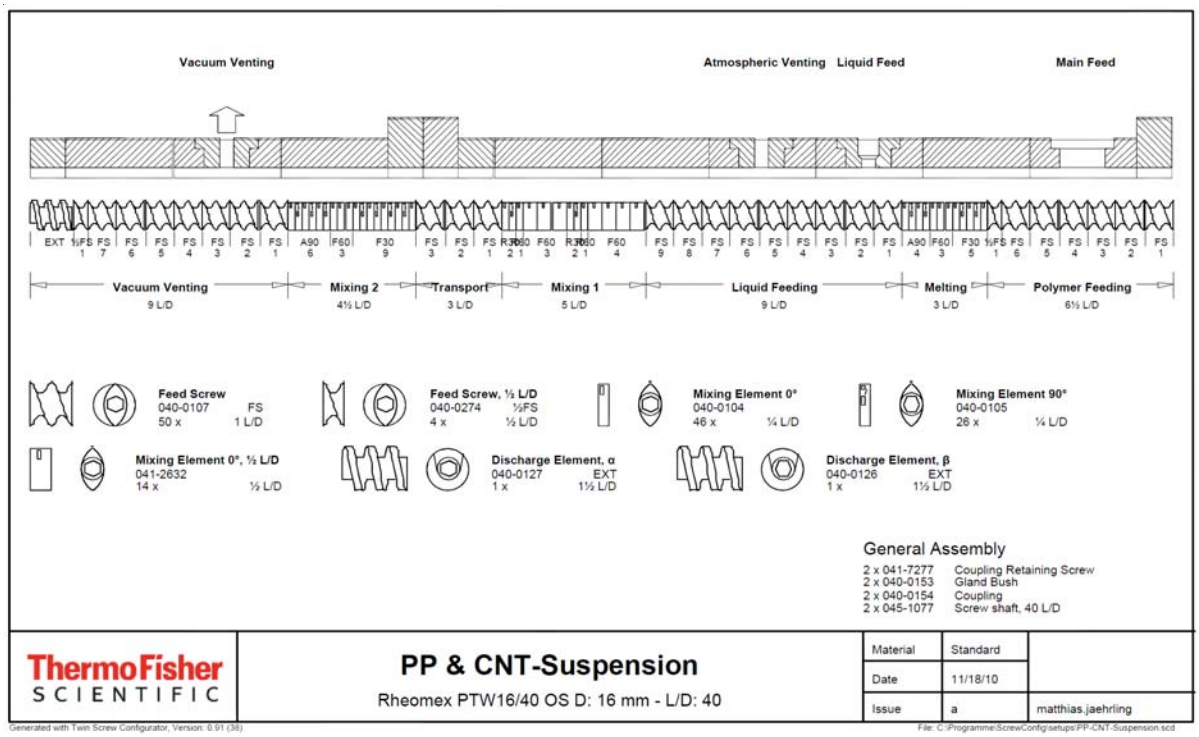

Fig. 1: Extruder- and Screw Configuration

One test was done with the pure Polypropylene, one test with the addition of CNT suspension “1“ and one with CNT suspension “2“.
Clearly it can be seen that the pressure increases when a CNT suspension is added. The pressure difference between the two different suspensions itself was not significant.
The extruded material was then formed into a strand, cooled down in a water bath and cut into pellets by a pelletizer.
Using the Thermo Scientific Haake MiniJet mini injection moulding machine those pellets were injection moulded into test specimens like discs and DMA bars for further investigations.
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 “2“. This picture shows a large amount of agglomerates. The dispersion seems to be much worse than the results from suspension “1“.

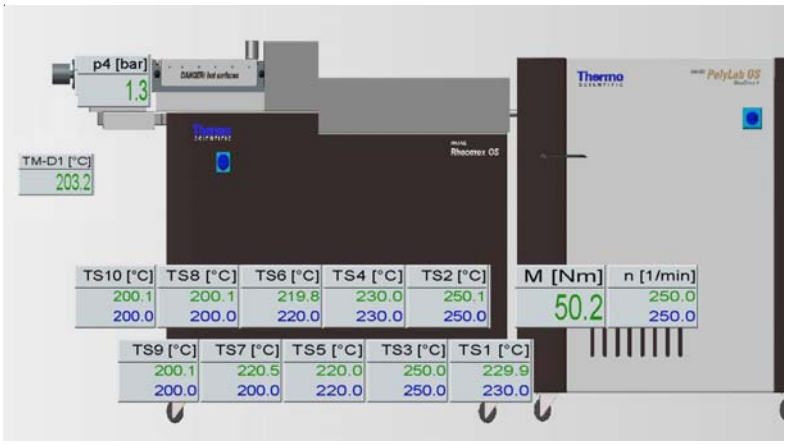

Fig. 2: Extrusion Conditions

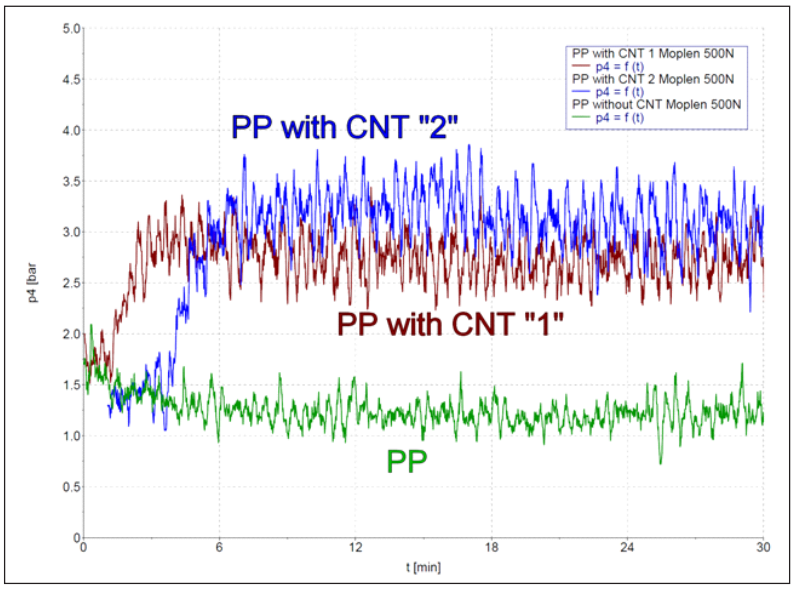

Fig. 3: Pressure at Die-Head

Fig. 4: PP with 0.5% of CNT “1“

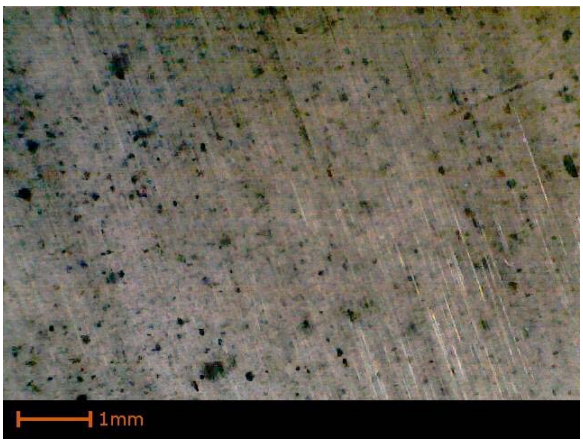

Fig. 5: PP with 0.5% of CNT “2“

Conclusion

The PolyLab System with the lab scale twin screw compounder Thermo Scientific Rheomex PTW16 can be used to prepare compounds from polymers and CNTs using CNT suspensions. The result of these tests show significant differences between the compounds made with the differently functionalized CNTs, and proves that there is significant scope for safe testing of CNT compounds with extrusion equipment in a lab environment.

Acknowledgement

We would like to thank the Fraunhofer Institute for Manufacturing Engineering and Automation IPA in Stuttgart for their input and the Research Company RESCOLL in Pessac/France for their kind co-operation and supplying the CNT-suspensions.

Process Instruments

International/Germany
Dieselstr. 4,
76227 Karlsruhe
Tel. +49(0)721 40 94-444
info.mc.de@thermofisher.com

Benelux
Tel. +31 (0) 76 5 87 98 88
info.mc.nl@thermofisher.com

China
Tel. +86 (21) 68 65 45 88
info.mc.china@thermofisher.com

France
Tel. +33 (0) 1 60 92 48 00
info.mc.fr@thermofisher.com

India
Tel. +91 (20) 66 26 7000
info.mc.in@thermofisher.com

Japan
Tel. +81 45 453 9167
info.mc.jp@thermofisher.com

United Kingdom
Tel. +44 (0) 1606 54 81 00
info.mc.uk@thermofisher.com

USA
Tel. 603 436 9444
info.mc.us@thermofisher.com

www.thermoscientific.com/mc

LR-68_31.03.11