Improved Pigment Dispersion with Micronized Wax

what is precious to you?
Content:

- Processes of the Masterbatch Industry
- Clariant Waxes for Masterbatch Applications
- Improved Pigment Dispersion – Theory, Influence of the Particle Size
- Methods to Determine the Dispersion Quality
- Trial Results with Ceridust in Masterbatch: PE, PP and Engineering Resins
- Summary
Processes of Masterbatch Preparation - a Simplified Scheme

Components

- Plastic
- Pigment
- Additives e.g. waxes

Premix

- Cold mixing
- Hot mixing

Compounding

- Premix-process main-feeding single/ twin/ multiple-screw
- Split feed-process side-feeding single/twin-screw
- Special-process e.g. kneader

Granulation

- Strand pelletization
  - hot cut process standard
  - cold cut process standard
  - special process under water

Public, Improved Pigment Dispersion with Micronized Wax
Christian Lechner, BU Additives, Technical Marketing Waxes, 13.05.2013
Clariant Waxes for Masterbatch Applications

- Montan-wax
- Ceridust® Micronized waxes
- PE-wax
- PP-wax
- Amide wax
Advantages of Waxes for Masterbatch Production?

- Lower coloration costs
- Good pigment wetting
- Improved dispersion
- Increased pigment concentration
- Viscosity adjustment
The Clariant Wax Range

Polyolefin Waxes
- non-polar
- polar
  - Polypropylene Waxes
    - Licocene® PP 6102
    - Licocene PP 1302
    - Licocene PP 1502
    - Licocene PP 1602
    - Licocene PP 2602
    - Licocene PP 6502
    - Licocene PP 7502
    - Ceridust® 6050 M
  - Polyethylene Waxes
    - Licowax PE 130
    - Licowax PE 520
    - Licowax PE 190
    - Licocene PE 4201
    - Ceridust 3620
  - oxidized Polyethylene Waxes
    - Licowax® PED 191
    - Licowax PED 521
    - Licolub® H 12
    - Ceridust 3715
  - modified Polypropylene/ Polyethylene Waxes
    - Licowax® PED 191
    - Licowax PED 521
    - Licolub® H 12
    - Ceridust 3715

Amide Waxes
- Licowax C
- Ceridust 3910
- Licolub FA 1 powder vegetable based

Montan Waxes
- Licolub WE 4
- Licolub WE 40
- Licolub WM 31
- Licowax OP
- Licowax E
- Licomont® CaV 102
- Licomont NaV 101
- Ceridust 5551

Polyethylene Waxes
- Licocene PE MA 6252
- Licocene PE MA 6452
- Licocene PE MA 7452
- Licocene PE MA 4221
- Licocene PE MA 4351

Hydrocarbon Wax
- Licolub H 4

Highly modified Polymer Wax
- Licolub CE 2 TP
- Ceridust 8020 TP

Wax on renewable basis
- Licocare SBW 11 TP

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Improved Pigment Dispersion - Theory

Pigment Wetting
- Wax penetrates into the pigment agglomerates

Dispersion
- Wax breaks up of the pigment agglomerates

Stabilization
- Wax prevents pigment particles from reagglomeration
Dispersing Aids
- Influence of Particle Size

Pigment- and wax particles show similar sizes.

Wax particles are significantly bigger than pigment particles.

Energy input (e.g. high speed mixing, extrusion, kneading)

Wax particles acting as distance keeper between pigment particles and also providing an optimum pigment wetting.

Uneven and varying coating of pigment particles.
Particle Size of Clariant Waxes

- Granules/flakes particle size between 4 to ~10 mm

- fine grain particle size: \(< 2 \text{ mm}\)
  (mainly 0.6 – 0.8 mm)

- powder particle size: \(< 0.5 \text{ mm}\)
  (mainly 0.15 – 0.25 mm)

- Ceridust® particle size: \(< 0.025 \text{ mm}\)
  (mainly 10 – 20 µm)

recommended products
for the production of masterbatches
Particle Sizes – a Comparison
The Ceridust® Range – Micronized Waxes from Clariant

Ceridust®

Polyolefin Waxes
- non-polar
- polar
  - PE-wax
    - Ceridust® 130
    - Ceridust® 3620
  - oxidized PE-wax
    - Ceridust® 3715

Amide Waxes
- Ceridust® 3910

Montan Waxes
- Ceridust® 5551
  - New development: Highly polar modified Polymer Wax Ceridust 8020

Compound Waxes
- Ceridust® 9615 A
Improved Pigment Dispersion - Theory

The reduction of agglomerates of pigments within the masterbatch is one main key factor in order to get a fine dispersion

- Important:
  Every pigment is showing a self-reliant characteristic of dispersion – prediction of the dispersion behavior is hardly possible

- determination and comparison of pigment-dispersion-quality:

  - determination of the relative color yield (DIN 55986)
  - reduction in screen filter blockage / filter pressure value (DIN EN 13900-5)
  - determination of specks, spots and surface irregularities at the final article (e.g. blown film) (Clariant method)
Determination Methods
- Filter Pressure Value

- Filter pressure value (FPV) is defined as an increase of the pressure [bar] per pigment in [g] and will be calculated according to:

$$FPV = \frac{(p_{\text{max}} - p_s)}{m_c}$$

- The lower the FPV [bar/g] the finer the dispersion of the pigment-particles in the polymer.
Determination Methods
- Filter Pressure Value

- Typical pressure profile

1. initial stage
2. initial pressure
3. possible pressure decrease due to various rheological properties
4. variation of pressure dependent on test compound
5. rinsing process with 100g virgin polymer

\[ p \] pressure
\[ p_b \] initial pressure
\[ p_{max} \] maximum pressure
\[ t \] time
\[ t_s \] measurement of \( p_b \) and fill in of the test compound
\[ t_b \] complete feed end of test compound
\[ t_e \] end of the record and determination of \( p_{max} \)
Determination Methods
- Relative Color Yield

- Determination of relative color yield (DIN 55 986)

production of a masterbatch-compound for injection molding

production of the color yield plates

measurement of the color yield, (X,Y,Z values, or L*a*b* values DIN 53 235)
Determination Methods
- Relative Color Yield

- Determination of the relative color yield (DIN 55 986)
  - measurement of norm chromaticity (X,Y,Z) according e.g. DIN 53 235
  - calculation of rel. color yield according to:

  \[
  F = \frac{m_B}{m_P} \times 100
  \]

  - The color yield (F) in [%] above 100 % means a higher color yield intensity of the test sample
Determination Methods
- Evaluation of the Film Quality

– Evaluation of the film quality via film note

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<tr>
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<td>∞</td>
<td>5</td>
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Production of a masterbatch-compound for the film blowing line

Production of blown film virgin resin + 2 % of the masterbatches

Inspection of the film to identify undispersed particles

– The lower the number of specks, spots and surface irregularities, the finer the dispersion of the pigment-particles in the polymer
Trial Results with Ceridust in Masterbatch PE, PP and Engineering Resins

– Masterbatch quality tested according to before mentioned determination methods
  - Filter pressure value
  - Colour yield
  - Film quality

– for different pigments in different resins with different waxes

– Investigation of additional influences on the dispersion:
  - Amount of wax
  - Kind of wax
  - Hot mixture vs. cold mixture
  - Extruder throughput
Influence of Particle Size and Amount of Wax

- 30 % Fast blue A2R in LLDPE

Filter value [bar/g pigment]

without wax | 10 % Licowax PE 520 | 15 % Licowax PE 520 | 20 % Licowax PE 520 | 15 % PE 520 + Ceridust 3620 (2:1) | 20 % PE 520 + Ceridust 3620 (2:1)

Filter value: DIN-EN 13900-5
Filter: 40 µm, 725 mesh
Premix: cold mix

Improved dispersion due to finer particle size and higher amount of wax
Influence of Particle Size and Amount of Wax in LLDPE

- 40 % Pigment Red 57:1 + 30 % LLDPE (MFR 25)
Influence of Extruder Throughput on the Dispersion

- 40 % Pigment Green 7 + 30 % LLDPE (MFR 25)

⇒ Lower Throughput = Better Dispersion

Filter value [bar/g pigment]

<table>
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<th>Throughput</th>
<th>15 kg/h</th>
<th>18 kg/h</th>
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<tbody>
<tr>
<td>30% Licowax PE 520 powder</td>
<td>1.2</td>
<td>1.0</td>
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<tr>
<td>30% PE 520 powder + Ceridust 3620 (2:1)</td>
<td>0.8</td>
<td>0.6</td>
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<tr>
<td>30% Licowax PE 520 powder</td>
<td>1.4</td>
<td>1.2</td>
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<tr>
<td>30% PE 520 powder + Ceridust 3620 (2:1)</td>
<td>1.0</td>
<td>0.8</td>
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</table>

Film surface irregularities (specs) [in number]

Filter value: DIN-EN 13900-5
Filter: 14 µm, 724 mesh
Premix: cold mix
Film note: Clariant norm
Influence of the Particle Size

- Highly crystalline, low melting, low viscous, micronized PP wax gives the best results regarding dispersion in contrast to other PP waxes.

Formulation: 40% Pigm.Red 48:2 (e.g. Lithol Scharlach K 4461); 20 - 30% wax; 30 - 40% PP, MFR 30.
Extruder: ZE25x40D co-rotating; temp. profile: 180-200-190-170-150-170-190-180-175°C; screw speed: 500 rpm; throughput: 5.0 kg/h; main feed.
Influence of the Particle Size

- 40 % Pigment Yellow 155 + 40 % PP HG 245 (Borealis)

Filter value [bar/g Pigment]

- 20 % PP-Wax (1700 mPas)
- 20 % Licocene PP 6102 FG
- 13 % PP 6102 FG + 7 % Ceridust 6050 M
- 13 % PP 1302 + 7 % Ceridust 6050 M

Filter value: DIN-EN 13900-5
Filter: 14 µm, 725 mesh
Premix: hot mixing
Film note: Clariant norm
Influence of the Particle Size

- 30 % Pigment Violett 19 + 40 % PP HG 245 (Borealis)

Filter value [bar/g Pigment]

Rel. color yield [%]

without wax

30 % PP-Wax (1700 mPas)
+ Ceridust 6050M (2:1)

30 % Licocene PP 6102 +
Ceridust 6050M (2:1)

Filter value

Color yield

filter value: DIN-EN 13900-5
filter: 25 µm
premix: cold mixing
color yield: DIN 55986
Influence of the Particle Size

- 40 % Pigment Green 7 + 45 % PP HG 245 (Borealis)

Filter value [bar/g Pigment]

rel. color yield [%]

15% Standard PP wax FG

10 % Licocene PP 6102 + Ceridust 6050M (2:1)

15% Licocene PP 6102 + Ceridust 6050 M (2:1)

filter value: DIN-EN 13900-5
filter: 25 µm
premix: hot mixing
color yield: DIN 55986
Influence of Hot and Cold Mix on Dispersion

- 40% Pigment PV Fast Violet RL (violet 23) + 30% PP powder (MFI 25 g/10 min)

<table>
<thead>
<tr>
<th>Filter value [bar/g pigment]</th>
<th>Color yield [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>20% Licocene PP 6102 FG</td>
<td>100</td>
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<tr>
<td></td>
<td>110</td>
</tr>
<tr>
<td></td>
<td>120</td>
</tr>
<tr>
<td>13% PP 6102 FG + 7% Ceridust 6050 M</td>
<td>95</td>
</tr>
<tr>
<td>13% PP 1302 FG + 7% Ceridust 6050 M</td>
<td>100</td>
</tr>
<tr>
<td>13% PP 6102 FG + 7% Ceridust 6050 M</td>
<td>105</td>
</tr>
<tr>
<td>13% PP 6102 FG + 10% Ceridust 6050 M</td>
<td>110</td>
</tr>
<tr>
<td>20% PP 6102 FG + 10% Ceridust 6050 M</td>
<td>115</td>
</tr>
<tr>
<td></td>
<td>120</td>
</tr>
</tbody>
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Filter value: DIN-EN 13900-5  
Filter: 14 µm, 724 mesh  
Color yield: DIN 55986
Improved Dispersion with Micronized Waxes in Engineering Resins - Polycarbonate

– 17 % Pigment Green 7 + 80 % PC powder

highest color yield with mixture of Licowax E & Ceridust 5551

lowest correlated haze with Ceridust 5551

premix: cold mixing

color yield: DIN 55986
Improved Dispersion with Micronized Waxes in Engineering Resins - Polycarbonate

- TEM photographs of polycarbonate + 17 % Pigment Green GNX samples
- Magnification 4000

Sample preparation: Ultrafine sections of granules, thickness 60-80 nm; Cutter type Ultracut E (manufacturer Reichert+Jung, Germany)
Improved Dispersion with Micronized Waxes in Engineering Resins - Polyester PET

- 30 % Pigment Green GNX + 70-65 % PET milled (intrinsic viscos. ~0,63)

![Graph showing filter value and color yield for different pigments and waxes.]

Filter value: DIN-EN 13900-5
Filter: 14 µm, 724 mesh
Color yield: DIN 55986
# Licolub® CE 2 TP and Ceridust 8020 TP as a Potential Alternative to Montan Waxes

- 30 % Pigment Green 7 + 65 % PET (Bripet 2000 BST)

<table>
<thead>
<tr>
<th></th>
<th>Filter value [bar/g]</th>
<th>Color yield [%] (2 % in Polyclear 3304)</th>
</tr>
</thead>
<tbody>
<tr>
<td>without wax</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 % Ceridust 8020 TP</td>
<td>10</td>
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<td>5 % Licolub CE 2 TP</td>
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<td>5 % Licolub WE 4</td>
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<td>120</td>
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<tr>
<td>5 % Ceridust 5551</td>
<td>10</td>
<td>110</td>
</tr>
</tbody>
</table>

Filter value: DIN-EN 13900-5  
Filter: PZ 14  
Premix: cold mixing  
Color yield: DIN 55986
Improved Dispersion with Micronized Waxes in Engineering Resins – Polyamide 6

– 30 % Pigment Blue 15:1 A2R + 70 - 65 % PA milled

Filter value [bar/g pigment]

Filter value: DIN-EN 13900-5
Filter: 25 µm, 614 mesh

Color yield: DIN 55986

Extrusion: Barrel temp. 250-220-225-230-255-260-240°C, 500 rpm, 6.0 kg/h, cold mix - main dosed

VN 82358 - 82367
Summary

Ceridust® - products, which meet your demands and bring your ideas to success

- Micronized waxes provide important advantages in masterbatch preparation like:
  - economical processing and cost compensation
  - increased colour yield, reduced amount of (expensive) organic pigments necessary
  - reduction of the agglomerates
  - reduction in screen filter blockage
  - reduction of specks, spots and surface defects
  - suitable for the wide variety of pigments
  - suitable micronized wax grades for every polymer

always a step ahead with:

Ceridust®
Thank you for your attention -

Questions?