RESEARCH REPORT VTT-PRO C-0201 & C-0202
April 26th, 2002

Assessment of Conical Extruder Performance

On-line mixing of carbon black

Part 1; Mixing with pipe die (PRO C-0201)

Part 2; Mixing with high back pressure die (PRO C-0202)

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1. INTRODUCTION

This report is part of the two year development project started 1999 by Conenor Ltd. and VTT Processes at Tampere, Finland to develope a new mechanical design of Conenor conical extruder 300-4 equiped with two rotors for extrusion of four material layers in tubular form – and to verify the performance of the extruder in various new extrusion processes being developed simultaneously.

Conical extruder is an invention by Mr. Kari Kirjavainen since 1989 and the process technology has been developed under Conenor leadership at VTT in a joint project over 10 years participated by a group of Finnish industrial companies and co-financed by Tekes, the National Technology Agency of Finland. In recent years the conical extruder technology has been applied not only for extrusion of plastics products like pipes, cables and films, but also according to criteria of various other industries like medical and food.

This report characterizing the mixing performance of conical extruder contains 11 pages plus cover page.

Description of two rotor/four material layer conical extruder 300-4

In the compact geometry of the conical extruder the screw of a conventional single- or twin screw extruder is replaced by a short and deep conical rotor consisting of multiple spiral channels on both inner and outer surfaces for material flow, melting and mixing. As one rotor extrudes simultaneously two individually selected materials – by stacking conical rotors one after each other, the number of material layers in the extruded product is multiplied by factor 2 according to number of rotors in the extruder; i.e. four layer products with two rotor conical extruder and six layers with three rotors.

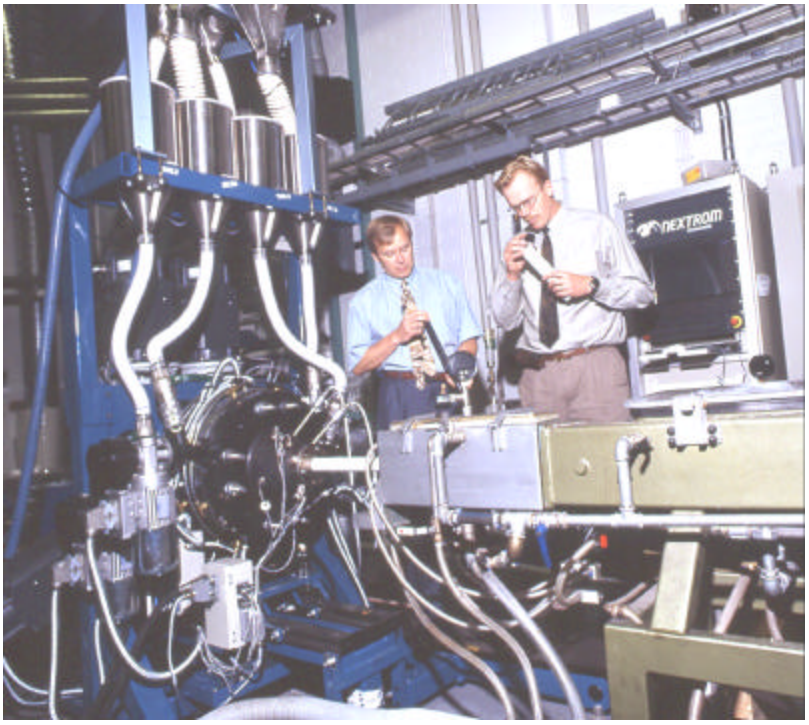
The definition “300-4” indicates that the extruder is of size 300 mm characterized by the nominal diameter of the rotors at rear end and may be used for extrusion of products up to four different material layers without side extruders.

Rotors are separated from each other by stationary housings called stators which also carry spiral channels for controlled and efficient material flow and mixing in the extruder. Stators are heated by help of heating rods and bands and divided into sections for setting and controlling the preferred temperature profile over the extruder and process in question. Rotors are rotated by individual drives thru a speciality constructed gear box.

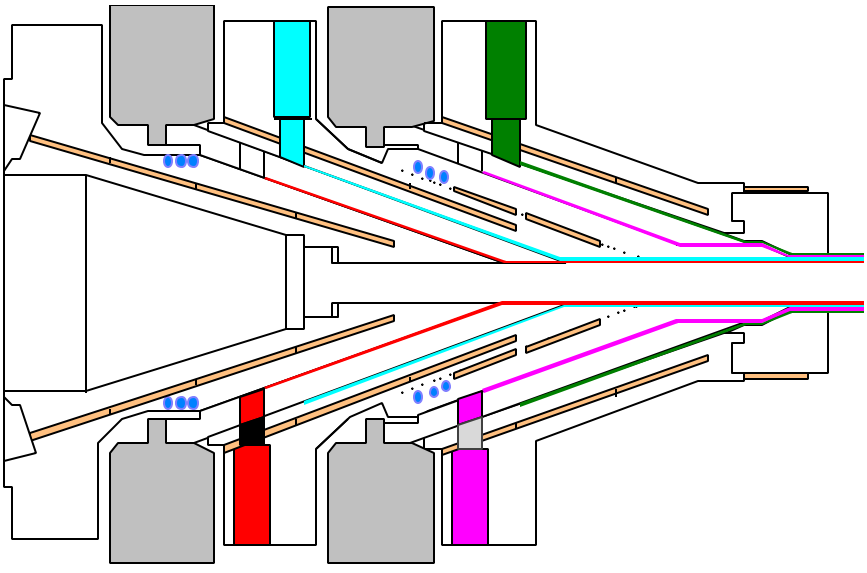
The two melt flows from inside and outside surfaces of a rotor merge inside the extruder at rotor tip and form an axisymmetric horizontal flow – or vertical flow if the extruder was set in vertical position – of two material layers pro rotor. The material flows coming from each rotor merge inside the extruder and at latest prior to entering the sizing die. The sizing die e.g. for each pipe diameter is a very simple and inexpensive construction without a need for melt separators and thus providing a seamless product.

Feeding of each layer of the rotor is realized with a small individual feeding screw attached to the extruder. Material to a feeding screw is provided from a hopper installed directly on top of the screw or alternatively via a transport hose from the hopper installed on a rack above the extrusion line.

Mixing of material flows in individual spiral channels along the rotor and stator surfaces has been most efficiently solved for homogenous melt with a mixing configuration named *SuperMixer*. This unique design of the conical extruder, that utilises both rotor and stator surfaces, is self cleaning and cuts the flow patterns hundreds of times without inducing excessive shear heat and is tested to be applicable also in reactive processes.
Also mixing configuration solutions used by conventional extruders, like barrier flights etc. can - and have been - applied to conical extruders but show lower efficiency than the *SuperMixer*-design.



Picture 1. Extrusion of pipes with two rotor/4-material layer conical extruder 300-4



Picture 2. Schematic diagram of a two rotor/4-material layer conical extruder

2. MIXING PERFORMANCE ASSESSMENT

Place and date of trials:	VTT Processes/Materials Technology laboratory n:o 8 at Tampere, Finland, Part 1; March 4th, 2002 & Part 2; December 4th and 5th, 2001	
Extruder :	Conical Extruder 300-4 coded "MODEX III", with *SuperMixer* rotor/stator geometry in each four layers, extruder owner Conenor Ltd.	
Die sets :	Part 1; pipe die	B2/M3, C1/M3, D1/M3, E3/M3, G3/M3
	Part 2; high back pressure die	B4/M3, C1/M3, D4/M3, E3/M3, G3/M3

The aim of the trials :

The aim of the trials was to assess the mixing performance of a two rotor/four layer conical extruder 300-4 with *SuperMixer* geometry with trials using either polyethylene based carbon black (50%) masterbatch granules or pure carbon black powder mixed and simultaneously extruded with a typical pipe grade polyethylene powder.
The final content of carbon black in polyethylene extrudate was dosed for 1% w. into the hopper of each material layer of the extruder in every trial.

Materials used in the trials were :

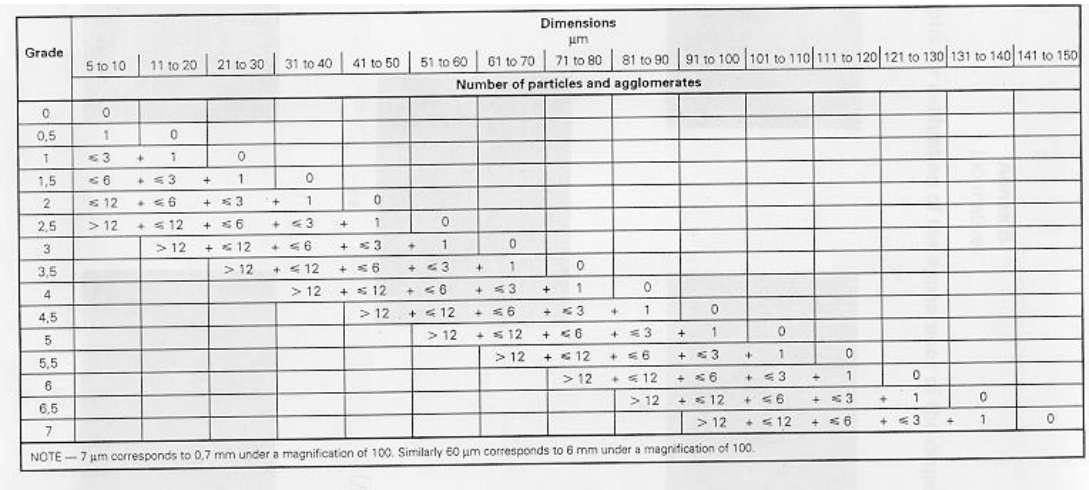
- polyethylene HE 2550 in natural powder form
- polyethylene based carbon black (50%) masterbatch Preblack PE 702-50 in granule form
- pure carbon black powder Printex L

Material combinations in samples were :

Samples 1-4: HE 2550 powder + Preblack PE 702-50 masterbatch
Samples 5-9: HE 2550 powder + Printex L carbon black powder

The standard ISO 11420 "Method for the assessment of the degree of carbon black dispersion in polyolefin pipes, fittings and compounds" was used to analyze the samples in Table 2 and Table 5 and eventually quantify the extruder mixing performance.

The principle of analyses is the following:
Six microtome specimens with thickness 25 µm ± 10 µm are cut from each test point. Particle size and agglomerates from specimens are examined with microscope (magnification at least x100). The largest dimension of each particle and agglomerate is measured and recorded ignoring those less than 5 µm. ISO 11420 grades according to the size categories are given in Table 1. In addition, uniformity of appearance was evaluated by comparing specimens to normative photomicrographs in ISO 11420. Appearance rating A1 is the best, and decreasing as A2, A3, B1, B2, C1, C2, D1, D2, E1 and E2 being the worst.



Grade	Dimensions µm														
	5 to 10	11 to 20	21 to 30	31 to 40	41 to 50	51 to 60	61 to 70	71 to 80	81 to 90	91 to 100	101 to 110	111 to 120	121 to 130	131 to 140	141 to 150
	Number of particles and agglomerates														
0	0														
0,5	1	0													
1	≤ 3	+ 1	0												
1,5	≤ 6	+ ≤ 3	+ 1	0											
2	≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0										
2,5	> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0									
3		> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0								
3,5			> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0							
4				> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0						
4,5					> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0					
5						> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0				
5,5							> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0			
6								> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0		
6,5									> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0	
7										> 12	+ ≤ 12	+ ≤ 6	+ ≤ 3	+ 1	0

NOTE — 7 µm corresponds to 0,7 mm under a magnification of 100. Similarly 60 µm corresponds to 6 mm under a magnification of 100.

Table 1. ISO 11420 Grades for particles and agglomerates.

According to this ISO 11420 standard, the following acceptance limits are recommended:

- Grade: average less than 3
- Appearance rate: not worse than B1

3. PART 1 – TRIALS WITH PIPE DIE

The mixing performance of conical extruder 300-4 was examined in all four layers (L1, L2, L3 and L4) with corresponding feeder rpm speed (F1, F2, F3 and F4) and the two individual rotor rpm speed RI and RII. Trial set values used are presented in Table 2.

Sample no.	RI (rpm)	RII (rpm)	F1 (rpm)	F2 (rpm)	F3 (rpm)	F4 (rpm)	Output (kg/h)
Masterbatch							
1	10	18	55	60	55	55	46
2	15	20	80	80	90	90	69
3	20	30	130	130	150	150	114
4	20	40	160	130	190	190	137
Pure carbon black							
5	10	18	55	60	55	55	33
6	15	20	80	80	90	90	50
7	20	30	130	130	150	150	84
8	20	40	160	130	190	190	100
9	30	48	220	200	280	280	142

Table 2. Carbon black and extruder feeders' and rotors' rpm set values used for each trial

3.1 RESULTS

3.1.1 Grade and Appearance rate results

Test results according to ISO 11420 are shown in Table 3.

Masterbatch samples

Sample		Dispersion (average)	Appearance rate
1	L1	1,2	A1
	L2	1,3	A1
	L3	0,9	A1
	L4	1,6	A1
2	L1	0,9	A1
	L2	1,4	A1
	L3	1,5	A1
	L4	1,7	A1
3	L1	0,7	A1
	L2	1,2	A1
	L3	1,5	A1
	L4	1,7	A1
4	L1	0,8	A3
	L2	1,3	A1
	L3	1,6	A1
	L4	1,5	A1

*) A3 for one of six samples
**) A3 for two of six samples

Pure carbon black samples

Sample		Dispersion (average)	Appearance rate	
5	L1	1,3	A1	*)
	L2	1,2	A1	**)
	L3	1,0	A1	*)
	L4	1,6	A1	
6	L1	1,1	A1	*)
	L2	1,3	A1	*)
	L3	1,4	A1	*)
	L4	1,3	A1	*)
7	L1	1,5	A1	
	L2	1,6	A1	
	L3	1,3	A1	
	L4	2,2	A1	
8	L1	2,1	A1	
	L2	1,3	A1	
	L3	2,3	A1	
	L4	2,5	A1	
9	L1	1,6	A1	
	L2	1,7	A1	
	L3	2,4	A1	
	L4	3,2	A1	

Table 3. Dispersion grades and appearance rates for individual carbon masterbatch samples (1-4) and pure carbon black samples (5-9).

Both Grade and Appearance rate give relatively high scores, which means that the analysed samples contain only a few agglomerates and they are small. Differences between masterbatch and pure carbon black samples at lower outputs are negligible equally as the differences between the material layers (see Table 4).
At higher outputs >100 kg/h the pure carbon black samples give slightly lower dispersion grade values than masterbatch samples especially in layers 3 and 4.

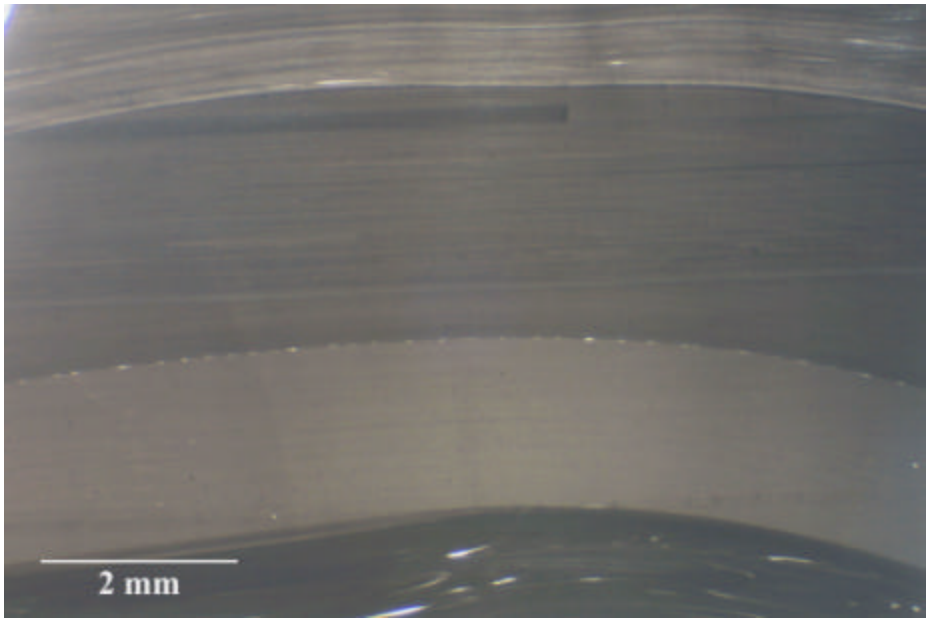
Sample	Layer L1	Layer L2	Layer L3	Layer L4	Average	
1	1,2	1,3	0,9	1,6	**1,25**	
2	0,9	1,4	1,5	1,7	**1,38**	
3	0,7	1,2	1,5	1,7	**1,28**	**Masterbatch average**
4	0,8	1,3	1,6	1,5	**1,30**	**1,30**
5	1,3	1,2	1	1,6	**1,28**	
6	1,1	1,3	1,4	1,3	**1,28**	
7	1,5	1,6	1,3	2,2	**1,65**	**Pure carbon**
8	2,1	1,3	2,3	2,5	**2,05**	**black average**
9	1,6	1,7	2,4	3,2	**2,23**	**1,70**
average	**1,24**	**1,37**	**1,54**	**1,92**	**1,52**	

Table 4. ISO 11420 average Grade values for masterbatch samples (1-4) and pure carbon black samples (5-9) in material layers L1, L2, L3 and L4.

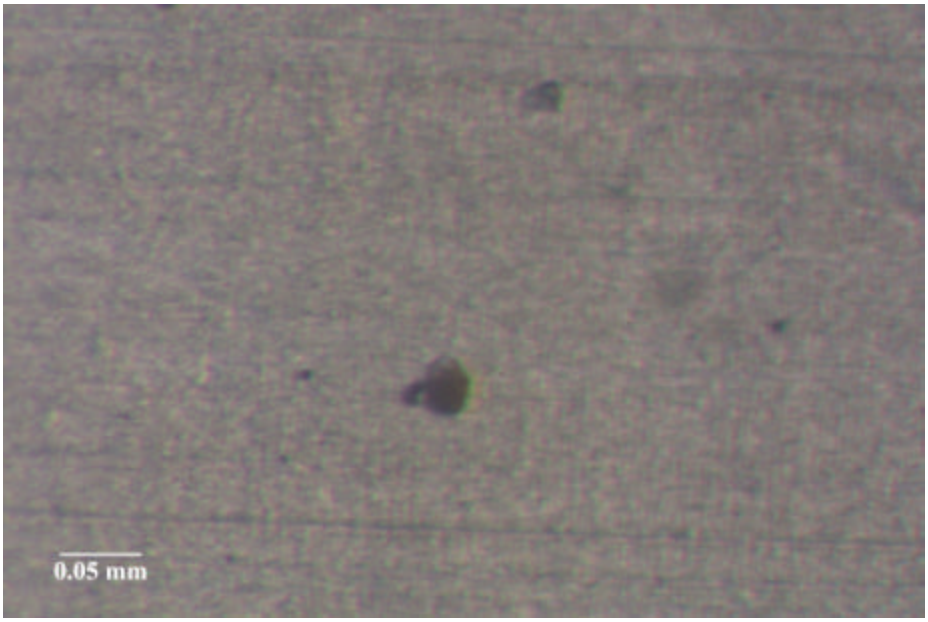
3.1.2 Optical analysis

For optical analysis a stereomicroscope type Wild M5A was used.

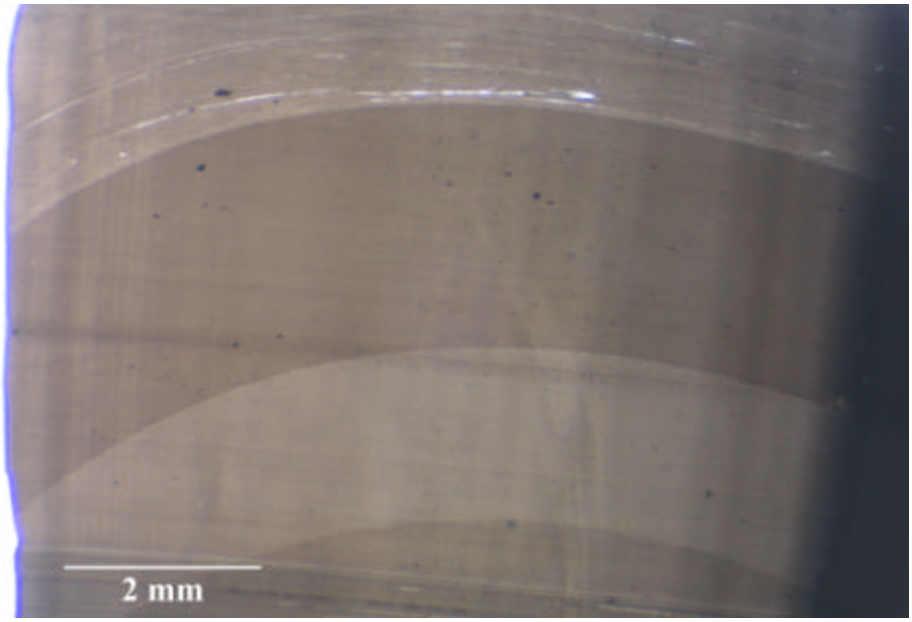
From the stereomicroscope images taken from the samples it can been seen that mixing efficiency in the layer with carbon black is uniform and that border lines to next adjacent layers are sharp.



Picture 3. Microscopic photo from sample 3 layers L1, L2, L3 and L4.



Picture 4. Microscopic photo from sample 3 layer L2 (magnitude x100)



Picture 5. Microscopic photo from sample 8 layers L1, L2, L3 and L4

4. PART 2 – TRIALS WITH HIGH BACK PRESSURE DIE

The mixing performance of conical extruder 300-4 was examined in three inner layers (L1, L2 and L3) with corresponding feeder rpm speed (F1, F2 and F3) and the two individual rotor rpm speed RI and RII which were though set to run in the same values. Trial set values used are presented in Table 5.

Sample no.	Masterbatch content (%)	Carbon black content (%)	RI=RII (rpm)	F1-F3 (rpm)	F4 (rpm)
1	2	1	5	5	5
2	2	1	5	15	3
3	2	1	10	15	5
4	2	1	15	30	5
	Pure carbon black				
5		1	5	5	0
6		1	5	15	0
7		1	10	15	5
8		1	15	30	0

Table 5. Carbon black and extruder feeders' and rotors' rpm set values used for each trial

4.1 RESULTS

4.1.1 Grade and Appearance rate results

Test results as Grade according to ISO 11420 are shown in Table 6.
Appearance rate for all samples 1-8 varied from A1 to A2.

Masterbatch samples

sample		1	2	3	4	5	6	average
1	**L1**	0,5	0,5	1	0,5	1	1	**0,8**
	L2	1,5	0,5	2	1	2	1,5	**1,4**
	L3	1,5	0,5	0,5	0,5	0,5	0,5	**0,7**
2	**L1**	1	0	1,5	0	0	0	**0,4**
	L2	1,5	1	0,5	1,5	0	1	**0,9**
	L3	1	1	0	1	1,5	1	**0,9**
3	**L1**	0,5	1,5	1	0	1	1,5	**0,9**
	L2	0,5	0	0,5	0,5	1	1	**0,6**
	L3	0,5	1	0,5	1,5	1	1	**0,9**
4	**L1**	1,5	0,5	1	1	1,5	1,5	**1,2**
	L2	0	1	1,5	0,5	1	1,5	**0,9**
	L3	1	0	1,5	0	1,5	2	**1**

Pure carbon black samples

sample		1	2	3	4	5	6	average
5	**L1**	1	1,5	1	1,5	1	0,5	**1,1**
	L2	1	0,5	1	1,5	1,5	1	**1,1**
	L3	1,5	1	0	1,5	0,5	1,5	**1**
6	**L1**	0,5	0	0,5	1,5	0,5	0	**0,5**
	L1	1	0,5	1	1	0,5	0	**0,7**
	L3	1	1	1	0,5	0,5	1,5	**0,9**
7	**L1**	0	0,5	0	1	0,5	0,5	**0,4**
	L2	0,5	0	0,5	1	0,5	0,5	**0,5**
	L3	0	0	1,5	0	1	1	**0,6**
8	**L1**	0,5	0,5	0	1,5	0	1,5	**0,5**
	L2	1	0	0,5	0,5	0	2	**0,7**
	L3	2	0	1	2	1,5	0	**1,1**

Table 6. Grades for individual carbon masterbatch samples (1-4) and pure carbon black samples (5-8).

Both Grade and Appearance rate give high scores, which means that the analysed samples contain only a few agglomerates and they are small. Differences between masterbatch and pure carbon black samples are negligible equally as the differences between the material layers (see Table 7).

Sample	Layer L1	Layer L2	Layer L3	ave
1	0,8	1,4	0,7	**0,97**
2	0,4	0,9	0,9	**0,73**
3	0,9	0,6	0,9	**0,80**
4	1,2	0,9	1	**1,03**
5	1,1	1,1	1	**1,07**
6	0,5	0,7	0,9	**0,70**
7	0,4	0,5	0,6	**0,50**
8	0,5	0,7	1,1	**0,77**
ave	**0,73**	**0,85**	**0,89**	**0,82**

Masterbatch
average value Grade 0,88

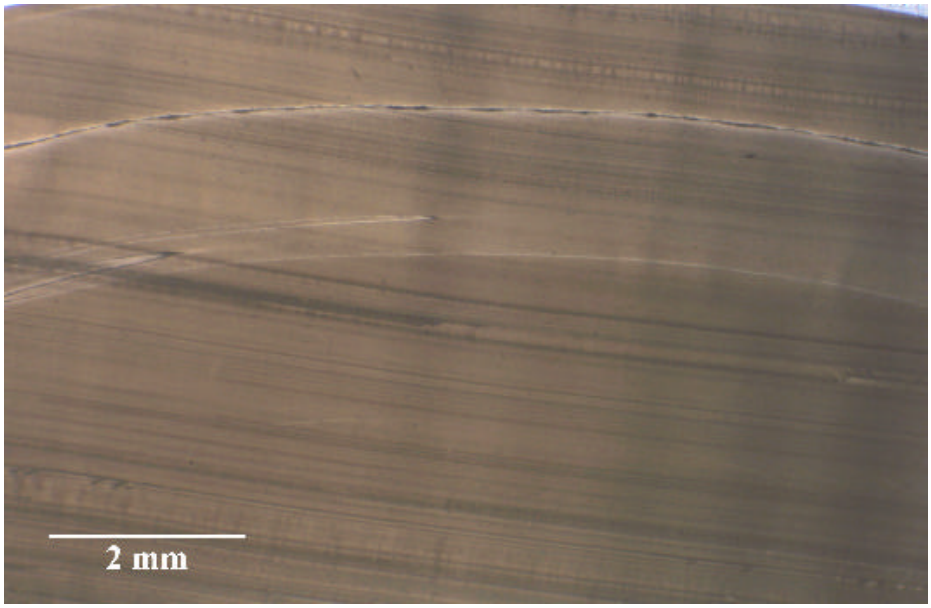
Pure carbon black
average value Grade 0,76

Table 7. ISO 11420 average Grade values for masterbatch samples (1-4) and pure carbon black samples (5-8) in material layers L1, L2 and L3.

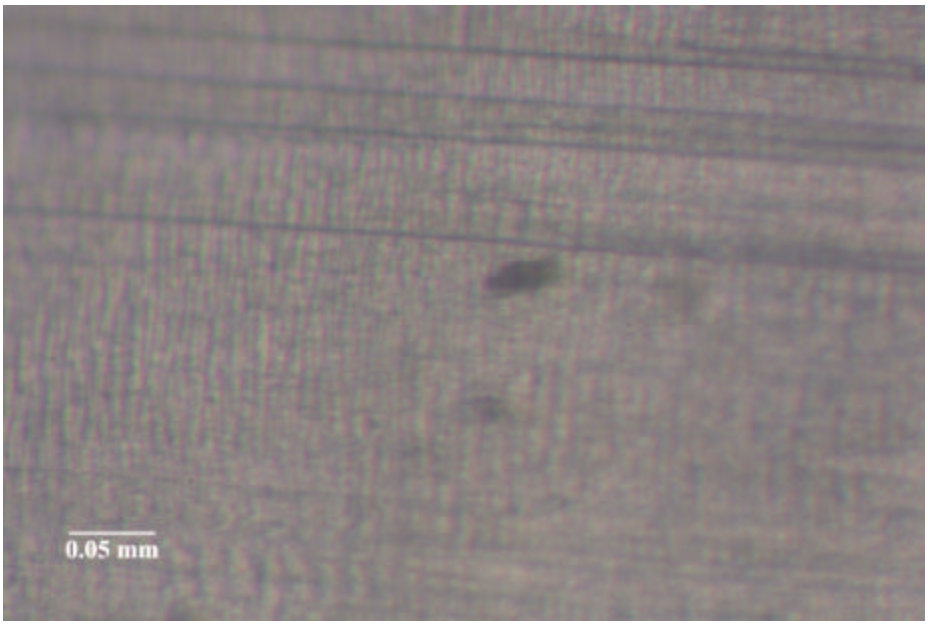
4.1.2 Optical analysis

For optical analysis a stereomicroscope type Wild M5A was used.

From the stereomicroscope images taken from the samples it can been seen that mixing efficiency in the layer with carbon black is uniform and that border lines to next adjacent layers are sharp.



Picture 6. enlarged 6x microscopic photo from sample 6 layers L1, L2 and L3.



Picture 7. Microscopic photo from sample 5 layer L3 (magnitude x100)

5. CONCLUSIONS :

The standard ISO 11420 " Method assessment of the degree of carbon black dispersion in polyolefin pipes, fittings and compounds" was used to evaluate the mixing efficiency of Conenor conical extruder 300-4.

5.1. PART 1 – TRIALS WITH PIPE DIE

Carbon black was dosed 1 w-% in polyethylene powder and dispersion studied in all four layers L1, L2 L3 and L4. In Part 1 trials were accomplished with standard conical extruder pipe die in representative outputs from about 50 to 150 kg/h.

The dispersion is described by a grade, which correlates with the amount and size of carbon black agglomerates. The average grade of all samples was 1.52. In the standard recommendation average value was less than three. A visual evaluating was carried out by comparing micrograps to standards normative graphs. With this method as well, samples achieved very high rates. Results prove that extremely well dispersed melt and homogenous product is achieved with *SuperMixer*.

5.2 PART 2 – TRIALS WITH HIGH BACK PRESSURE DIE

Carbon black was dosed 1 w-% in polyethylene powder and dispersion studied in three layers L1, L2 and L3. In Part 2 trials were accomplished with high back pressure die and therefore outputs were relatively low; from 1 to 5 kg/h.

The dispersion is described by a grade, which correlates with the amount and size of carbon black agglomerates. The average grade of all samples was 0.82. In the standard recommendation value was less than three. A visual evaluating was carried out by comparing micrograps to standards normative graphs. With this method as well, samples achieved very high rates. Results prove that extremely well dispersed melt and homogenous product is achieved with *SuperMixer*. Differences between masterbatch and pure carbon black were negligible as well as differences between different layers.

Authors

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Mr. Kari Kirjavainen, B.Sc. (Eng.), inventor and Technical Director of Conenor Ltd., Tampere, Finland, 22 patents and patent applications in conical extruder, since 1989 technical consultant for VTT polymer processes