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IS SUBMICRON TALC THE FUTURE HIGH COST PERFORMANCE NANO-MATERIAL FOR POLYPROPYLENE COMPOUNDS ?

by

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Introduction

The primary target of the introduction of nano- and submicron minerals in polymer matrices are extraordinary improvements in key aspects such as mechanical, electrical, and thermal properties. Equivalent and in some cases superior property enhancement is obtained with these fine particles at one-tenth to one-fourth of the loading of micronized reinforcements.

Platy minerals such as clay and talc are able to reinforce plastics due to their lamellar morphology. Typical addition rates for micronized talcs are 8-20%, whereas nanoclay producers promote loadings of 3-5% as sufficient.

The HiTalc Group already produces and markets talc of 1.0 and 0.6 µm (microns) medium particle size. Such submicron talc products have been used regularly in high performance automotive PP-compounds for more than two years. The objective of the study was to investigate even finer submicron talc products and to determine the fundamental changes in mechanical properties in PP-compounds with increasing fineness. The potential of some experimental submicron talcs was evaluated in comparison with Nanoclays.

Experimental

A new micronizing and delamination technique was investigated for talc in order to get products of less than 0.5µm medium particle size (D50). A new concept of jet-mill technology was identified and semi-industrial production scale samples were produced by PMT-Austria. The finest submicron talc product available for this study shows a D50 of 0.35µm and a topisze of 5-6µm. This seems to be the finest possible product to be processed by this dry grinding technology. Another already commercialized HiTalc®-grade with D50=0.6 µm was also included in this study. The product fineness was measured by Sedigraph 5100 (Micromeritics) and Scanning Electron Microscopy (SEM).

These talc products were compared in compounds with a commercially available nanoclay (Cloisite®20A, Southern Clay Products, USA) based on montmorillonite. A quaternary ammonium salt is included as an organic modifier; we find about 38% loss on ignition, in which 2% moisture content are included. The nanoclay data were taken from the datasheet and by SEM-measurement. D50 is reported as 6µm. However, after dispersion in the polymer matrix the agglomerates delaminate and show finer sizes.

Other constituents are :
MOPLEN F30G (Basell) a general purpose, good flow PP-homoplymer; Irganox B225 (CIBA-CH), a stabilizer package for PP; SCONA® TPPP2013FA (Kometra-D), a coupling agent for nanoclay compounds based on maleic-acid-anhydride, (MFR=10g/10min).

Table 1: Formulations – weight%

Sample	1	2	4	7
MoplenF30G	100	85	92	92
TPPP	-	7.5	-	-
Irganox B225	-	0.2	0.2	0.2
Nanoclay	-	7.5		
Talc 0.6	-		8	
Talc 0.35	-			8
Ash	-	4.9	8	7.8
Density g/cm^3	0.899	0.913	0.946	0.935

The compounding was made by the Institut für Polymerforschung Dresden (IPF), Germany. The compounds were processed on a co-rotating twin screw extruder. The clay-type masterbatches (50/50 clay/PP-g-MA) were processed in a preliminary stage, as common in PP-nanoclay-composite compounding.

Compounder : Co-kneader(CoperionBuss) MKS 30/18 E 40/6
Screw diameter: 30 mm
Processing unit: 18 L/D
Dosing: gravimetric, powder mixture (50/50) into the hopper
Venting: vacuum devolatilization of the melt
Pelletizing: strand die after water cooling
T-profile: 185/210/200/200/185/170°C
Screw speed: 300 min^{-1}
Output: 5 kg/h

The talc compounds could be made directly without masterbatch-intermediate step.

Compounder: Co-rotating twin screw ZE 25 UTS (Berstorff)
Screw diameter: 25 mm
Processing unit: 36 L/D
Dosing: talc - gravimetric via side feeder (16 L/D) into the melt
Pelletizing: strand die after water cooling
Temp. profile: 195/220/210…195/170°C
Screw speed: 200 min^{-1}
Output: 10 kg/h
Torque: 36 ... 45 Nm
Pressure before die: 19 ... 21 bar

The dispersion of the mineral fillers within the polymer matrix was investigated at the Institute of Mineralogy and Petrology, University Graz, using SEM and powder diffraction methods.

Sample moulding was done according to CAMPUS conditions.

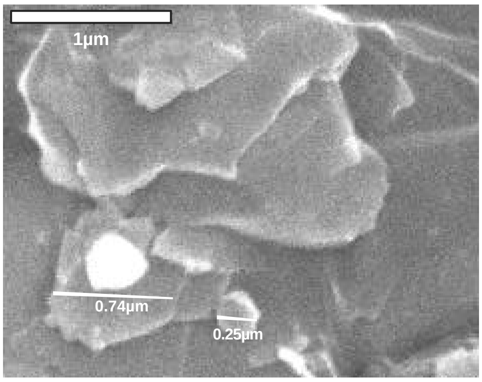
Equipment: Ergotech100/420-310 (Demag) clamping force 1000 kN
Temperature profile: 220°C 230°C 235°C
Mould temperature: 40°C
Multipurpose test specimens (ISO 3167 type A)
HDT: Bar 127 x 12,7 x 3,2 [mm]
Moulding shrinkage: Plates 80 x 80 x 4 [mm]
Heat ageing & colour: Plates 80 x 80 x 1 [mm]
All variants two-cavity moulds.

The performance tests of these compounds (mechanical, optical, ageing properties) were made at IPF-Dresden and some at Superlab-Salvaterra, Italy, using standard techniques and international norms.

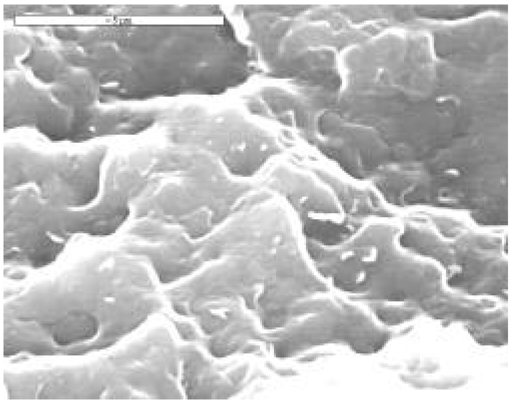
Results and Discussion

As shown in Table 1, the test series used 7,5% of the organo-modified nanoclay (equals about 4.5-5% dry clay) and 8 % submicron talc. A good dispersion of the mineral within the polymer matrix is the most important aspect in all compounds. The SEM-photos show proper dispersion for both minerals within the compounds.

SEM-Photo 1 : Talc0.35 powder, 15.000x SEM



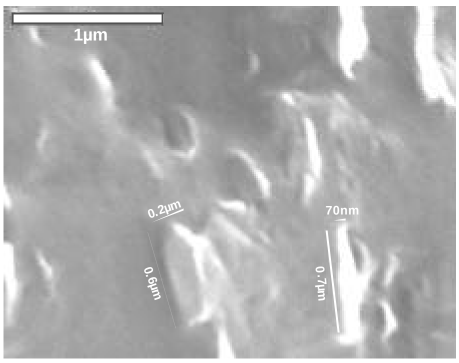
SEM-Photo 2: 7.5% Nanoclay in PP; 15.000x



In SEM-Photo2 it can be seen that the nanoclay appearing as white particles on the polymer surface is very well dispersed. This is supported by the organic modification of the nanoclay and the two step compounding via a masterbatch.

All SEM-Photos of this study show the absolute difference between the minerals: submicron talc shows particles of 0.1µm to 3-5µm in size, whereas nanoclay particles show dimensions of 70nm to 2-3µm after compounding.

SEM-Photo 3 : Nanoclay in PP, 15000x



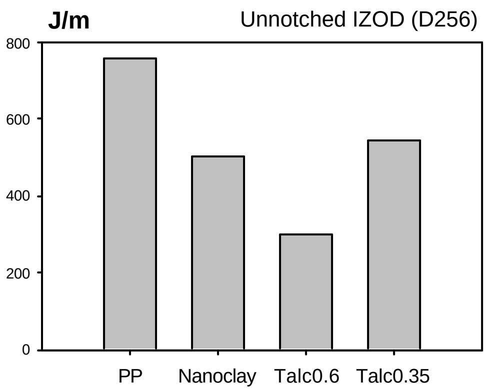
The comparison of the mechanical properties show the expected increase in reinforcement compared to the unfilled PP (formula 1) by nanoclay (formula 2) and the two different talc grades (formulae 3+4).

Table 2: Mechanical Properties

Property	Unit	1	2	3	4
Tensile stress at yield	MPa	31,8	36,7	35,0	35,0
Elongation at yield	%	8,9	6,9	6,7	6,7
Tensile modulus	GPa	1,40	2,14	2,13	2,12
Max.flexural stress	MPa	37,8	48,9	47,8	49,5
Flexural modulus	GPa	1,28	2,23	2,13	2,19
Notched impact + 23°C	kJ/m²	3,3	4,3	3,8	4,4
Notched impact - 10°C	kJ/m²	2,5	2,6	2,7	2,8
HDT A	°C	53,8	57,7	60,8	61,0
HDT B	°C	87,3	108,6	115,6	116,4
Ball indentation H 132/30		53	59	64	
H 357/30				63	64

It is remarkable that both submicron talc grades show equal stiffness and impact resistance as those with nanoclay. The 0.35 µm talc (4) causes slightly higher performance at notched impact compared to 0.6µm talc (3), which is a fundamental advantage of such a fineness. At unnotched impact, the finer talc grade dominates.

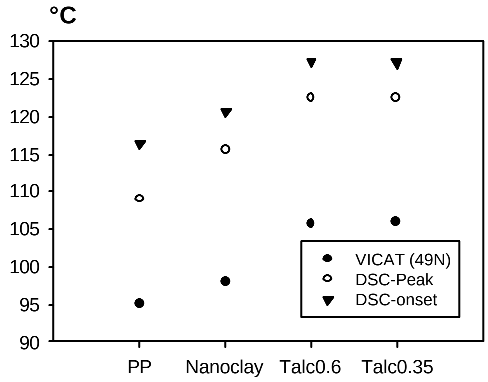
Graph 1: Impact Properties



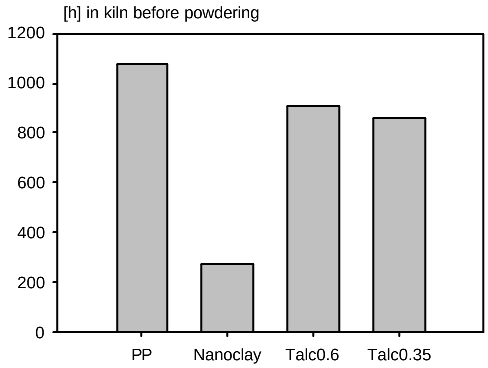
Both talcs show superior performance concerning thermal properties, e.g. Vicat (49N) and Crystallization Temperature.

Remarkable differences were seen concerning the thermostability in kiln (150°C). The selected nanoclay shows much lower resistance than both talcs; the organic modification of the clay certainly has a strong influence and requires different stabilizers resulting in much higher costs for the entire formulation.

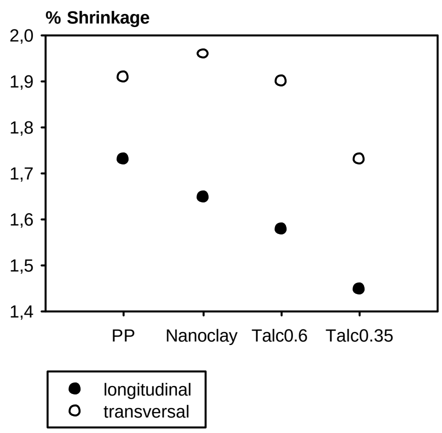
Graph 2: Thermal Properties



Graph 3: Thermostability



Graph 4: Lower mould shrinkage of talc filled compounds



Conclusions

The comparative tests showed encouraging data for submicron talc vs. nanoclay. Nevertheless, more efforts must be put on the production of the submicron talc, especially for a 0.35µm talc, for which industrial scale production equipment is not available yet. Considering that submicron talc with a fineness of D50>0.4µm should be available at price levels under 2€/kg, we may conclude that such a submicron talc is truely competitive with nanoclays concerning both quality and cost performance.