Improved Pigment Dispersion with Micronized Wax

CLARIANT

Public

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BU Additves
Technical Marketing Waxes
13.05.2013

what is precious to you?

Content:

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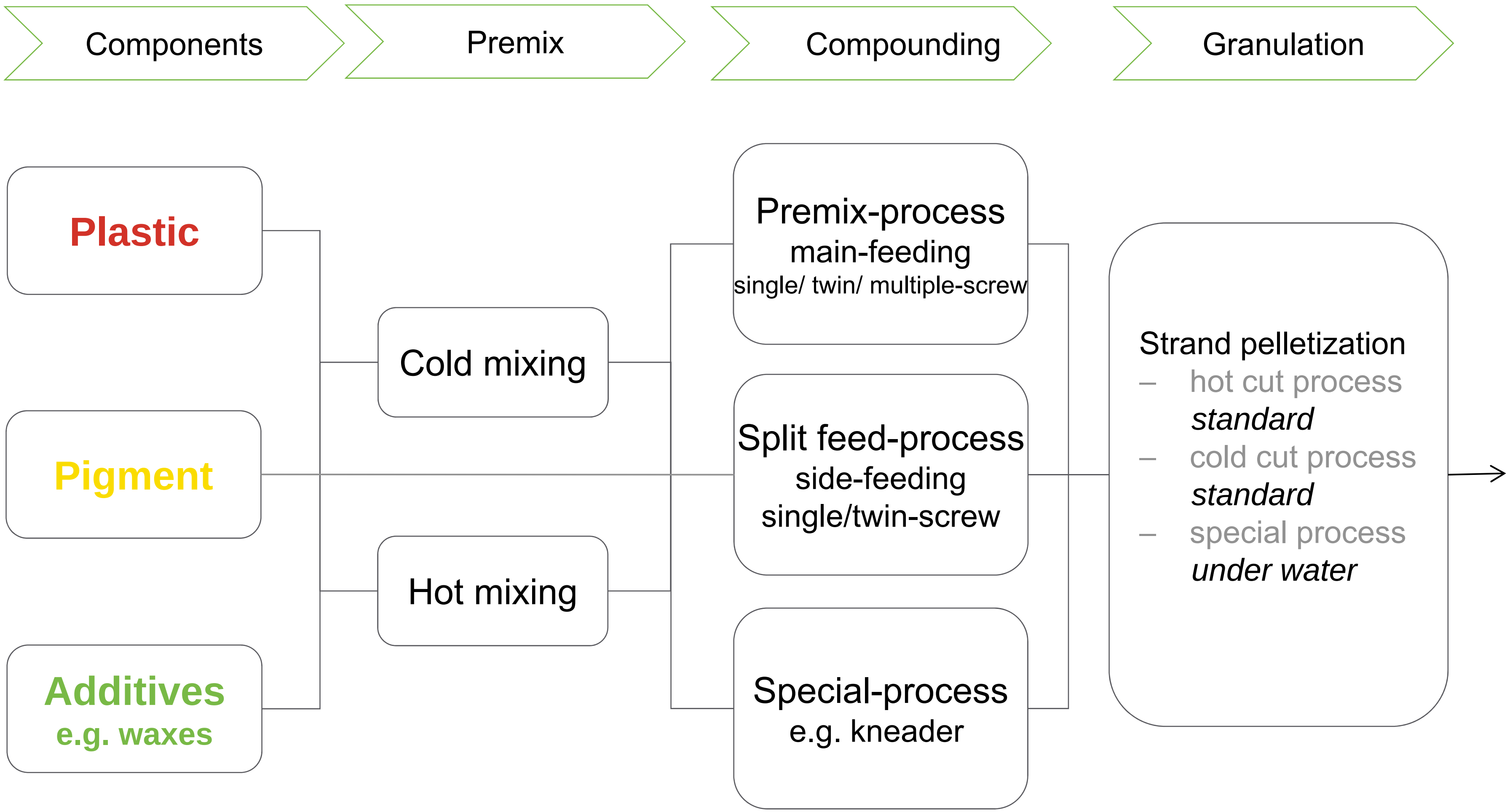
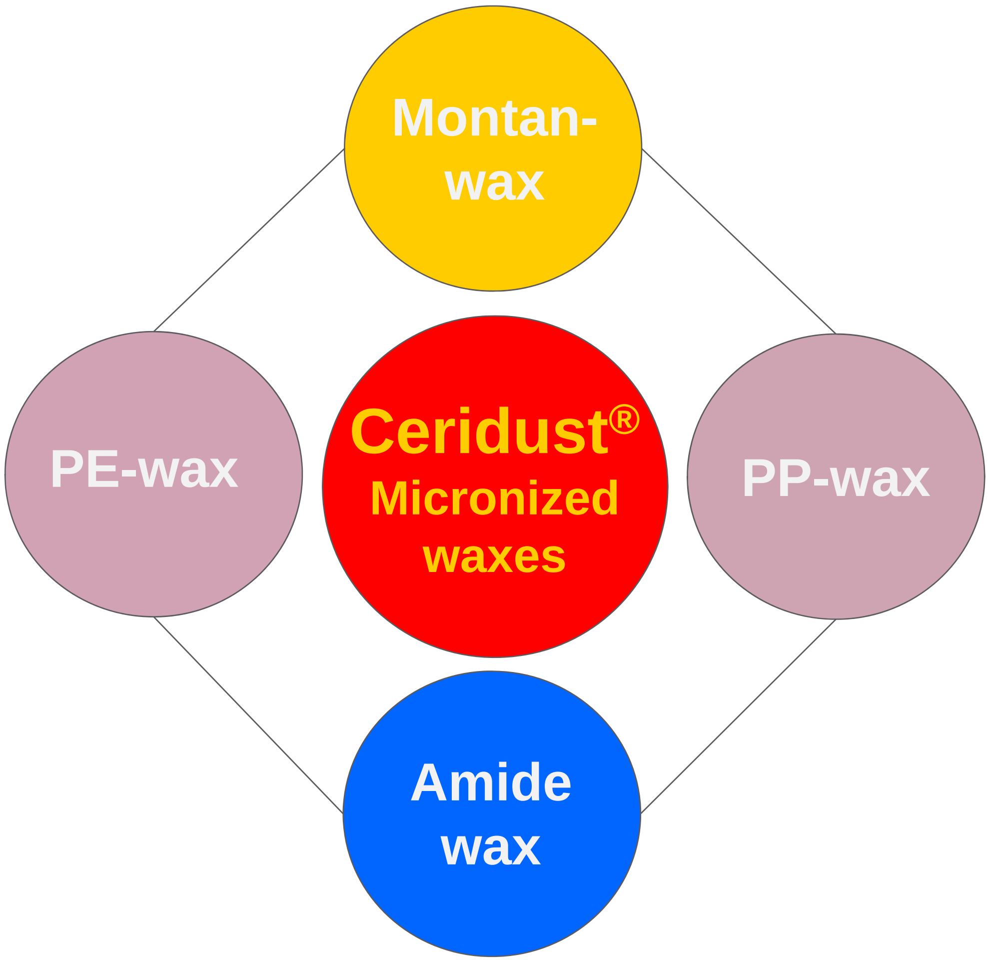
Processes of Masterbatch Preparation - a Simplified Scheme



Clariant Waxes for Masterbatch Applications



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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**
    - 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
      - Liocwax PE 190
      - Licocene PE 4201
      - Ceridust 3620
  - **polar**
    - oxidized Polyethylene Waxes
      - Licowax® PED 191
      - Liocwax PED 521
      - Licolub® H 12
      - Ceridust 3715
    - modified Polypropylene/ Polyethylene Waxes
      - Licocene PP MA 6252
      - Licocene PP MA 6452
      - Licocene PP MA 7452
      - Licocene PE MA 4221
      - Licocene PE MA 4351
- **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
- **Hydrocarbon Wax**
  - Licolub H 4
- **highly modified Polymer Wax**
  - Licolub CE 2 TP
  - Ceridust 8020 TP
- **Wax on renewable basis**
  - Licocare SBW 11 TP

Christian Lechner, BU Additves, Technical Marketing Waxes, 13.05.2013

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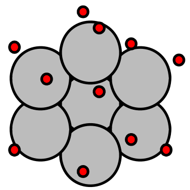
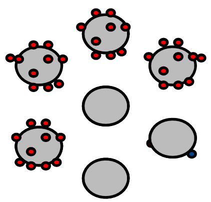
Improved Pigment Dispersion - Theory

Pigment Wetting



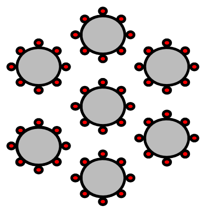
Wax penetrates into the pigment agglomerates

Dispersion



Wax breakes up of the pigment agglomerates

Stabilization



Wax prevents pigment particles from reagglomeration

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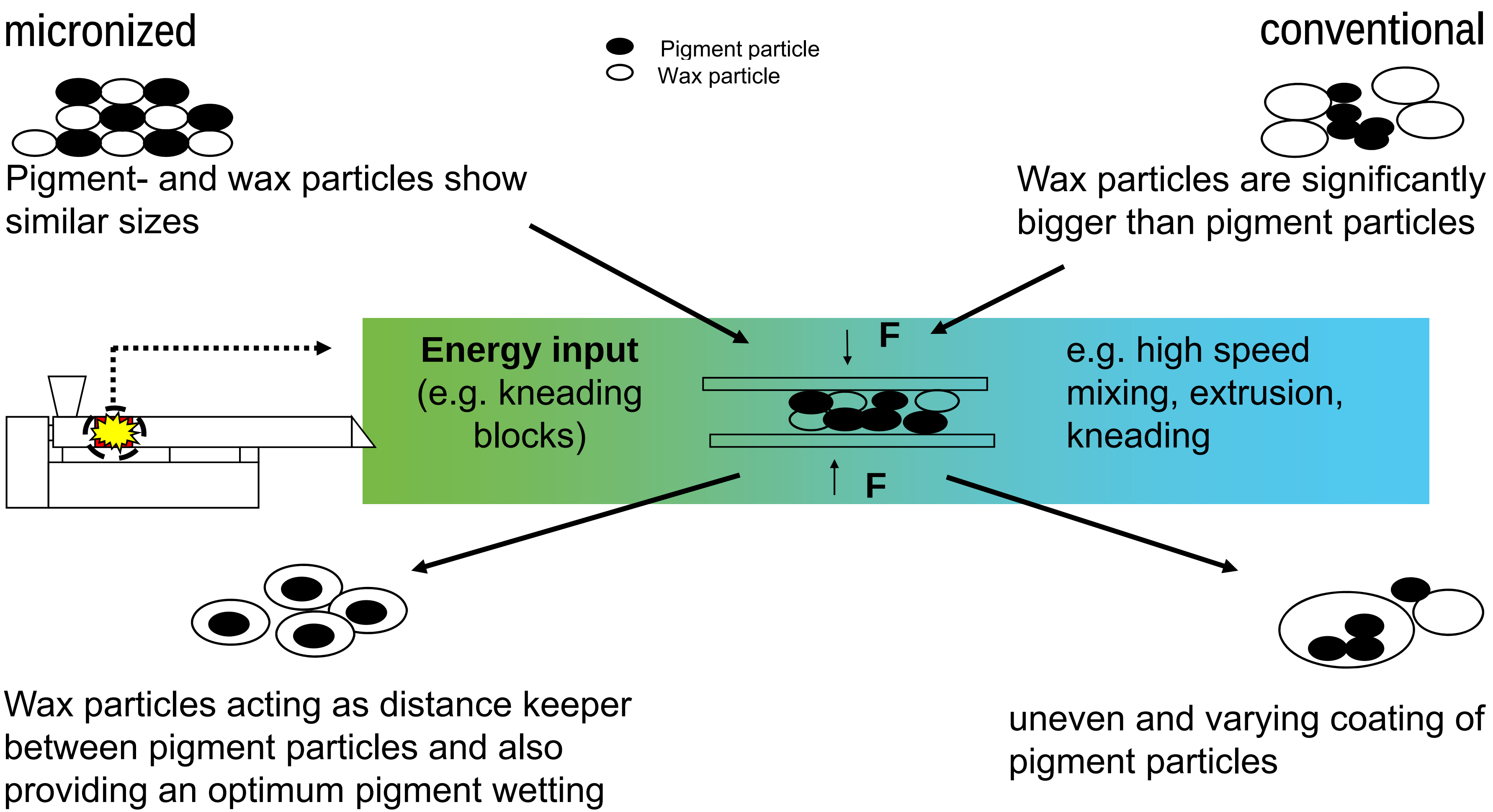
Dispersing Aids
- Influence of Particle Size



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Particle Size of Clariant Waxes

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

– fine grain particle size: < 2 mm
(mainly 0,6 – 0,8 mm)

– powder particle size: < 0,5 mm
(mainly 0,15 – 0,25 mm)

– Ceridust® particle size: < 0,025 mm
(mainly 10 – 20 µm)

recommended products for the production of masterbatches

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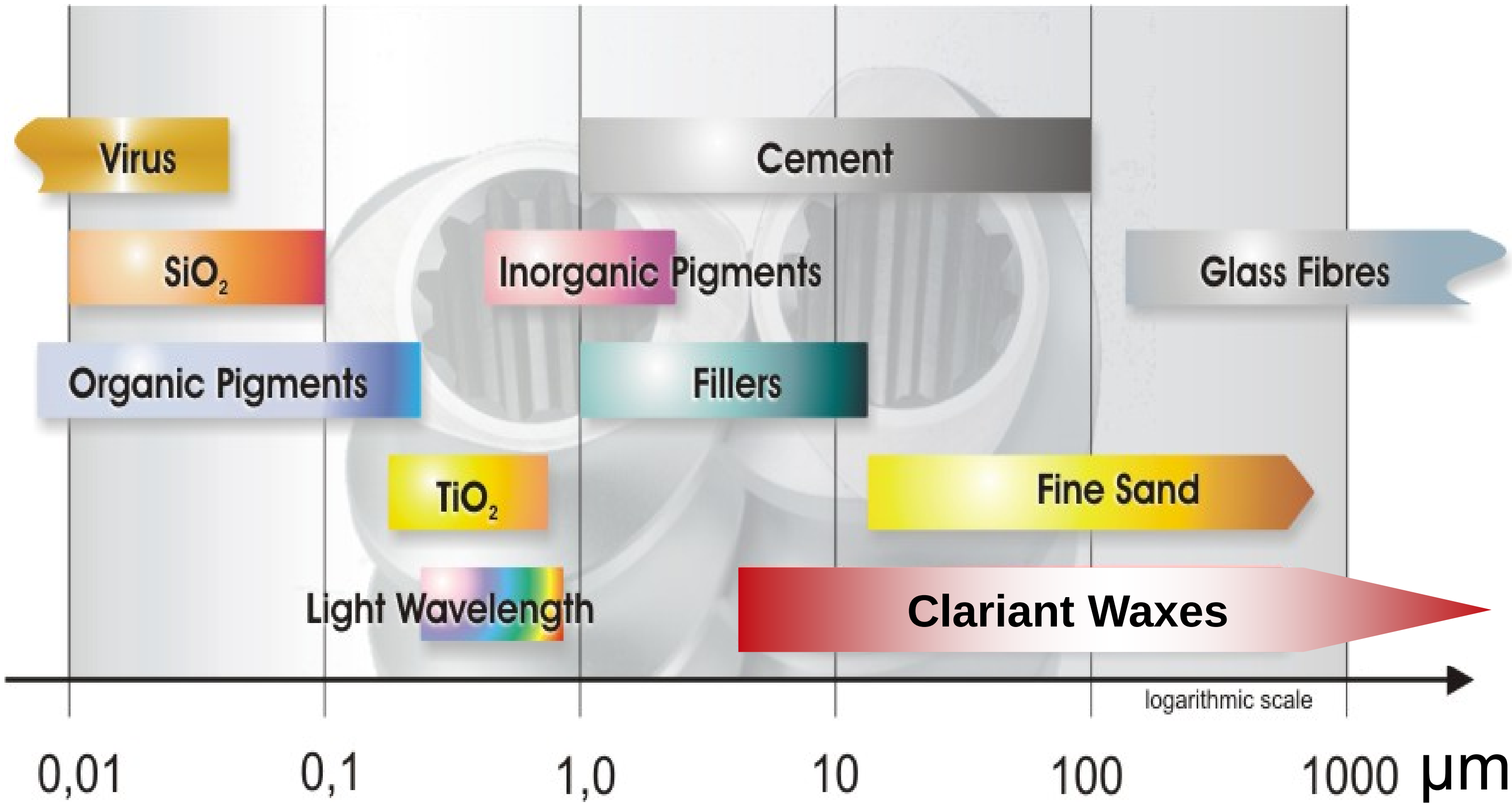
Particle Sizes – a Comparison



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The Ceridust® Range –Micronized Waxes from Clariant

Ceridust®

- **Polyolefin Waxes**
  - *non-polar*
    - PE-wax
      - **Ceridust® 130**
      - **Ceridust® 3620**
    - PP-wax
      - **Ceridust® 6050 M TP**
  - *polar*
    - oxidized PE-wax
      - **Ceridust® 3715**
- **Compound-Waxes**
  - **Ceridust® 9615 A**
- **Amide Waxes**
  - **Ceridust® 3910**
- **Montan Waxes**
  - **Ceridust® 5551**
  - **New development:**
  - **Highly polar modified Polymer Wax Ceridust 8020**

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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)

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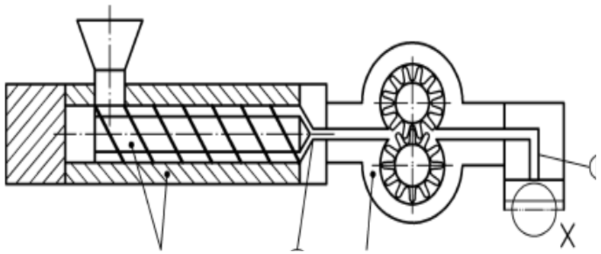
Determination Methods - Filter Pressure Value

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

$$FPV = (p_{max} - p_s) / m_c$$

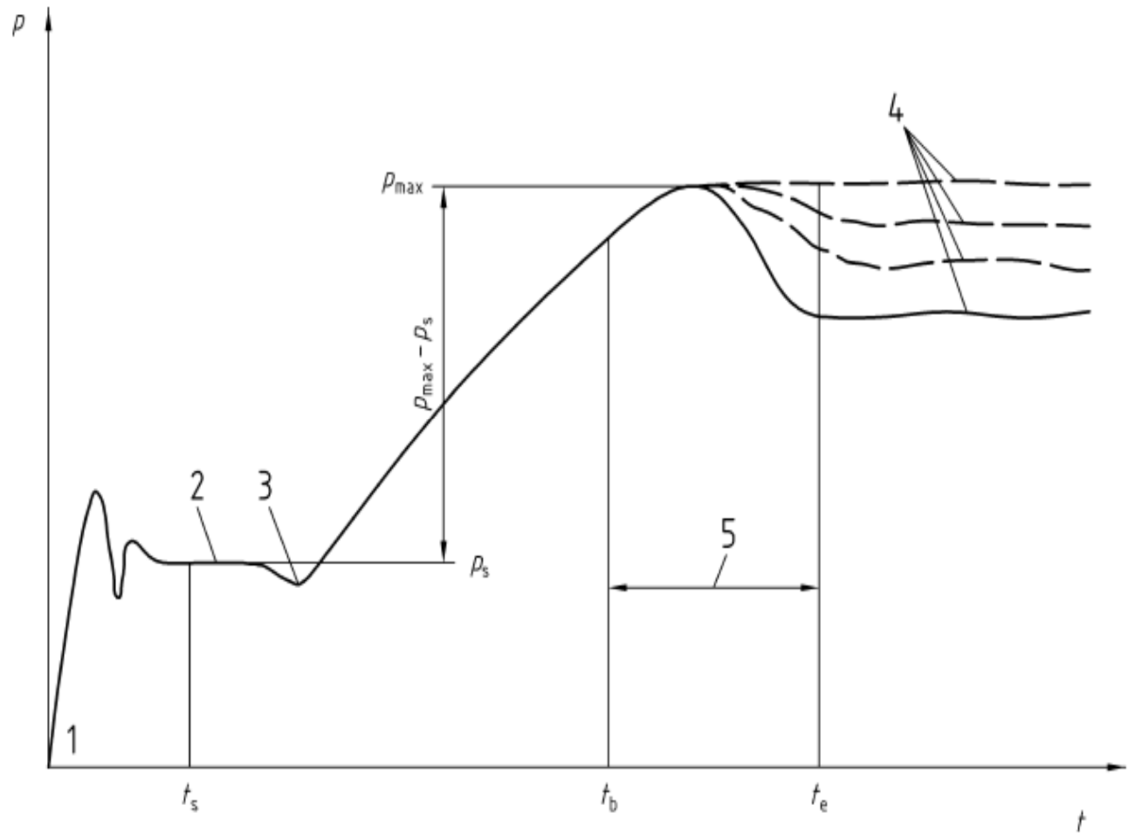
FPV	filter pressure value in [bar/g]
p_s	initial pressure in [bar]
p_{max}	maximum pressure in [bar]
m_C	inserted amount of pigment in [g]

– **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

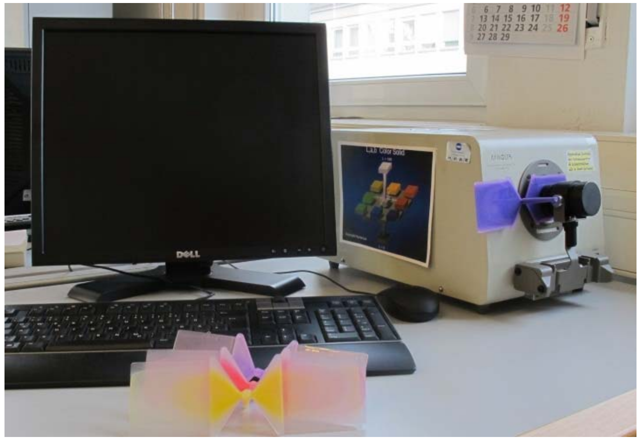
p	pressure
p_s	initial pressure
p_{max}	maximum pressure
t	time
t_S	measurement of p_s and fill in of the test compound
t_b	complete feed end of test compound
t_e	end of the record and determination of p_{max}

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)

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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:

$$F = m_B/m_P * 100$$

F	rel. color yield in [%]
m_B	amount of color pigment (base) in [g]
m_P	amount of color pigment (test sample) in [g]

– **The color yield (F) in [%] above 100 % means a higher color yield intensity of the test sample**

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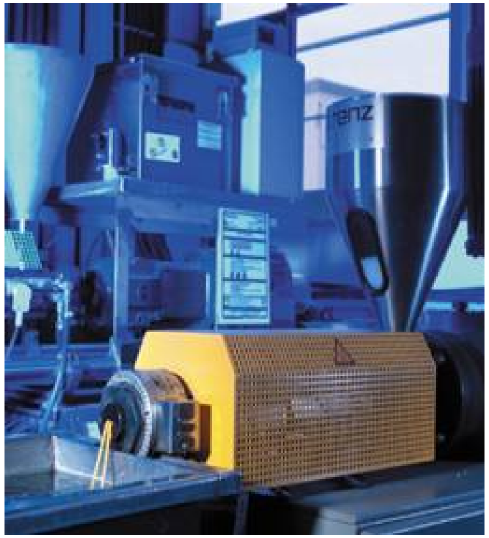
Determination Methods
- Evaluation of the Film Quality

– Evaluation of the film quality via film note



Production of a masterbatch-compound for the film blowing line

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

from	Up	note
0	5	1
6	10	1-2
11	100	2
101	200	2-3
201	300	3
301	400	3-4
401	600	4
601	1000	4-5
1001	∞	5

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**

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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

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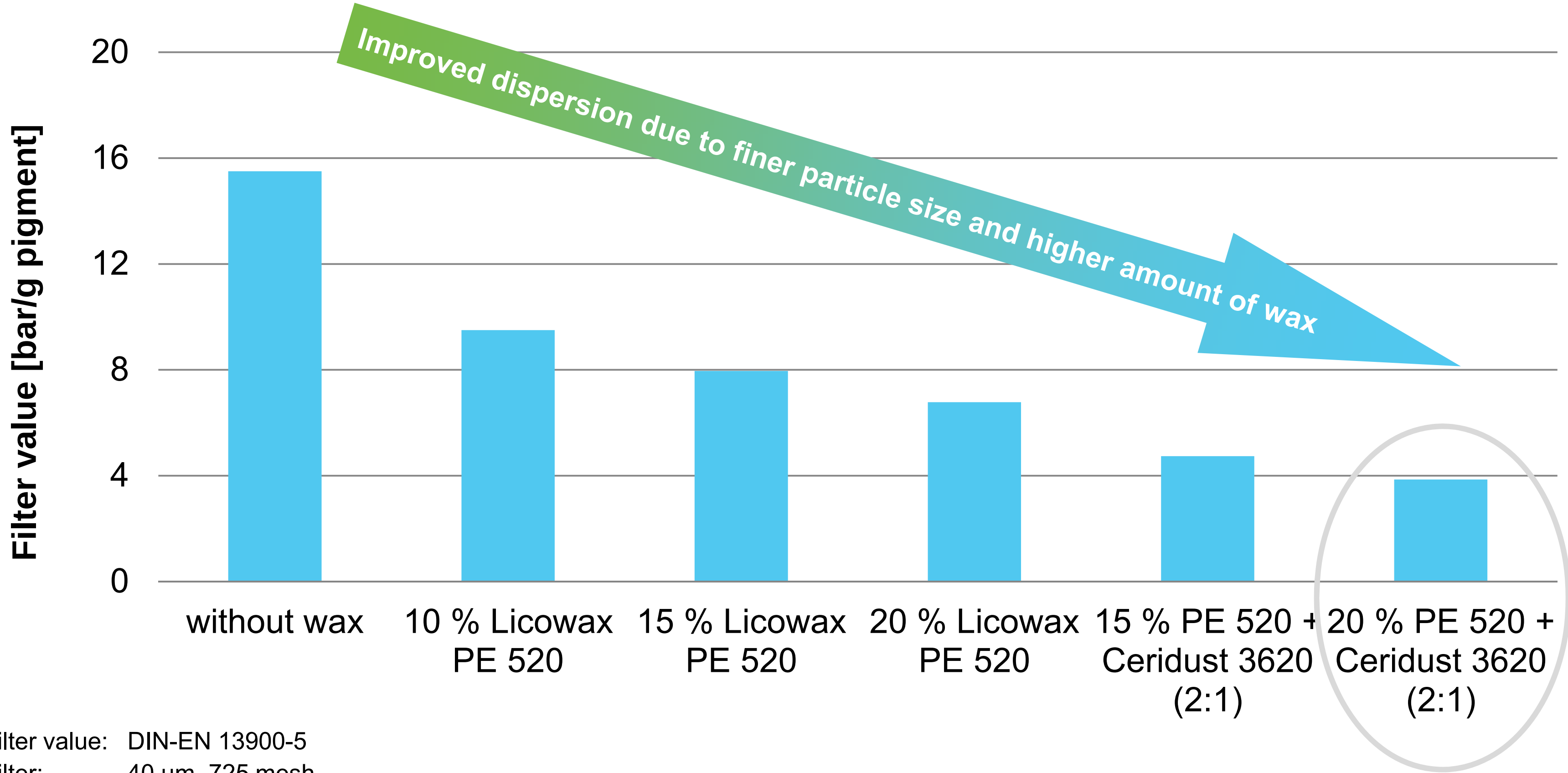
Influence of Particle Size and Amount of Wax

– 30 % Fast blue A2R in LLDPE



filter value: DIN-EN 13900-5
filter: 40 µm, 725 mesh
premix: cold mix

VN 86931 - 86941

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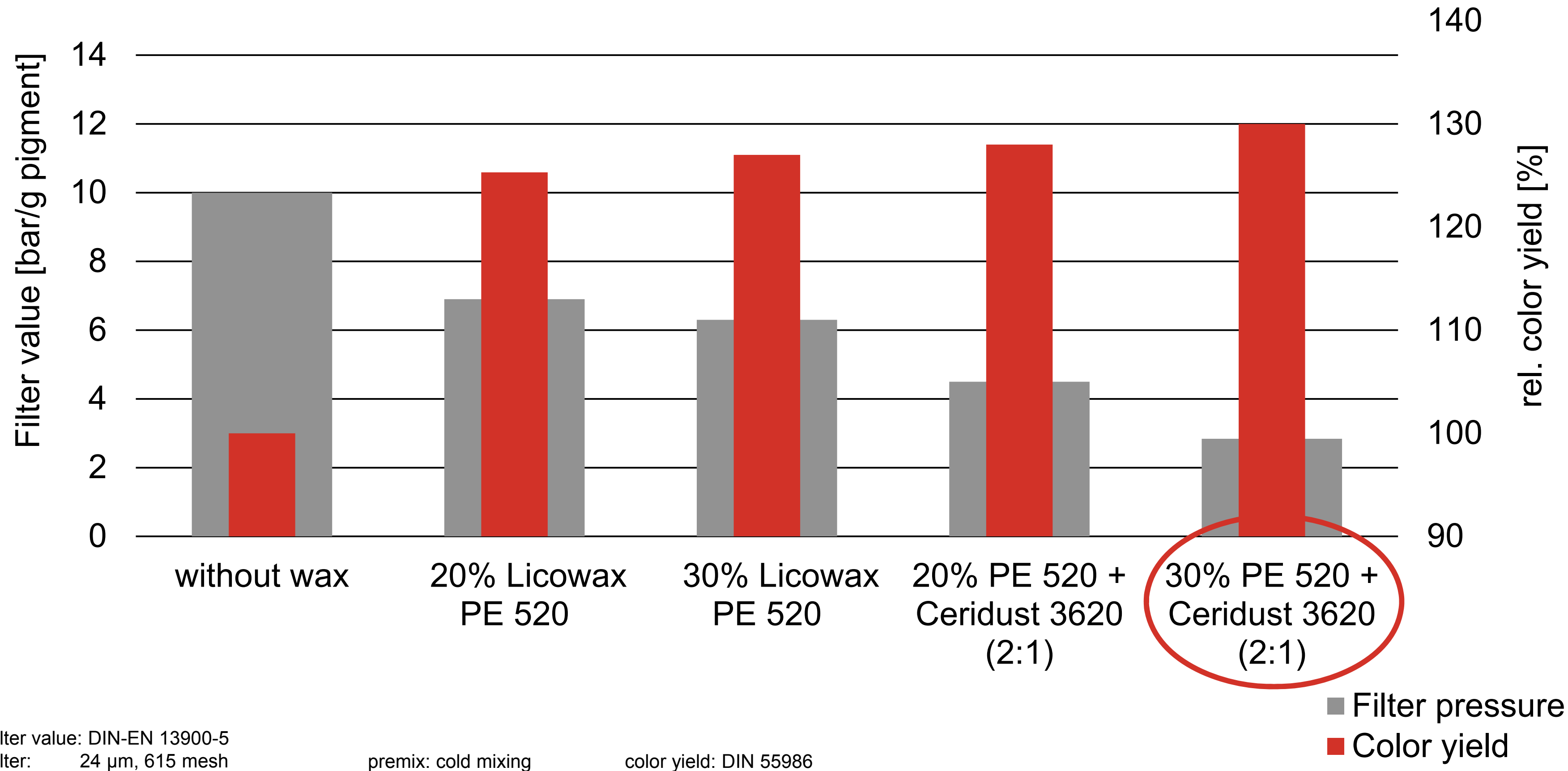
Influence of Particle Size and Amount of Wax in LLDPE

– 40 % Pigment Red 57:1 + 30 % LLDPE (MFR 25)



filter value: DIN-EN 13900-5
filter: 24 µm, 615 mesh

premix: cold mixing

color yield: DIN 55986

VN 86196- 86203

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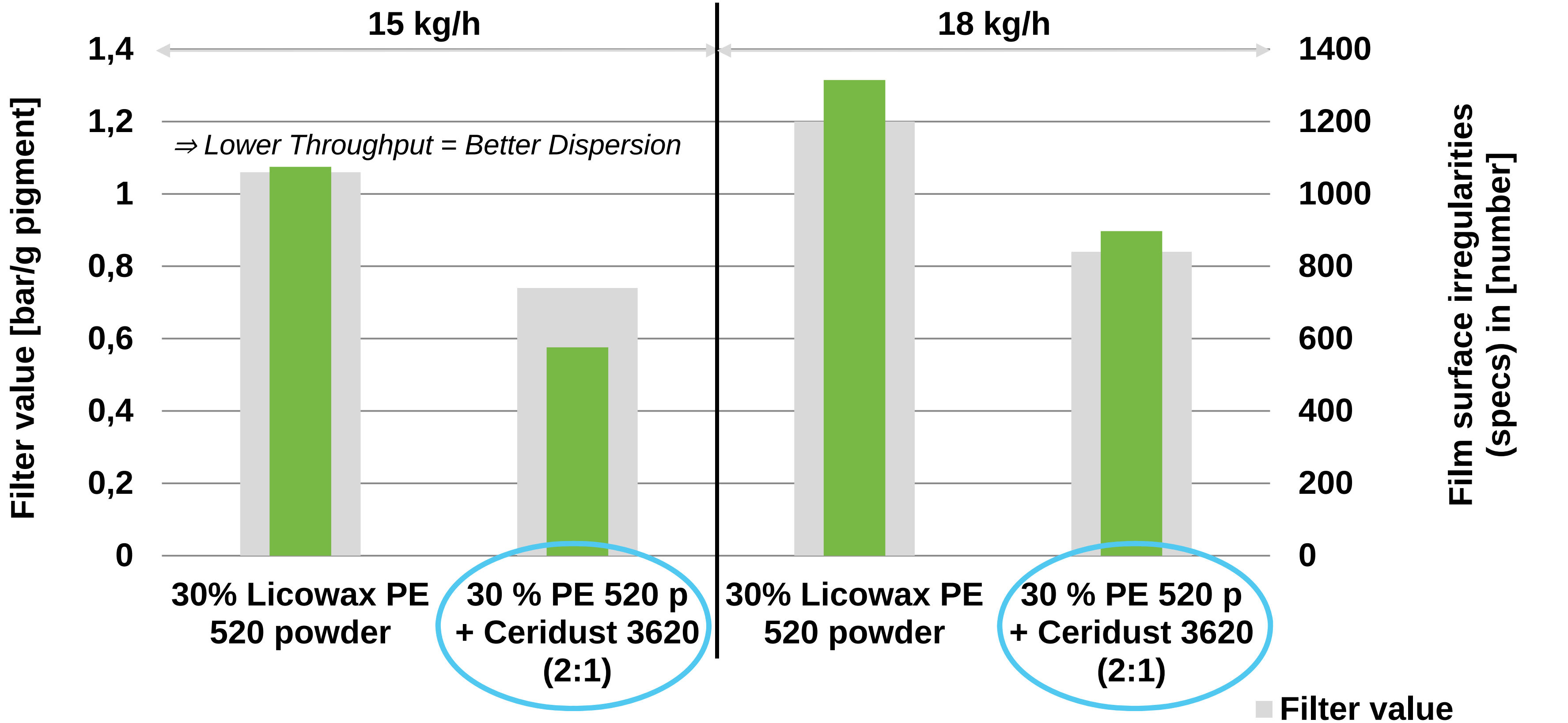
Influence of Extruder Throughput on the Dispersion

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



filter value: DIN-EN 13900-5
filter: 14 µm, 724 mesh

premix: cold mix

film note : Clariant norm

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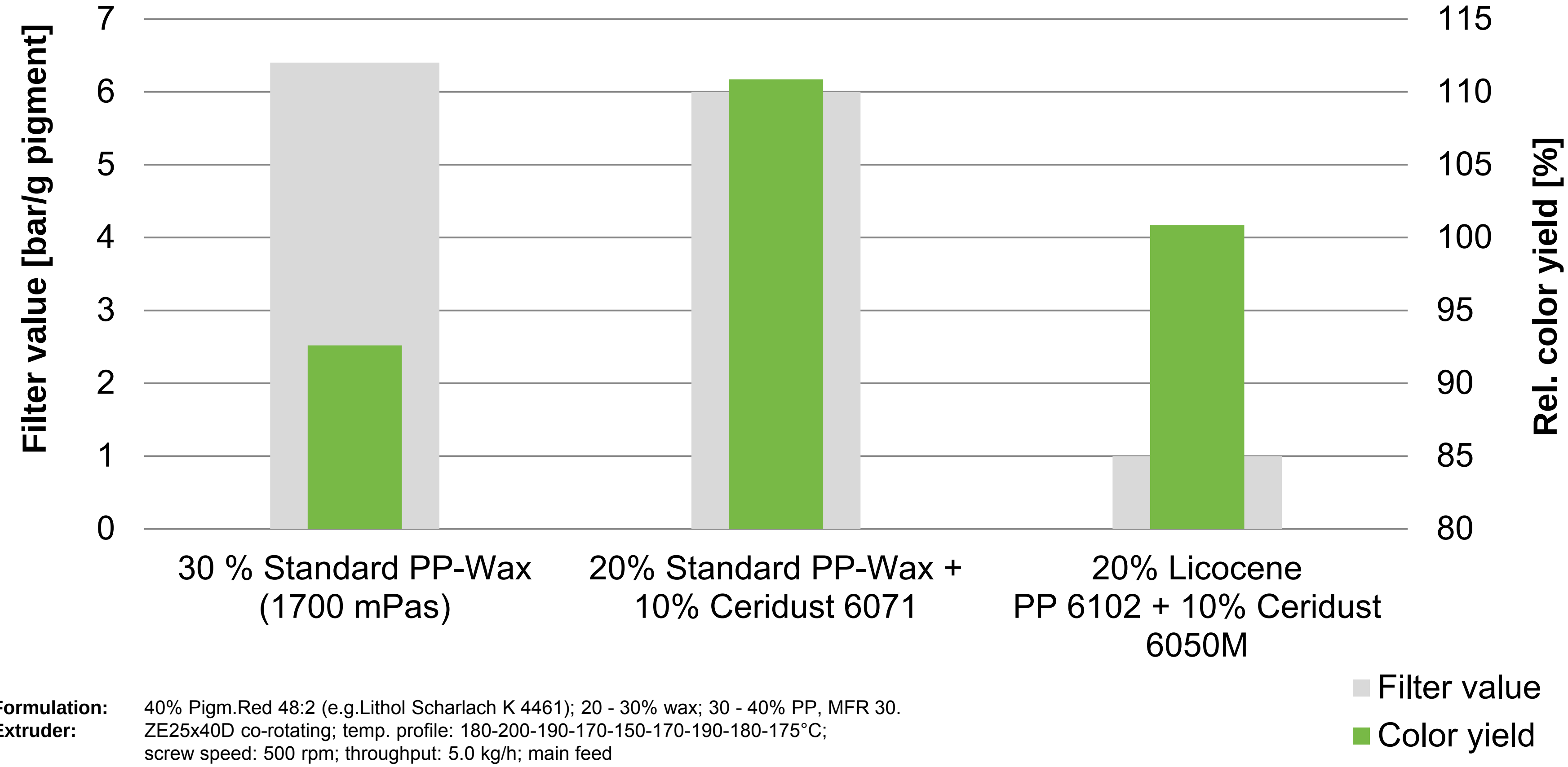
Influence of the Particle Size

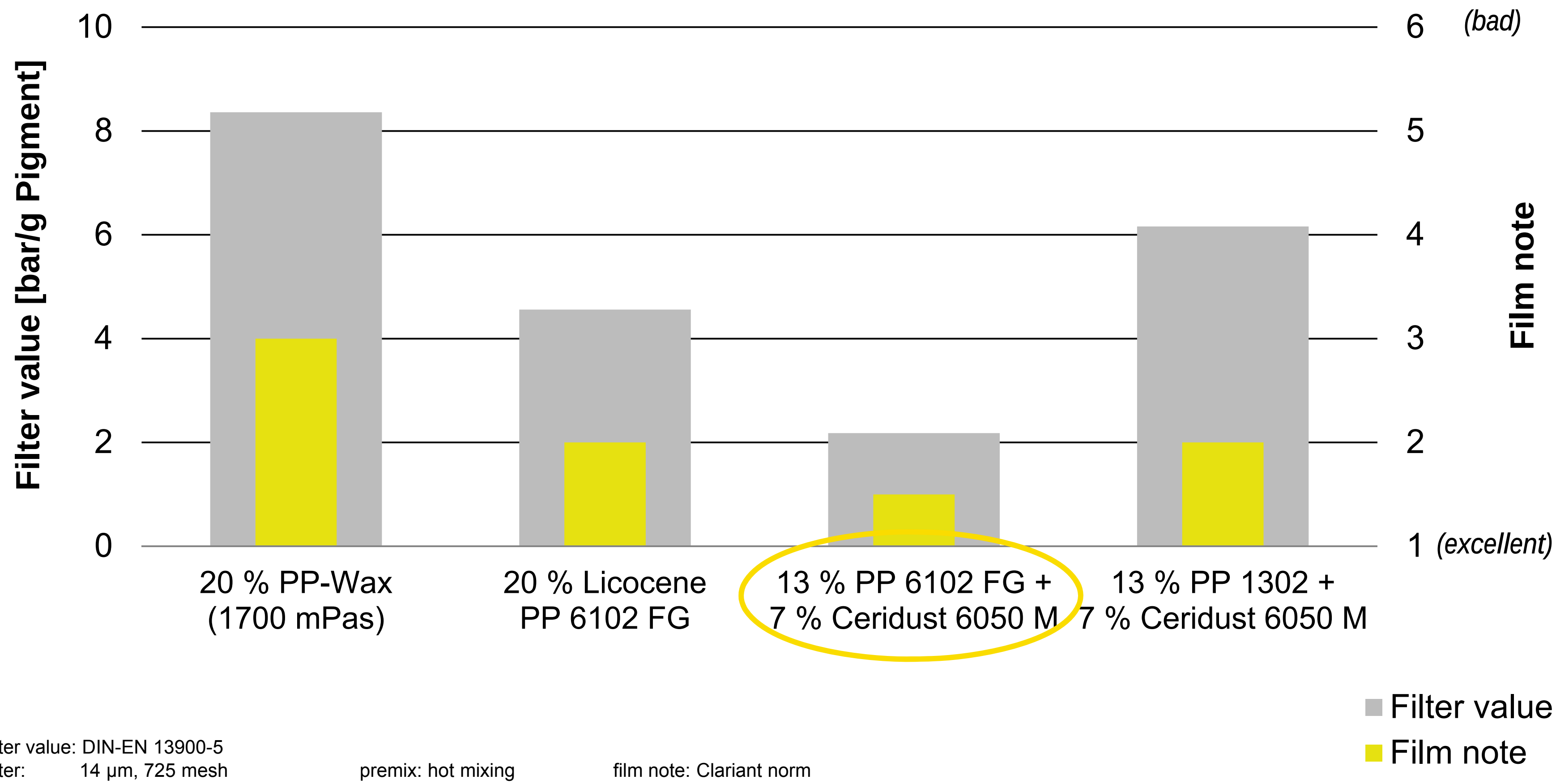
– Highly crystalline, low melting, low viscous, micronized PP wax gives the best results regarding dispersion in contrast to other PP waxes



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: DIN-EN 13900-5
filter: 14 µm, 725 mesh

premix: hot mixing

film note: Clariant norm

VN 94186 – 94201 // 94295 – 298

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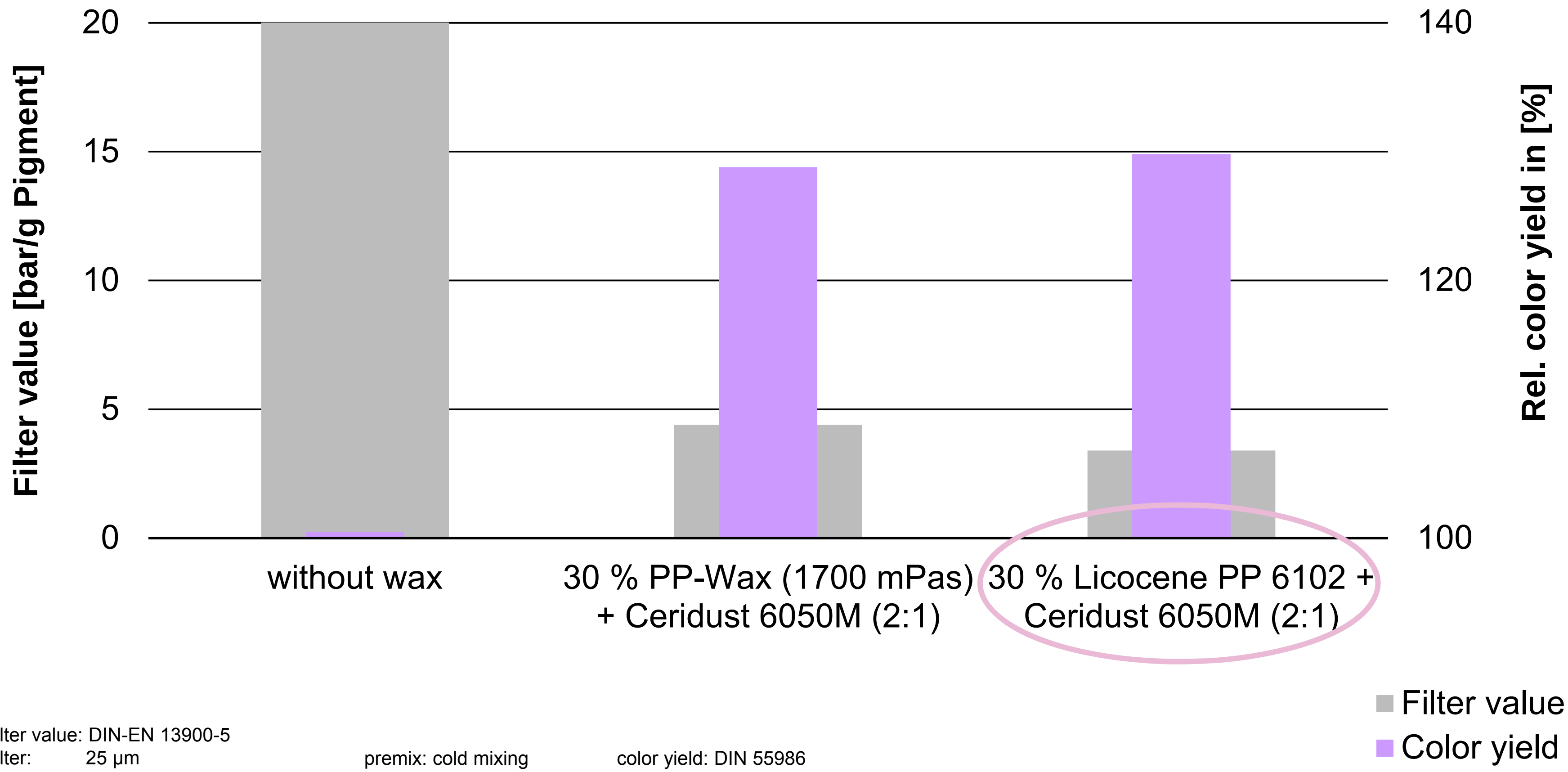
Influence of the Particle Size

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



filter value: DIN-EN 13900-5
filter: 25 µm

premix: cold mixing

color yield: DIN 55986

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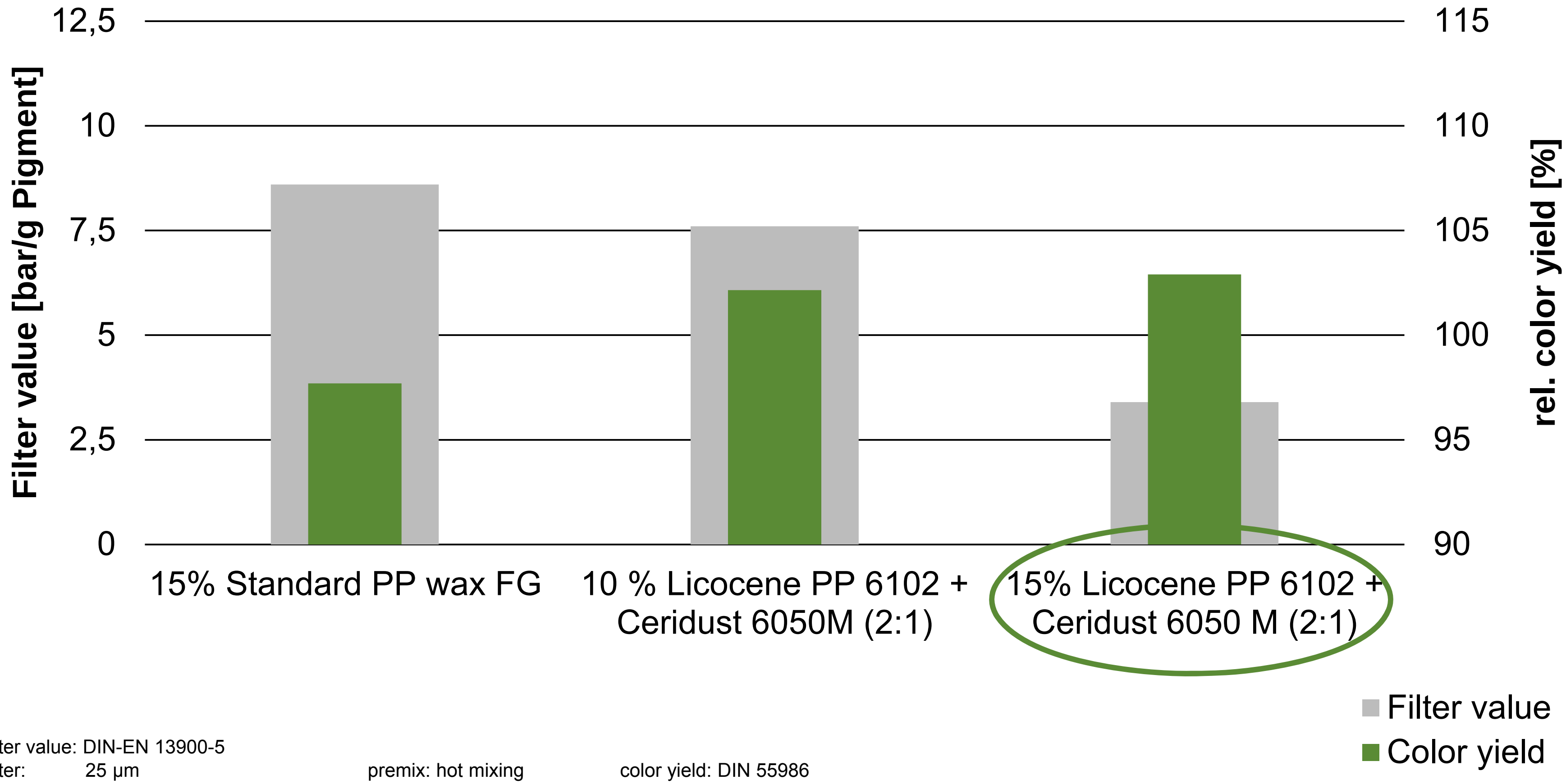
Influence of the Particle Size

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



filter value: DIN-EN 13900-5
filter: 25 µm
premix: hot mixing
color yield: DIN 55986

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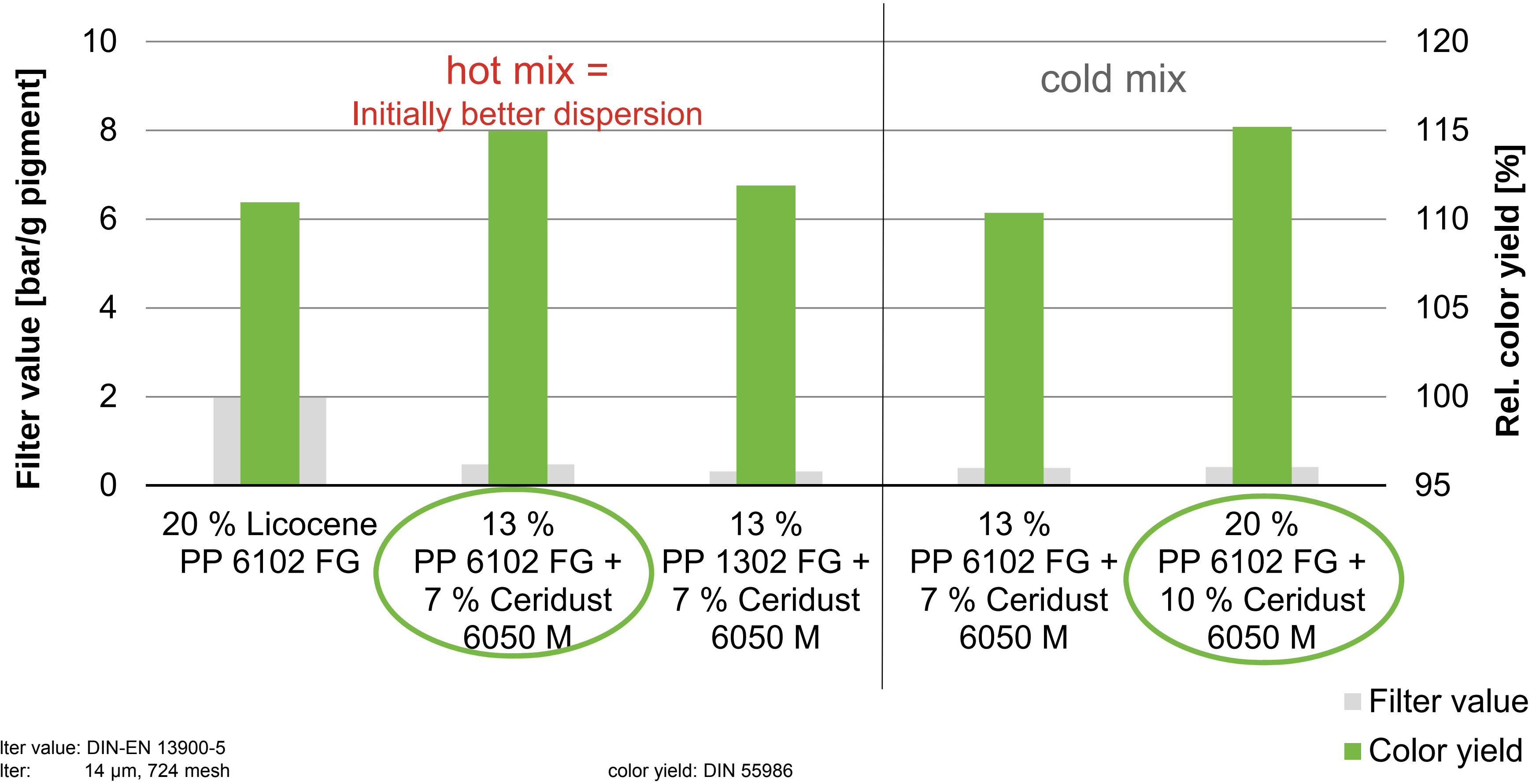
Influence of Hot and Cold Mix on Dispersion

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



filter value: DIN-EN 13900-5
filter: 14 µm, 724 mesh

color yield: DIN 55986

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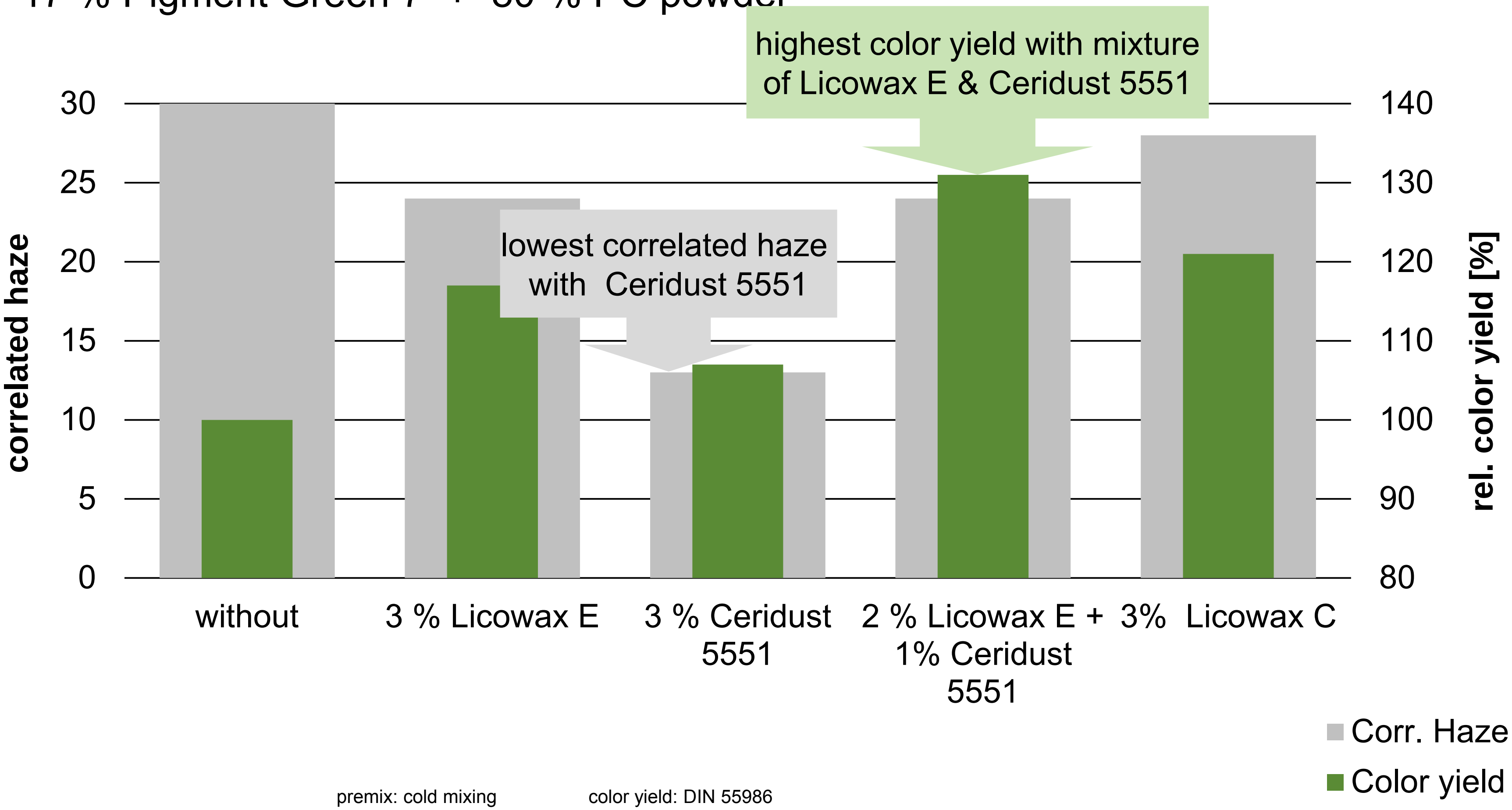
Improved Dispersion with Micronized Waxes in Engineering Resins - Polycarbonate

– 17 % Pigment Green 7 + 80 % PC powder



premix: cold mixing color yield: DIN 55986

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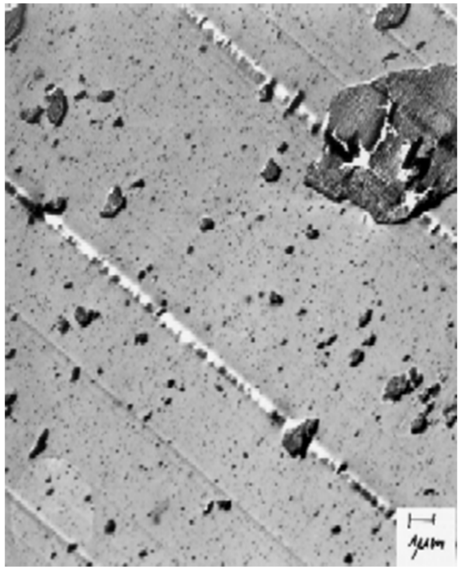
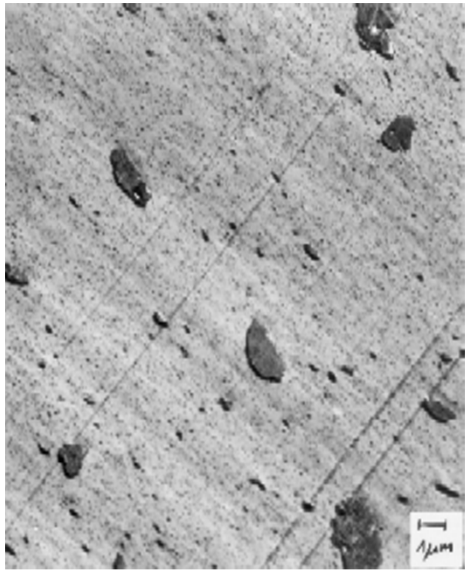
Improved Dispersion with Micronized Waxes in Engineering Resins - Polycarbonate

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



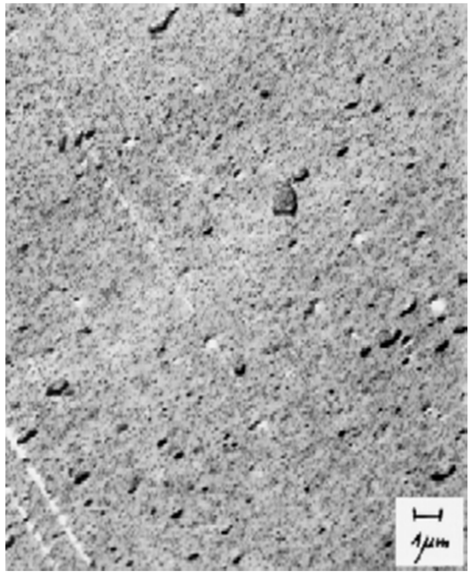
No wax

(colour yield = **100 %**)



3 % Licowax E

(colour yield =**117 %**)



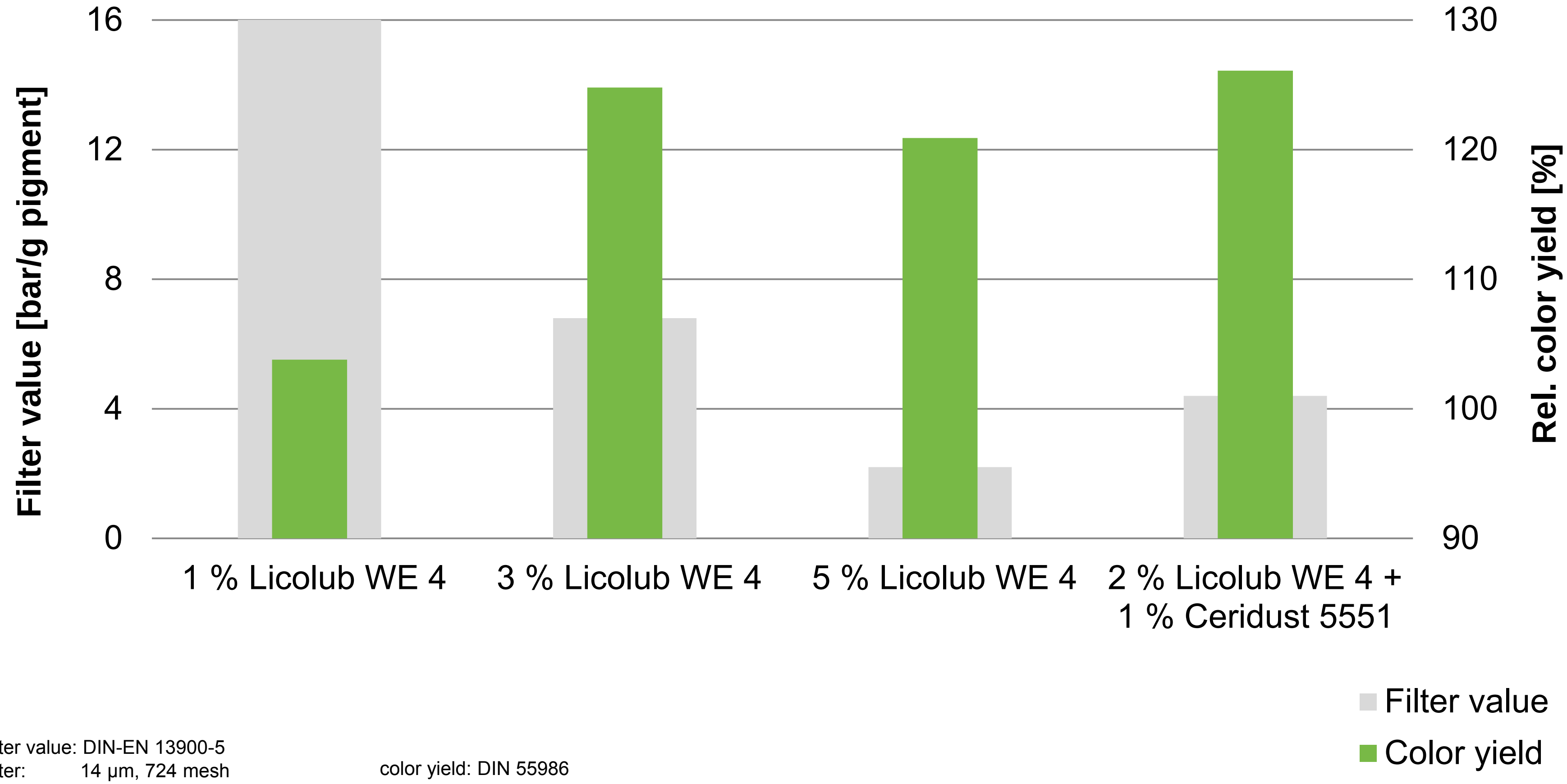
2 % Licowax E
1 % Ceridust 5551

(colour yield = **131 %**)

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)



filter value: DIN-EN 13900-5
filter: 14 µm, 724 mesh

color yield: DIN 55986

VN 80211 - 80230

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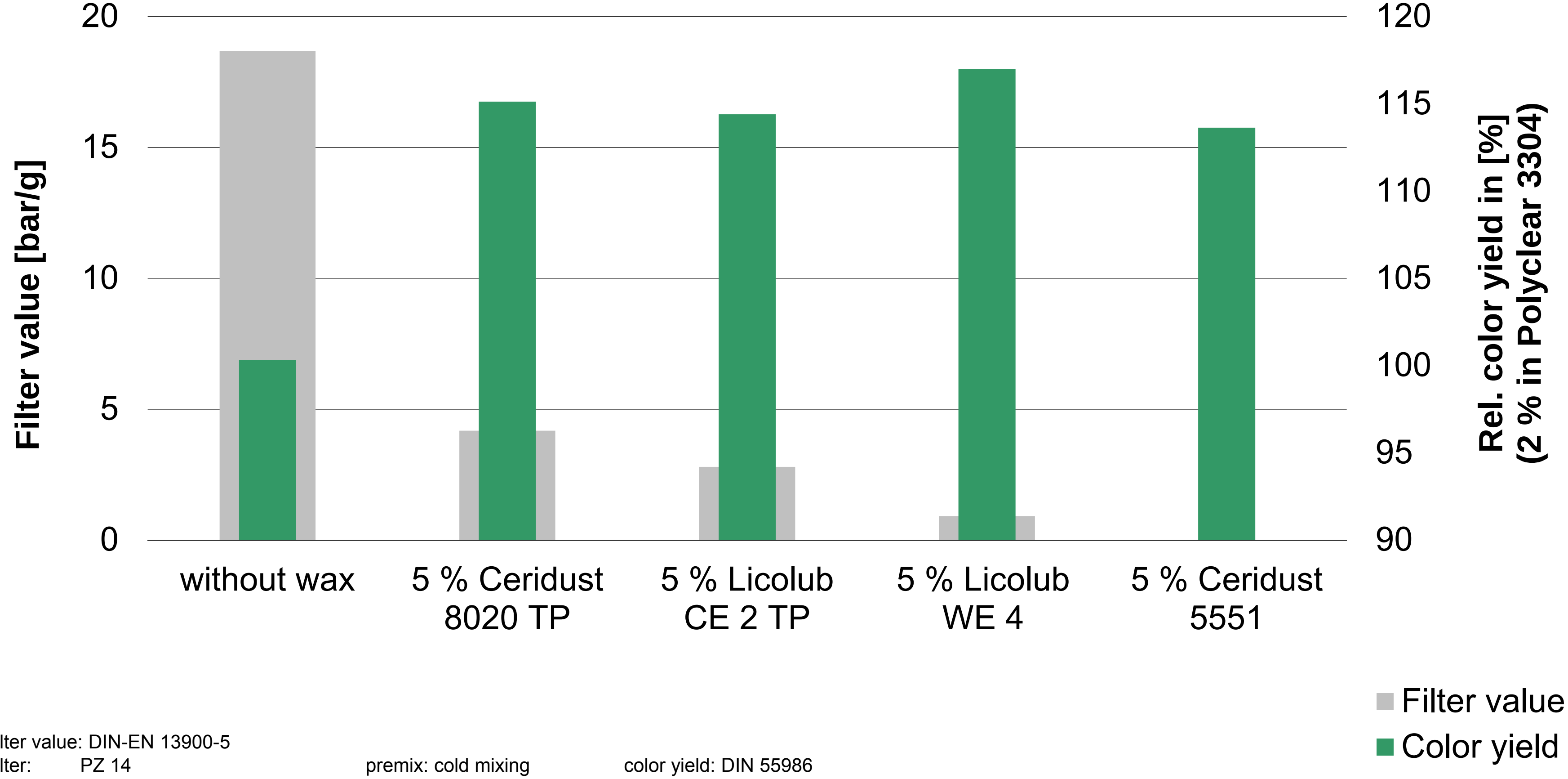
Licolub® CE 2 TP and Ceridust 8020 TP as a Potential Alternative to Montan Waxes

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



filter value: DIN-EN 13900-5
filter: PZ 14

premix: cold mixing

color yield: DIN 55986

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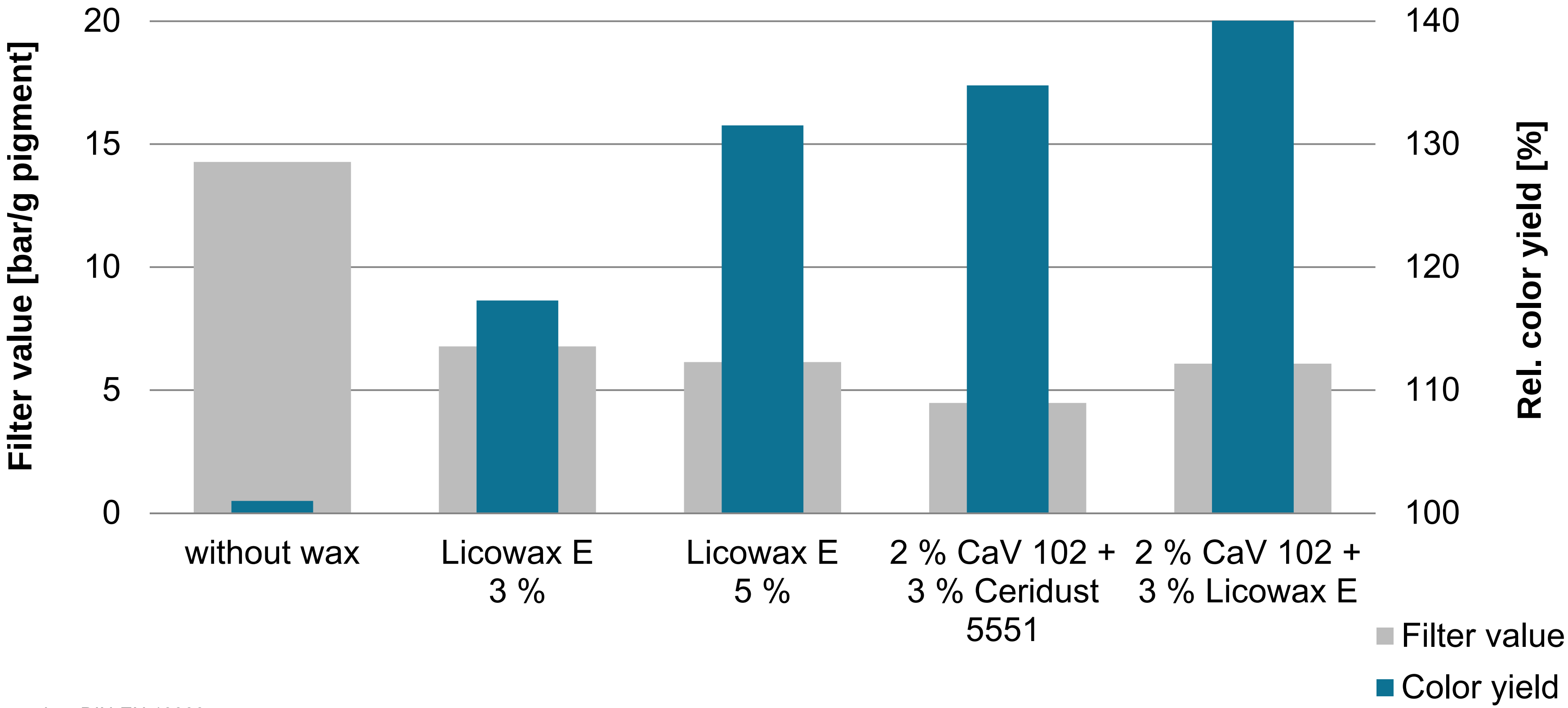
Improved Dispersion with Micronized Waxes in Engineering Resins – Polyamide 6

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



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

color yield: DIN 55986

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

VN 82358 - 82367

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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?

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