

Filter Pressure Value Test (FPV test) - a new standard to examine master batches, white and carbon black colorants

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Keywords:

- Filter Pressure Value Test

Introduction

For quite a period screen life tests for rating the quality and dispersibility tests of colorants in polymer melts have been performed. But the comparison of those tests were poor. The new standard EN 13900-5 describes now a procedure which increases the comparability of those tests. On one hand this standard describes which kind of extruder, melt pump, sieves and breaker plate have to be used and on the other hand the composition of mixture, the test routine and the evaluation is described. One parameter, the filter pressure value - FPV - describes the quality and dispersion of the pigments. Due to standardized quality control, an improvement of the products is possible. Also the ranking of the same products from different vendors by comparing the FPV results is now easier. The use of the FVP test to check raw materials ensures the quality of your products and the up time of screen changers is more predictable.

Application and operation

The procedure is suitable for testing colorants in the form of color concentrates in all polymers used for extrusion in melt-spinning processes. The test mixture is processed in a HAAKE PolyLab single screw extruder with a non grooved barrel and a compression screw with now addi-

tional mixing elements. The polymer melt is fed with a constant pressure of 30 to 60 bar (435 to 870 psi) to the Thermo Scientific HAAKE Melt Pump with a volume of 1.2 cm³/rpm. With this metering pump a constant volume of 50 to 60 cm³/min (setting 660 on the Melt pump) polymer passes the filter package. Particles and agglomerates over a certain size are retained and clog the filter package. A pressure increase is observed and is correlated with the quality of the colorant. The pressure difference between the initial pressure and the peak pressure is used for the calculation of FPV - filter pressure value.

Samples, sample preparation and screen packs

The color concentrate is a homogeneous mixture of colorant in a suitable appropriate thermoplastic polymer.

The basic test polymer is an appropriate thermoplastic polymer. The type and grade has to be agreed between the interested parties.

The test mixture is a homogeneous mixture of color concentrate and basic polymer. Practically there two test mixtures established.

Mixture 1 - recommended for tests with color pigments

A mixture of 200g basic test polymer (100 %) which contains 5.0 g of colorant (2.5 %) has to be tested. If the color concentrate contains 40 % colorant, the following quantities are required: 12.5 g color concentrate and 187.5 g of basic polymer.

Screen pack 1

The screen pack 1 (picture 2) is a 2 layer construction, where the first layer is a reverse plain Dutch weave 615/108 mesh and a wire of 0.042 mm/0.14 mm and the second layer (support mesh) is a square mesh plain weave 0.63 mm and a wire of 0.40 mm calendered (for further details see ISO 9044).

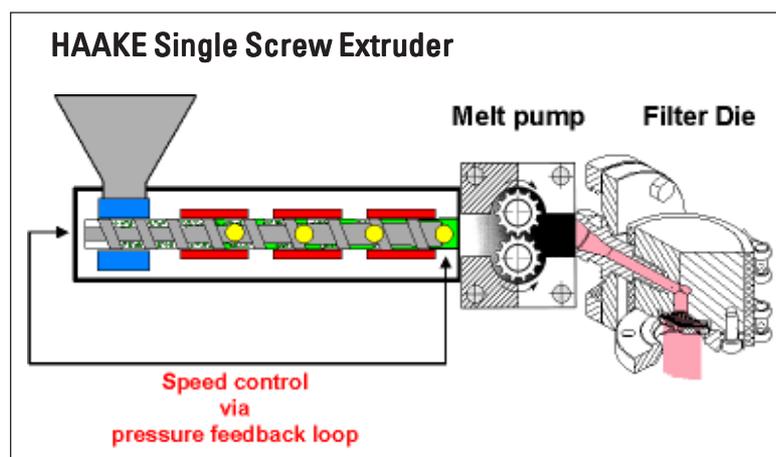
Mixture 2 - recommended for white and carbon black pigments

A mixture of 1000g basic test polymer (100 %) which contains 80 g of colorant (2.5 %) has to be tested. If the color concentrate contains 40% colorant, the following quantities are required: 200 g color concentrate and 800 g of basic polymer.

Screen pack 2

The screen pack is a 2 layer construction, where the first layer is a reverse plain Dutch weave 615/132 mesh and a wire of 0.042 mm/0.13 mm and the second layer (support mesh) is a square mesh plain weave 0.63 mm and a wire of 0.40 mm calendered (for further details see ISO 9044).

A standardized breaker plate (picture 3) supports the screen pack and determines the free area.



Picture 1: Schematic layout

Execution of the test

In preparation for the test so that polymer melt flows through the finer screen first. The aluminum rim of the screen pack performs as a sealing to prevent an undermining of the screen pack. The breaker plate and screen pack have to be heated up. Purging with basic test polymer reduces the time for temperature equalization. The melt temperature should be stable $\pm 2^\circ$ Celcius.

The basic test polymer is plasticized in the extruder and pumped through the screen-pack with a defined melt volume throughput until the melt temperature and pressure remain constant. The feeding pressure of the melt pump should be in the range of 30 to 60 bar (435 to 870 psi) and the volume flow is in the range of 50 to 60 cm³/min. The equipment should guarantee a constant melt temperature, with temperature deviations of less than $\pm 2^\circ$ °C.

The pressure P_s developed by the basic test polymer directly in front of the screen-pack is measured. The pressure P_s should be constant. When the hopper is empty, and the feed-screw is just visible, the test mixture is poured into the hopper.

Due to different rheological properties of the basic test polymer and the test mixture a pressure drop can occur.

When the hopper is empty, and the feed-screw is just visible basic test polymer is filled into the hopper. The test is finish after 100 g of basic test polymer have passed the screen pack and the pressure is constant. The screen pack and breaker plate are removed at operating temperature and the machine is purged with basic test polymer to prepare for the next test.

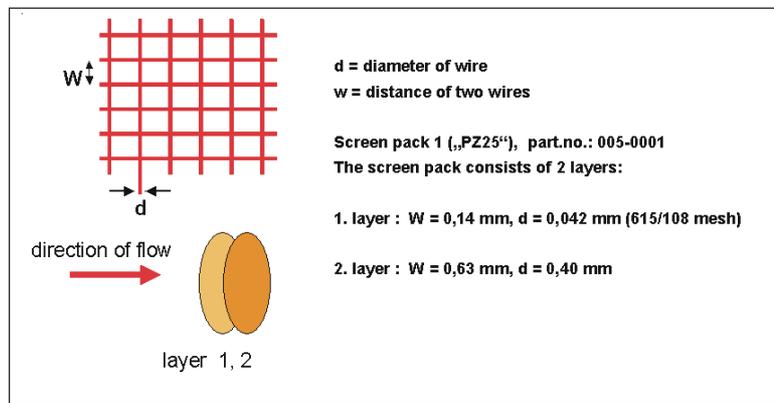
Result and evaluation

The filter pressure value [FPV] is defined as the increase of pressure per gram colorant and calculated using by the following equation:

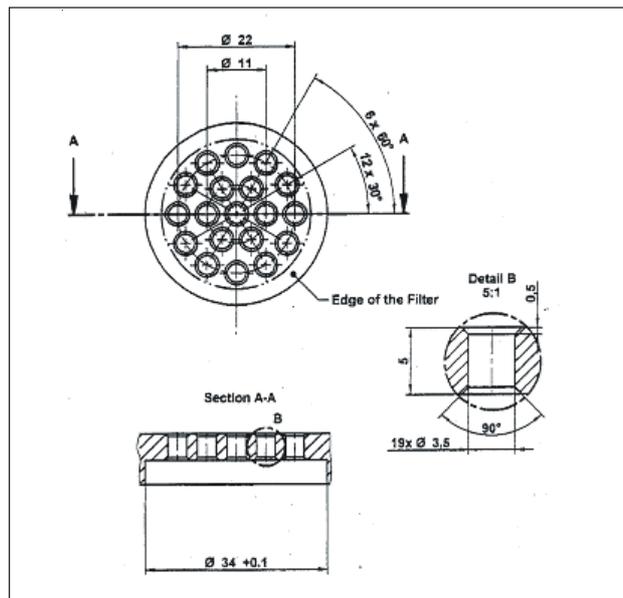
$$FPV = \frac{(P_{max} - P_s)}{m_c} \text{ [bar/g]}$$

where
 FPV Filter pressure value [bar/g]
 P_s Start pressure in bar
 P_{max} Maximum pressure in bar
 m_c Quantity of colorant in gram

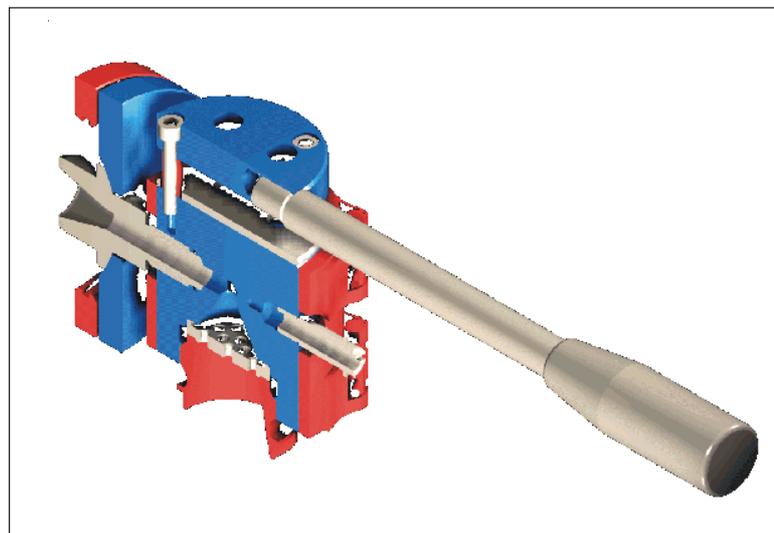
It is recommended to record the result accurately to one decimal place. In edition to the standardized evalua-



Picture 2: Screen pack



Picture 3: Breaker plate



Picture 4: Filter test die with breaker plate and central quick locking screw

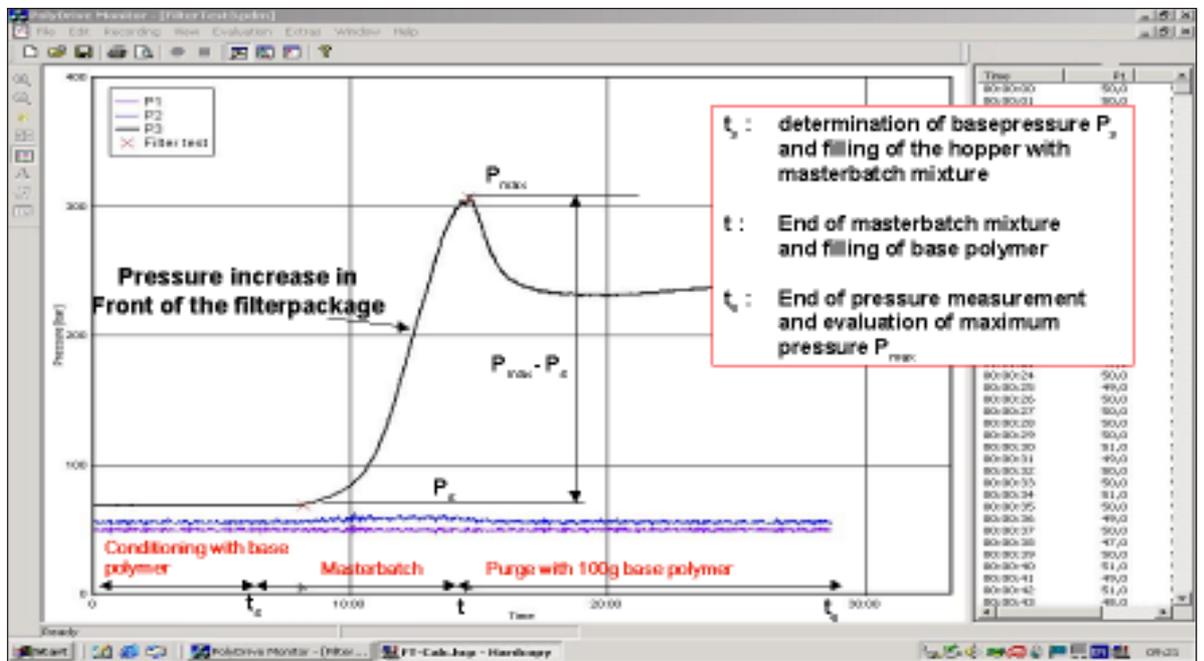
tion, it can be agreed on an individual evaluation.

The Thermo Scientific HAAKE Filter Test Software with an integrated formula editor supports a customizable evaluation.

The test report should provide the following information

Colorant concentrate:
 Masterbatch in PP (40 % colorant,

Color Index: 14)
 Basis test polymer:
 PP for fiber spinning,
 MFR 230 °C/2.16 kg = 25.4 g/10 min
 Test mixture:
 12.5 g Masterbatch and 187.5 g PP for fiber spinning
 Melt temperature: 230 °C
 Throughput: 55 cm³/min
 Screen pack: Number 1
 Filter pressure value: FPV = 9.9 bar/g



Picture 5: Filter Test Software with automatic evaluation of the filter test

Thermo Electron Processing Instruments Dieselstraße 4 D-76227 Karlsruhe Fon: +49 (0) 721/4094-0 Fax: +49 (0) 721/4094-300 info@thermohaake.com	HAAKE PolyDrive v1.40 Material : PP Company : Thermo Lot no. : 2388-2003 Remarks : Operator : drj	Date : 18.08.2003 Time : 22:05 Examiner : _____ Signature
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Filter test result Sample mass : 200 g Master batch : 14,5 % Initial Pressure : 69 bar Maximal pressure : 356 bar	
FPV : 9,89655 bar/g	

Remarks: Colorant concentrate: Masterbatch in PP (40 % colorant, Color Index: 14) Basis test polymer: PP for fiber spinning, MFR 230 °C/2,16 kg = 25,4 g/10 min Test mixture: 12,5 g Masterbatch and 187,5 g PP for fiber spinning Melt temperature: 230 °C Throughput: 55 cm ² /min Screen pack: Number 1 Filter pressure value: FPV = 9,9 bar/g	
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Picture 6: Automatic calculation and test report from the Filter Test Software

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