

Improved Output, Surface Effects & Physicals

Choosing the Right Wax

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Waxes for Plastics

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SPE Olefins 2017

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Clariant at a glance – An introduction

A GLOBALLY LEADING COMPANY IN SPECIALTY CHEMICALS



5807

Sales 2015 (CHF m)
from continuing operations

227

Net result 2015 (CHF m)
from continuing operations

4

Business Areas

853

EBITDA 2015 (CHF m)
before exceptionals

14.7%

EBITDA margin 2015
before exceptionals

110 in **53**

companies

countries

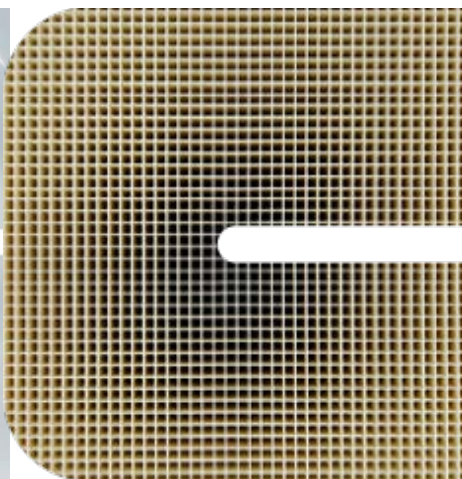
17213

Employees 2015

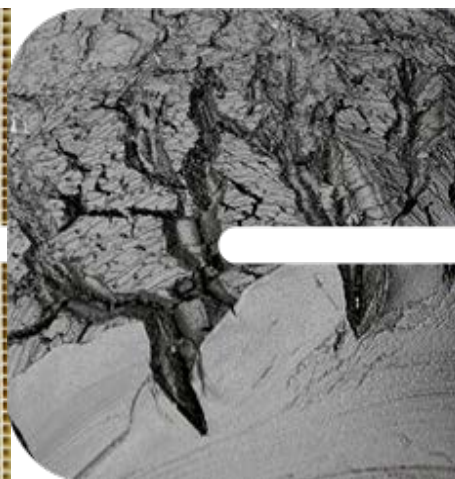
Four Business Areas -the right portfolio for growth



**Care
Chemicals**



Catalysis



**Natural
Resources**

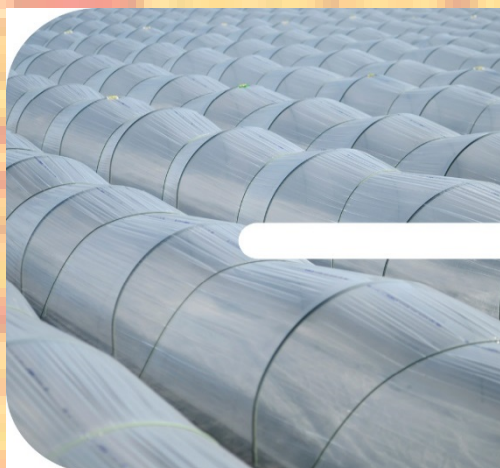


**Plastics
& Coatings**

BU Additives, of Clariant Plastics & Coatings – 3 Business Lines: FR's, Additives and Waxes



Flame Retardants:
Innovative products such as patented non-halogenated flame retardants provide environmentally compatible protection for electrical and electronic equipment.



Polymer Additives:
Polymer additives prevent oxidation, dissipate electric charge accumulation and improve heat, light and weather resistance



Waxes:
Waxes optimized for plastic applications, hot melt adhesives, polishes and protective coatings.

BL Waxes – Specialty Performance Waxes

Licolub™ & Licowax™ Montan waxes

Licowax™ & Licomont™ Saponified Montan Ester Waxes

Licocene™ Metallocene Olefin Waxes

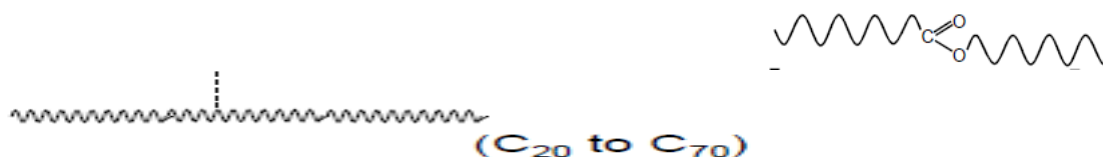
Ceridust™ Micronized Olefins (PE&PP), Oxidized Olefins, Montans and Amides

Licocene™ Maleic Anhydride-Grafted Metallocene Olefins

Licowax™ Amide Wax – EBS

Waxes Defined

- Chemical definition: A lipid made up of a chain of alkanes or esters made from fatty acids and alcohols. Source: *Oxford Dictionary of Chemistry*



- Engineering definition: A semi-solid or smooth solid featuring a low melting point and thermoplastic qualities; which may be of animal, plant or mineral origins. Source: *Dictionary of Engineering*.



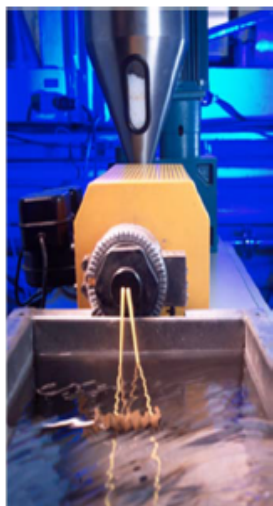
- Common English definition: A solid substance that becomes soft when warm and melts easily; often used to make candles. Source: *Cambridge Learner's Dictionary*



What Should Waxes Achieve in Polymers ?

During Mixing

- Homogenizing
- Compatibilizing
- Dispersion

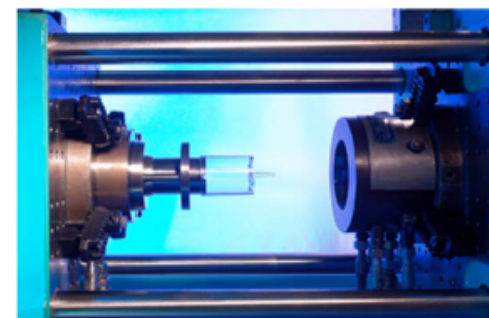


During Processing

- Flow Improvement
- Decrease pressure
- Release effect
- Smoothen surface
- Pigment dispersion
- Avoid over-shearing of the polymer
- Increase color yield
- Reduce filter pressure

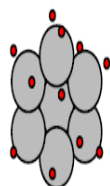
Article properties

- Provide antiblock effect
- Maintain printability
- Provide gloss
- Provide transparency
- Smooth surface



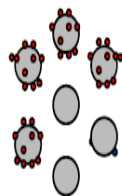
What Should Waxes Achieve with Pigments ?

Pigment Wetting



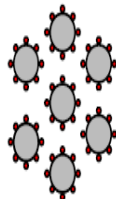
Wax penetrates into the pigment agglomerates

Dispersion



Wax breaks up of the pigment agglomerates

Stabilization



Wax prevents pigment particles from reagglomeration

Criteria for Choosing Wax(es):

- Viscosity
- Polarity
- Volatility
- Color Stability
- Particle Size
- Interactions

Particle Size Influences Dispersion

- Influence of Particle Size

micronized

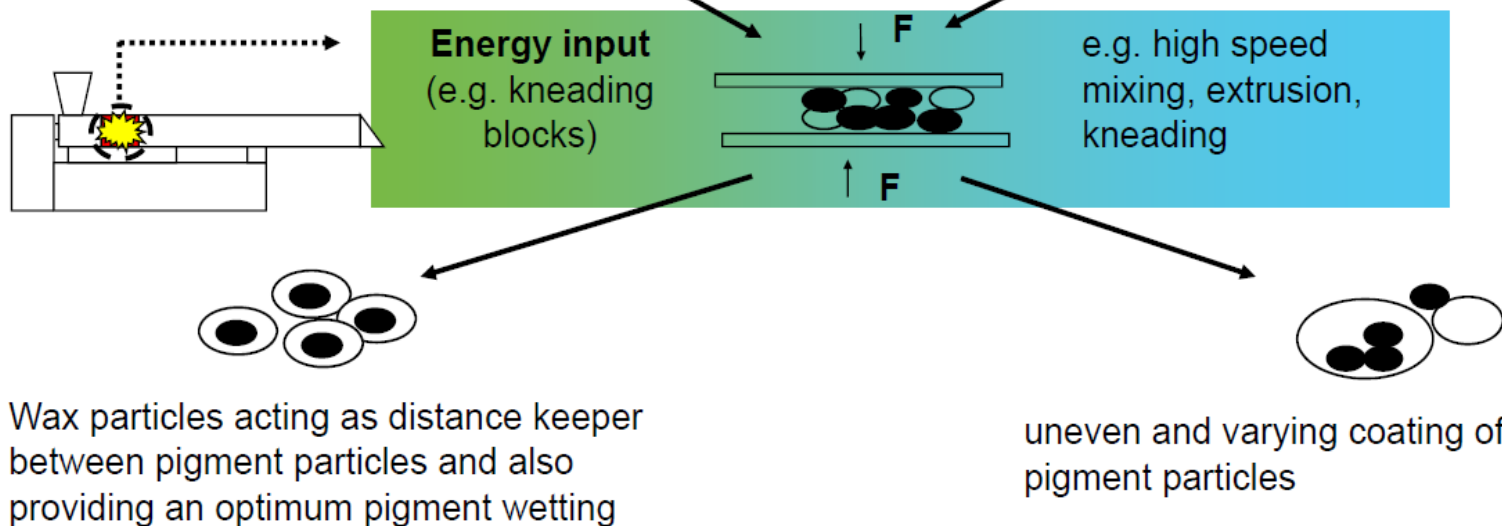


Pigment- and wax particles show similar sizes

conventional



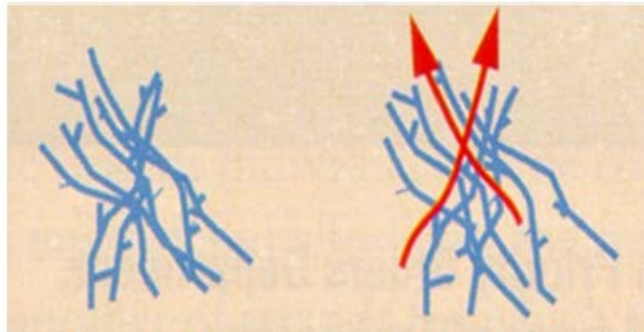
Wax particles are significantly bigger than pigment particles



Lubricant Mechanism's

How they work in polymers.

Internal lubrication between polymer chains



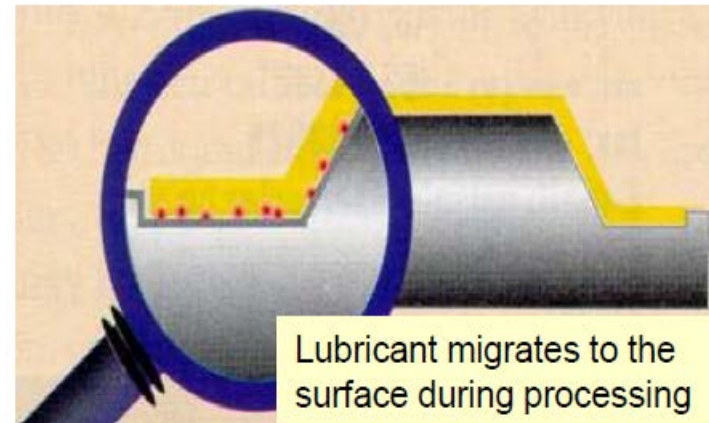
Without lubricant

With lubricant



Reduced viscosity
Improved flow properties

External lubrication on the surface



Improved release effect

Internal and External Lubrication – Further Explained

Internal Lubrication:

- promotes flow
- promotes weld line fusion
- improved die fill
- reduces die swell
- reduces die drool
- reduces head pressure
- can help keep clarity
- reduces HDT

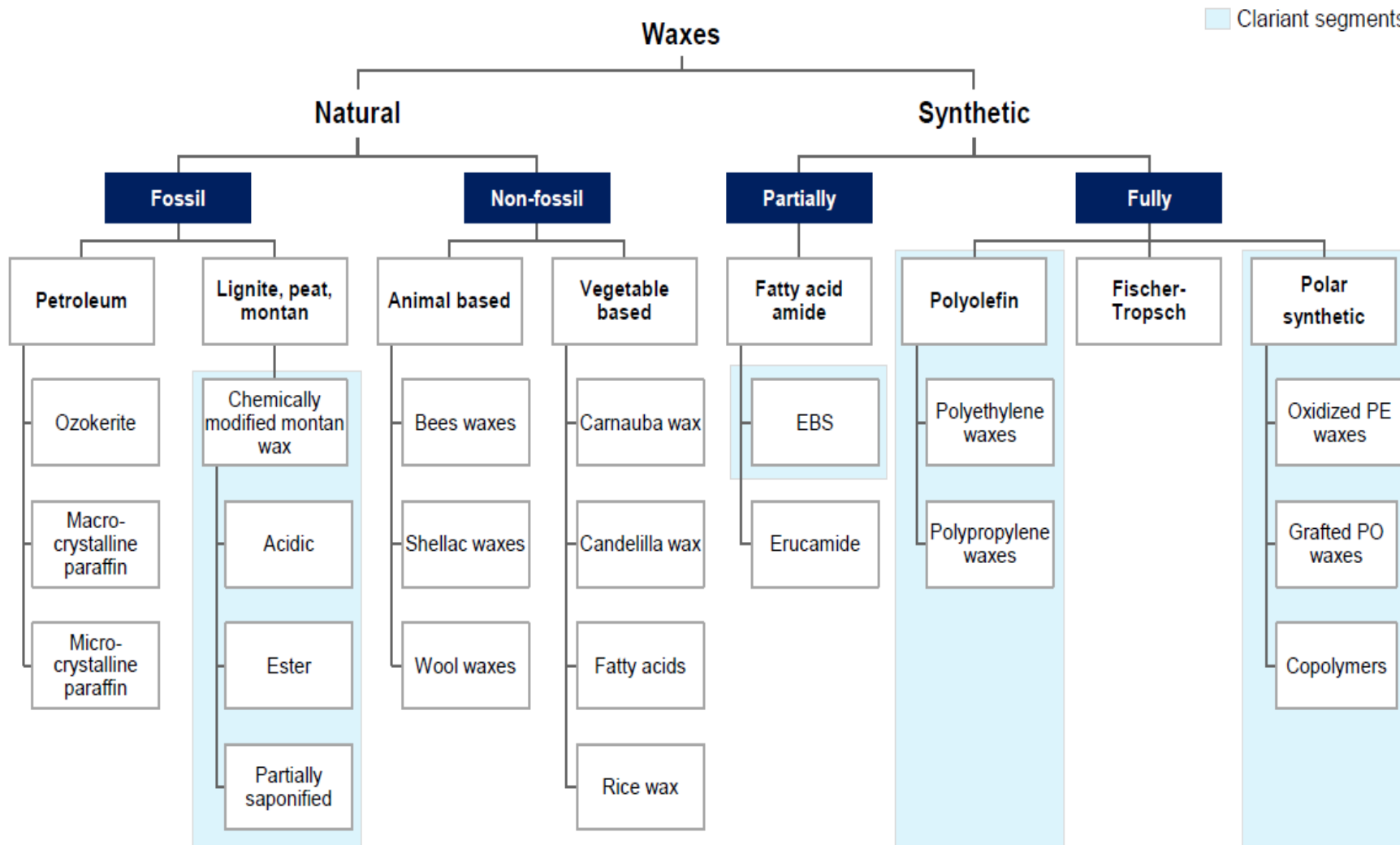
External Lubrication:

- metal release; i.e. “slip”
- reduces process temp
- can plate out [exusion]
- can slow recrystallization (Hc)
- can reduce weld line strength
- can “pool” in extruders; cause surge
- can interfere with printing
- can discolor; especially over long runs

In reality, and depending on the concentration, many waxes or lubricants act as both.

Differing Chemistries– Depending on Usage

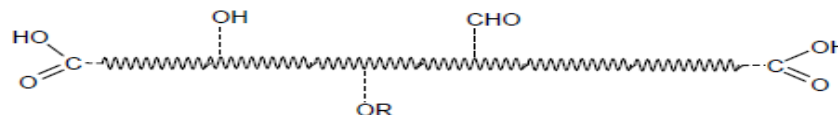
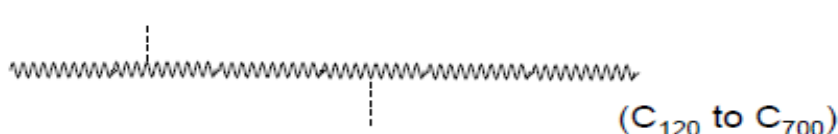
So, for Olefins, what is used?



Waxes Commonly Used in Olefins:

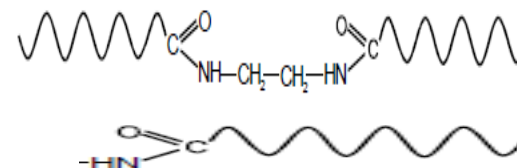
Olefin waxes; both polar and non-polar (i.e. PE, PP, Copolymer olefins)

- non-polar (i.e. PE, PP) more miscible in olefins, so mostly stay as an internal lube
- polar (oxidized, grafted or acid-copolymer) not as miscible in olefins, so it exudes to be more like an external lube. Best for functional polymers.



Amides (i.e. oleamide, erucamide, EBS)

- The materials have an aliphatic tail that prefers the polymer. However,.....
- Their amide (polar) end exudes to lubricate between the olefin and metal, providing the “slip” effect, from which they get their nickname.
- Amides can also act as anti-stats; especially with GMS



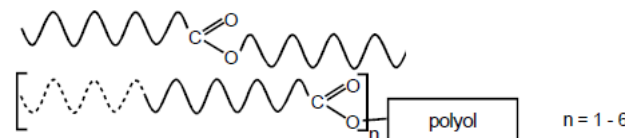
Waxes Commonly Used in Olefins (cont.):

Soaps (i.e. saponified metal carboxylates)

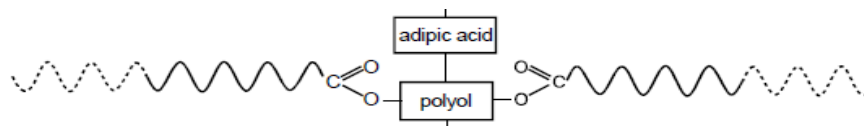


- lower m.w. types like zinc stearate, calcium stearate (more volatile, char sooner)
- higher m.w. types like sodium and calcium montanates (less volatile, resist char)

Esters (i.e. GMS, FACE, MACE)



- mono-esters of polyols (i.e. GMS) work as anti-stat too; especially with amides
- fatty acid polyesters of polyols reduce volatility and discoloration
- complex esters further reduce volatility; especially when higher m.w., montan waxes



Masterbatchers' Needs from Wax

- **Best Dispersion possible**
- **Highest concentration possible (for their clients' economies)**
- **Lowest influence on Color**
- **Color Consistency over a long run**
- **Minimal die drool or release of oxidized material (black specks)**
- **Lowest dosages possible**
- **Low organo-leptic contributions (smell, taste)**

Molders' Needs from Waxes

- **Flow Improvement; i.e. Longer flow for hard-to-fill, thin-walled parts**
 - **reduced surface drag (i.e. external, surface lubrication)**
 - **less molded-in stress (internal lubrication)**
 - **melt strength retention [read mol. wt. retention] when lubricating**
- **Faster hardening; i.e. quicker crystallization/nucleation, improved physicals**
- **Easier mold release = Reduced sticking; without affecting labelling/adhesives**
- **Resin-rich [smooth] surface and complete “wetting”; with filled polymers**
- **Improved color development and consistency or transparency when needed**

Extruders' Needs from Waxes

Faster output, while maintaining the following:

- Surface smoothness**
- Surface release (both metal “slip” and polymer “anti-block”)**
- Above release without excess wax inhibiting printing**
- Low volatiles to reduce build-up of additives on rollers**
- Reduced “die drool” at die lip (pressure drop & shear; low m.w. mat'l)**
- Improved compatibility / binding of dissimilar material; i.e. WPC**

How do **you** know if all needs are met?

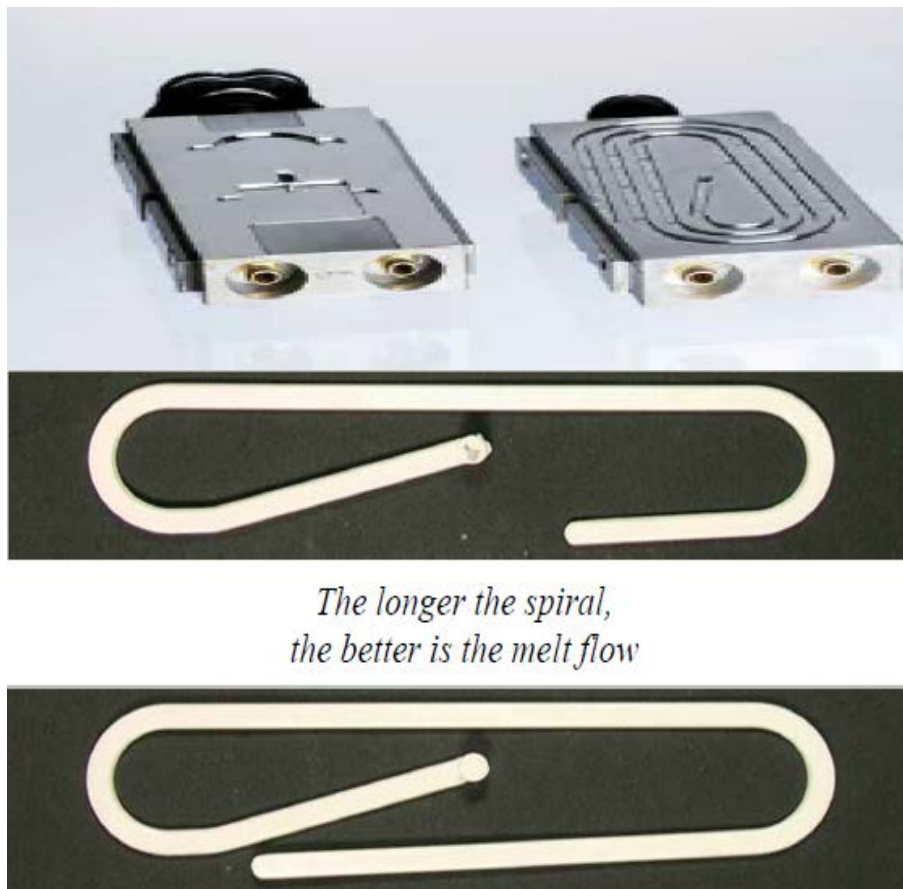
- Processing as rapidly as the machinery allows
- Aggressive processing (i.e. high heat) does not discolor the polymer or cause char (specks and streaking)
- Wax does not hinder print adhesion
- Minimal die drool (extrusion)
- Minimal vent plugging and gate blush (injection)
- Color consistency and development



How do **we** know if we are designing waxes properly?
Testing to Theory.

- **Mold flow rate Improvement**
- **Mold release force reduction**
- **Reduced surface roughness**
- **Reduced extruder pressures**
- **Reduced volatility, vs. standard waxes**
- **Reduced charring in heated ovens, vs. standard waxes**
- **Improved pigment dispersion; less specking**

Mold Flow Improvement Measured - *While Keeping Polymer Integrity*



Spiral Melt Flow Testing



Haake Kneader – Viscosity Testing

Easier Mold Release - Quantified



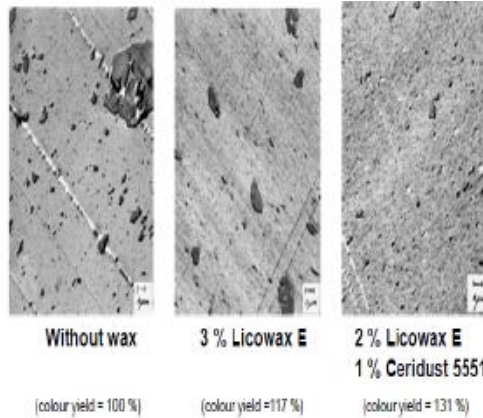
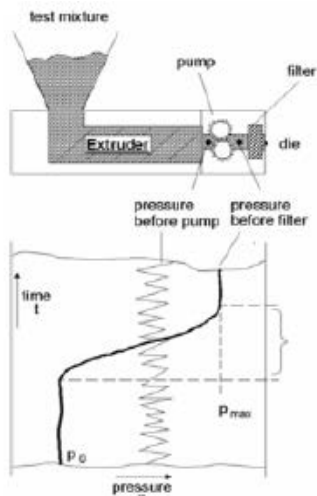
Molding and Integrated
Release Quantifying
Force (N) Unit



Molded Cylinders >



Pressure Rise & Color Development Testing



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

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]



production of the color
yield plates



measurement of the
color yield, (X,Y,Z values,
or L*a*b* values DIN 53 235)

$$F = m_B / m_P * 100$$

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

Testing Volatility by TGA & Air Draft Oven



What TGA Can Tell You

- Thermal Stability of Materials
- Oxidative Stability of Materials
- Composition of Multi-component Systems
- Estimated Lifetime of a Product
- Decomposition Kinetics of Materials
- The Effect of Reactive or Corrosive Atmospheres on Materials
- Moisture and Volatiles Content of Materials



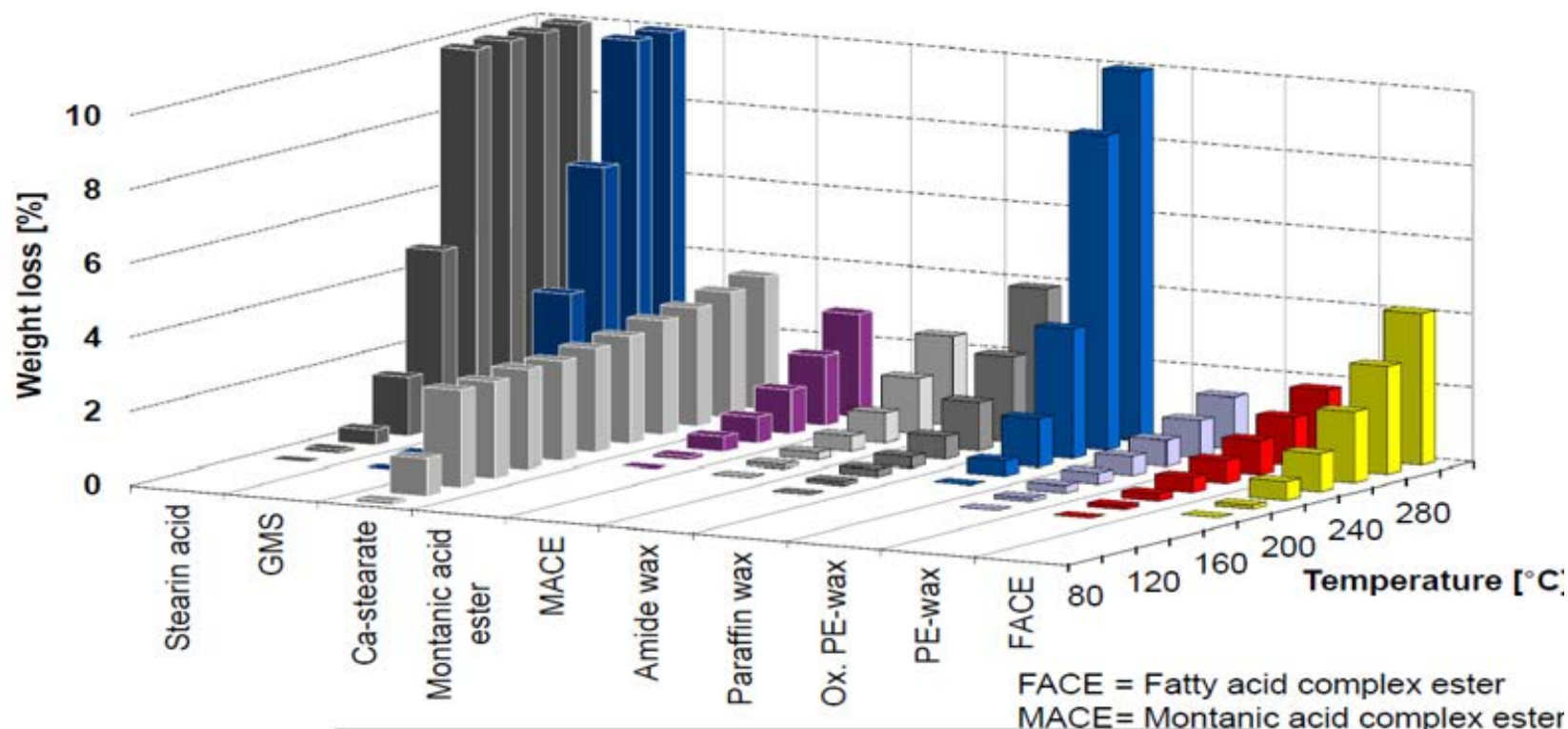
TGA for small lab samples.

Air Draft for larger samples to show color development, Charring, etc.

What do These Tests Show?

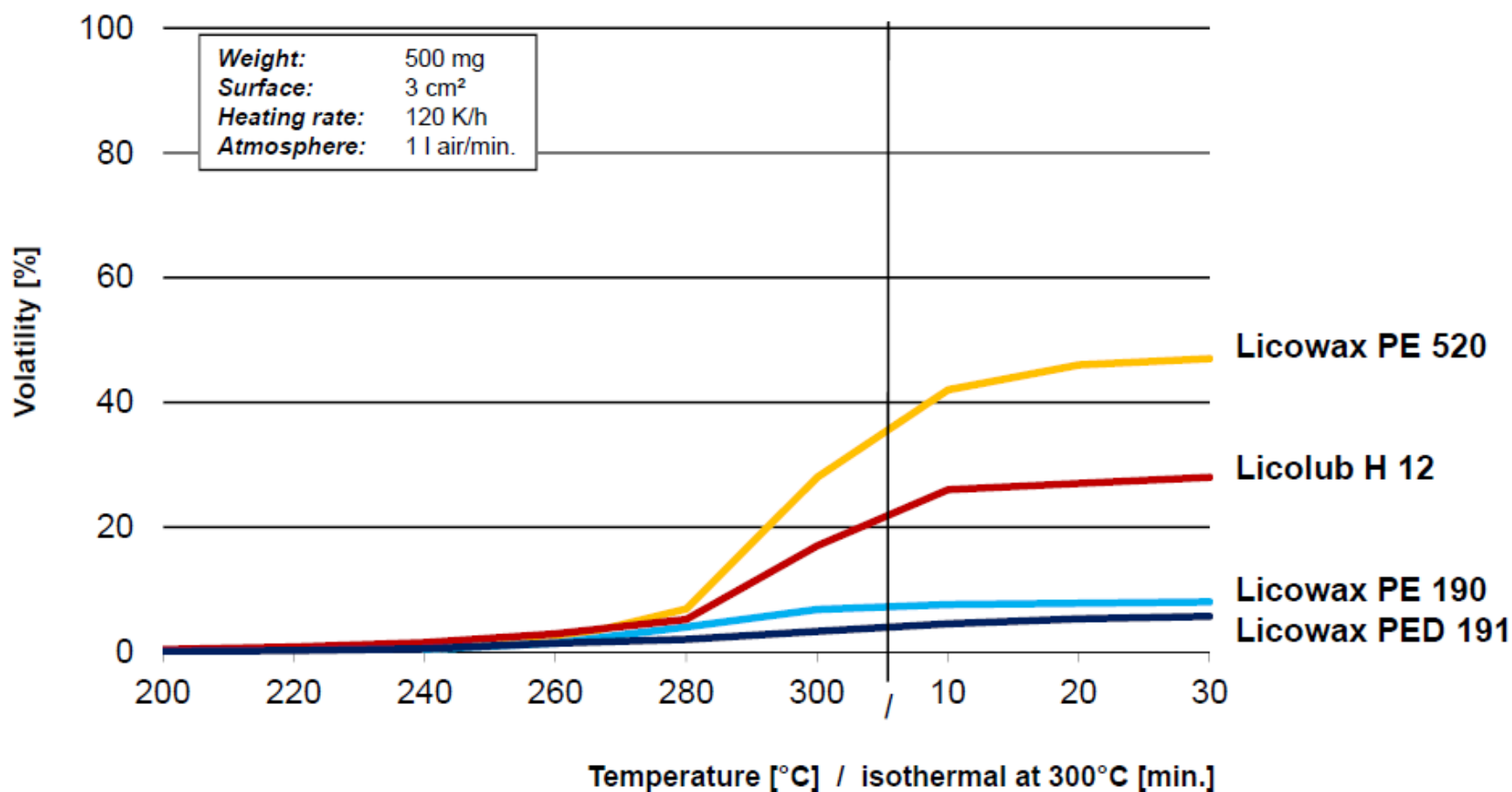
Volatility Results of Various Waxes

Volatility of Lubricants

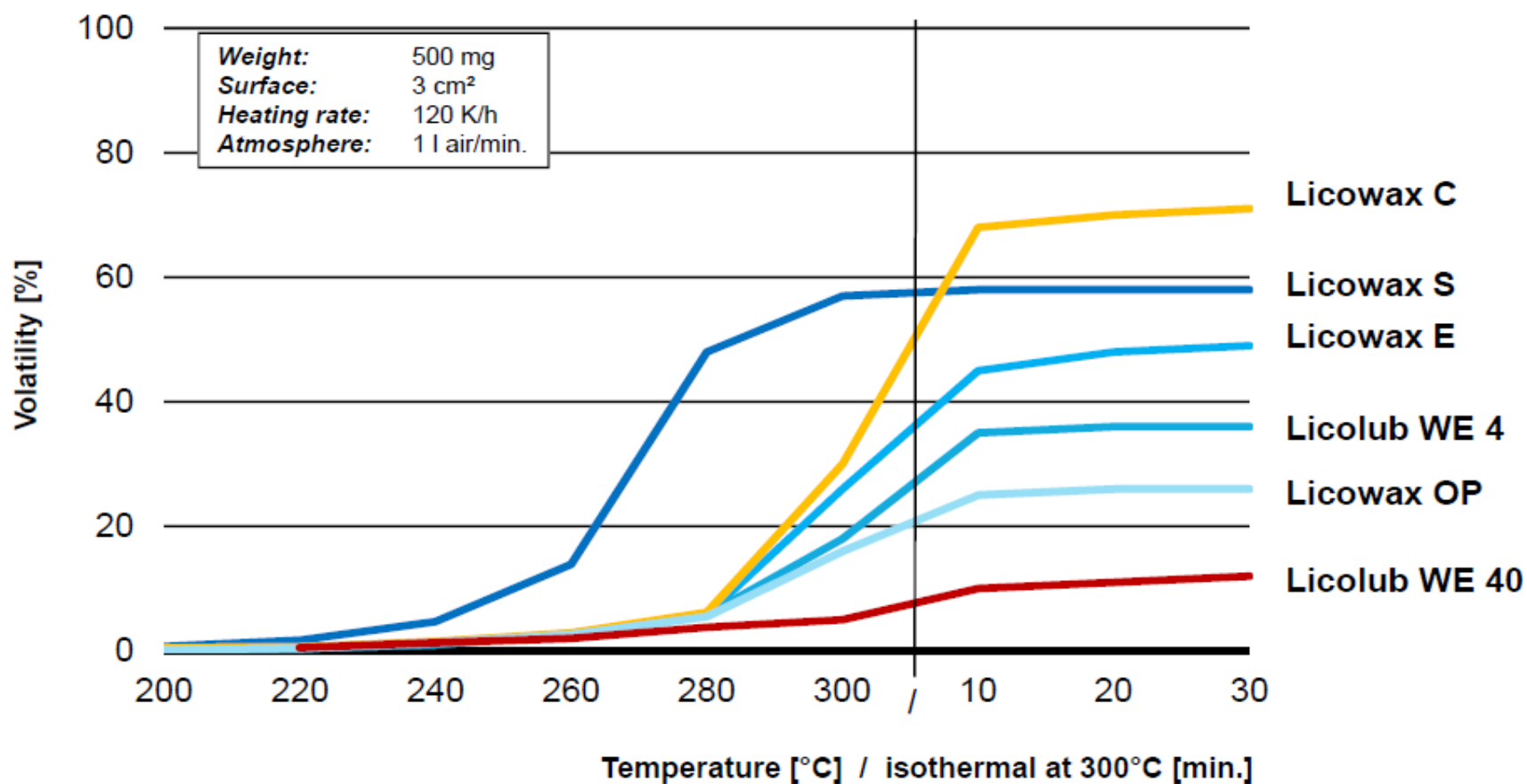


TGA results from Clariant – Gersthofen Lab

Volatility (TGA) of Polyolefin Waxes



Volatility (TGA) of Ester and Amide Waxes



Reduced Charring of Montan Waxes vs. Stearates

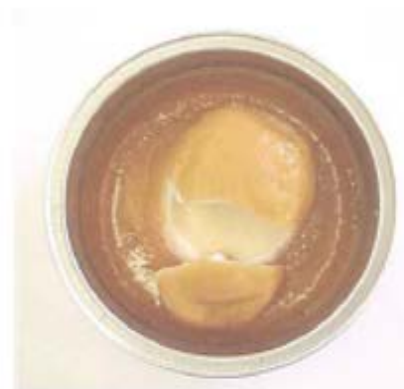
Air Draft Oven at 250C, for 30 min.



Licolub WE 40



Licolub WE 4



Calciumstearate SP



Calciumstearate
Liga 860



Licowax E



Licowax OP



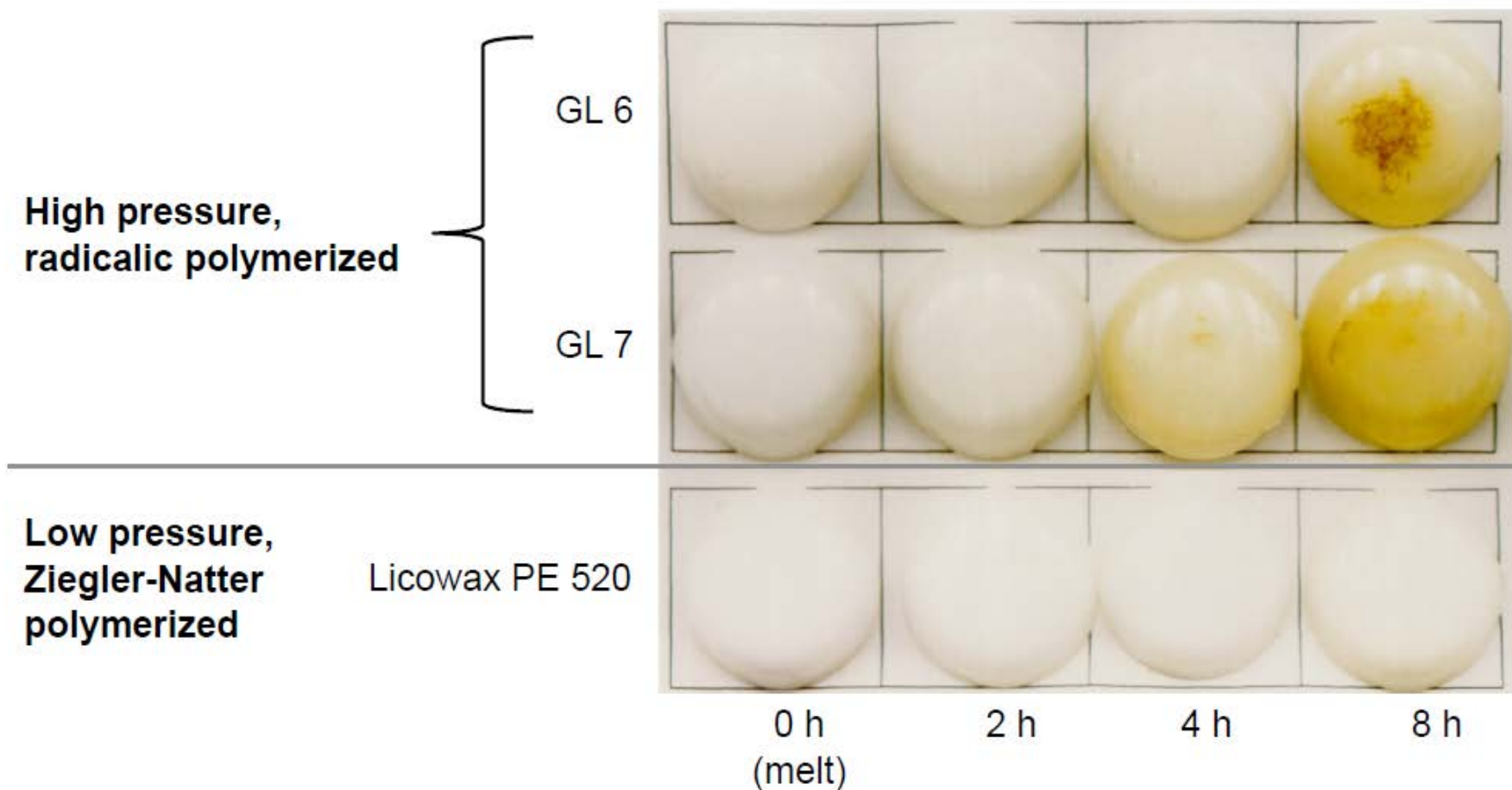
Magnesiumstearate SP



Zinkstearate SM 101

Reduced charring of Licolub PE Waxes

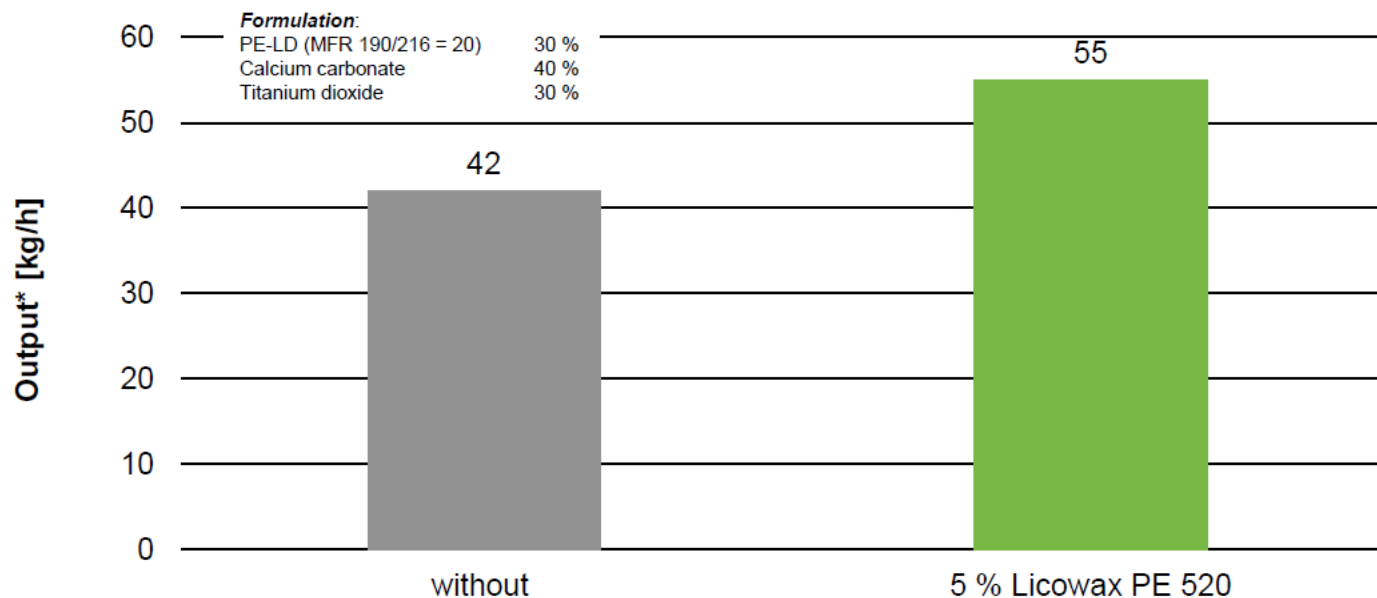
Tempering of PE-Waxes (Oven test 200 °C, Air)



Melt Flow Data with PE Wax

Licowax PE 520 in Polyethylene Flow Improvement

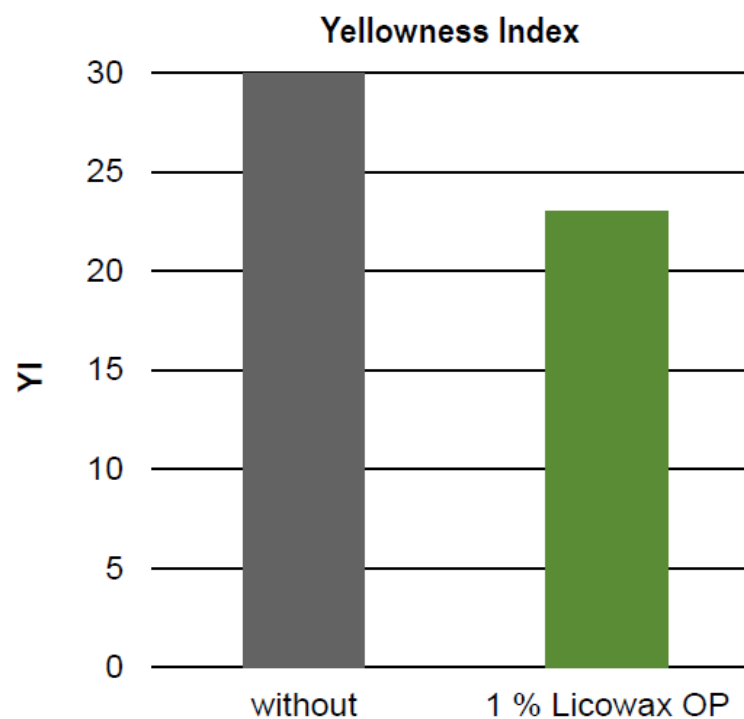
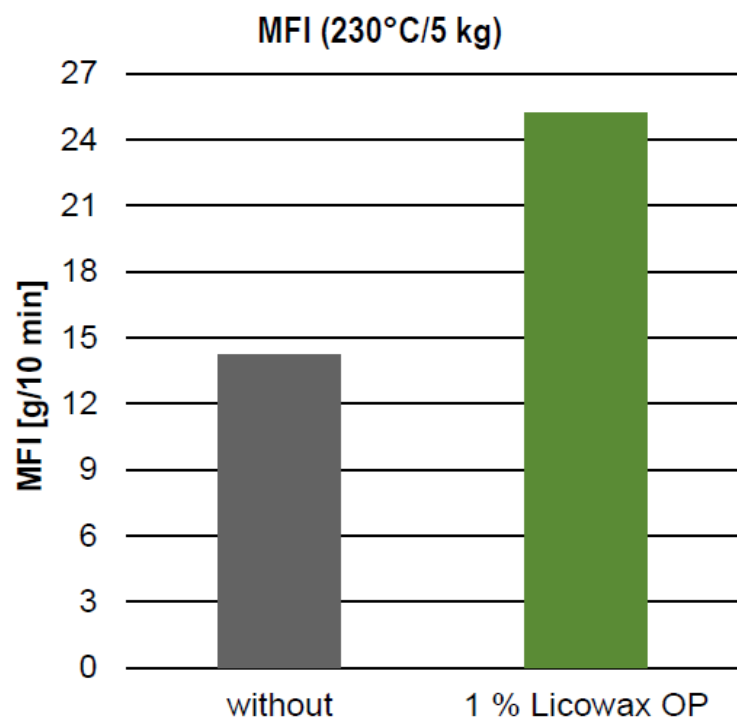
Influence on output rate



* determined at 90 % power uptake of extruder

Flow Data with Partially Saponified Montan Wax

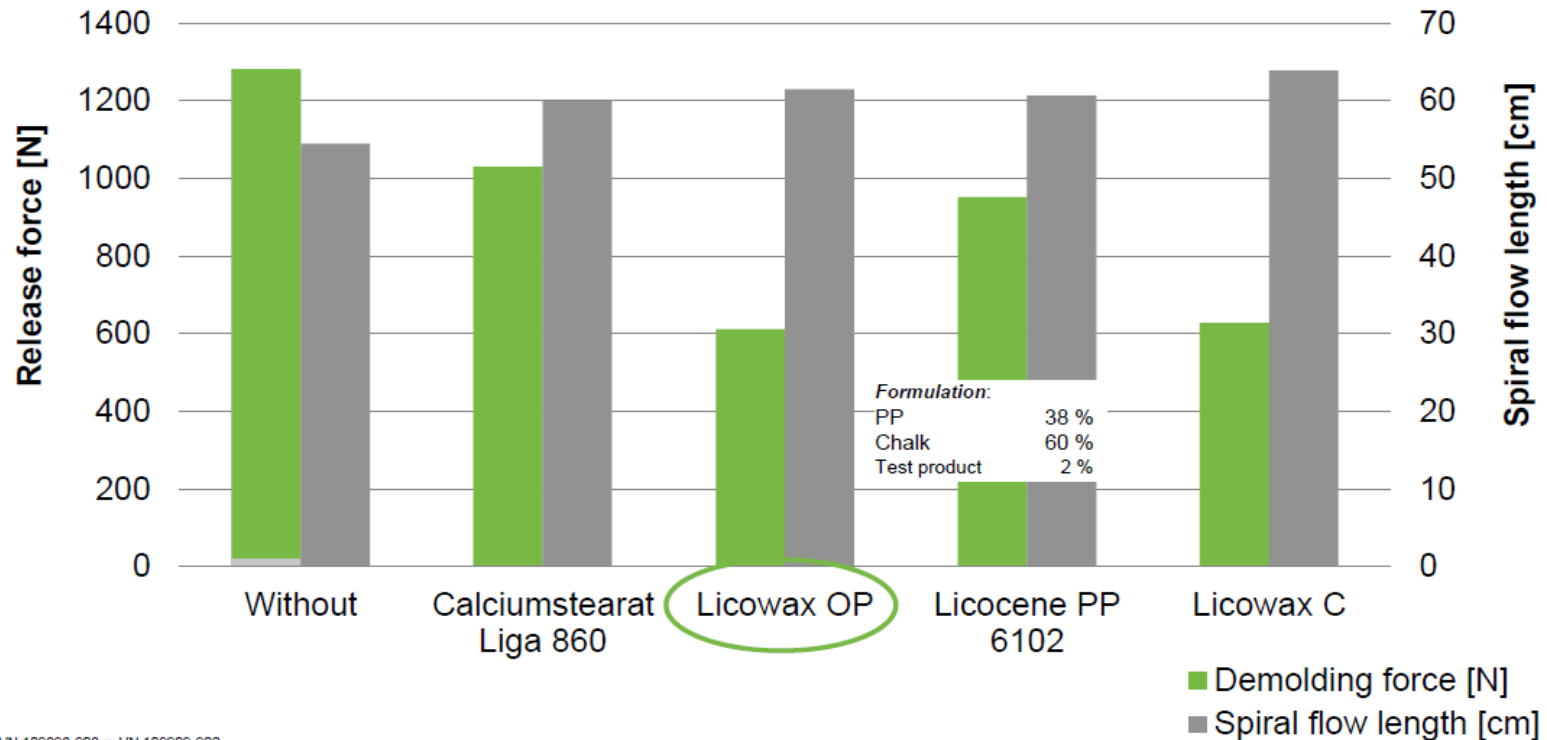
Licowax OP for
Talc-Filled Polypropylene Compounds (30 wt.-%)
- Flow Improvement and Color Behavior



Flow & Mold Release Data – Various waxes

Licowax OP for Chalk-Filled Polypropylene Compounds - Release Force and Spiral Flow Test

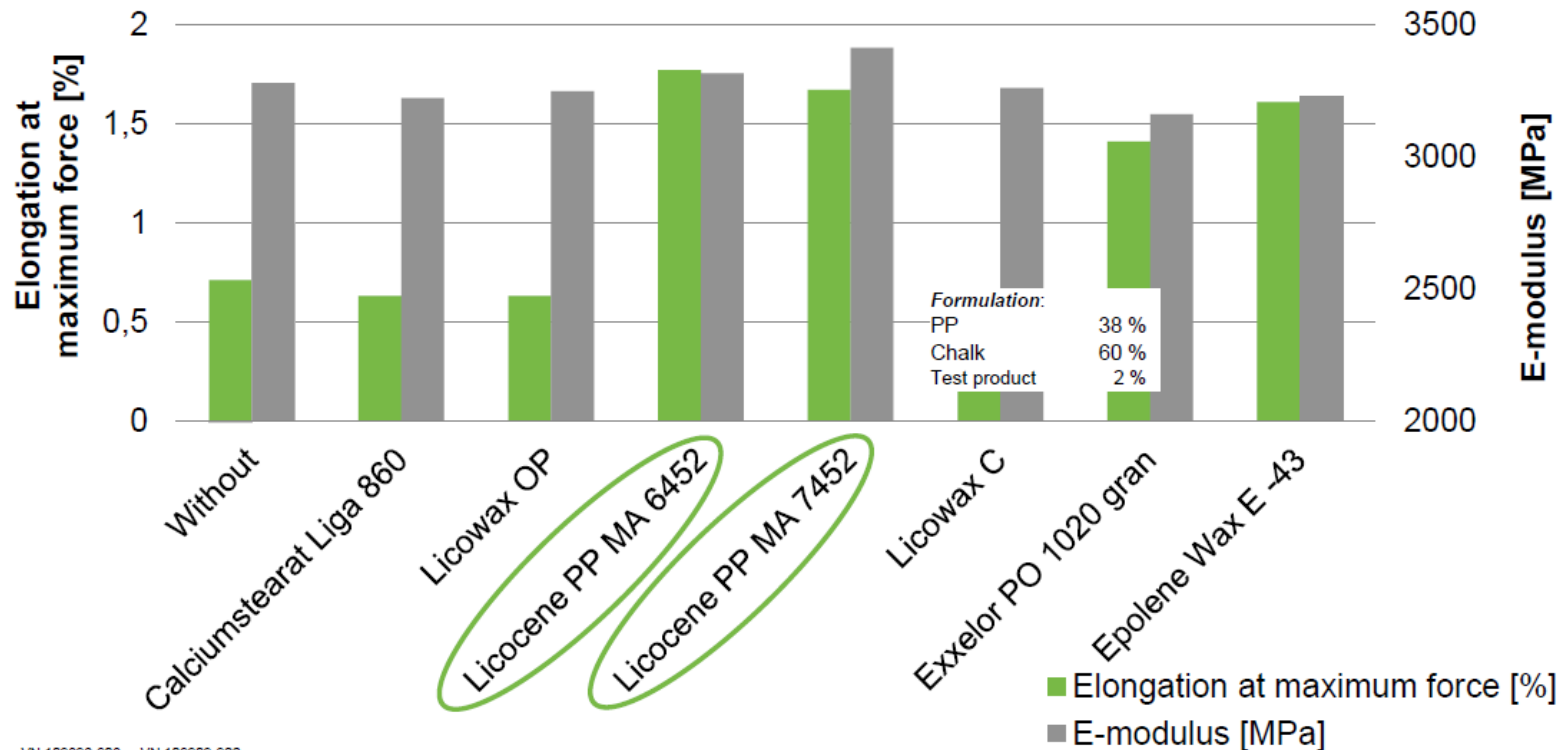
Lowest release force, good flow improvement with Licowax OP



Filled PP Physical Testing Data

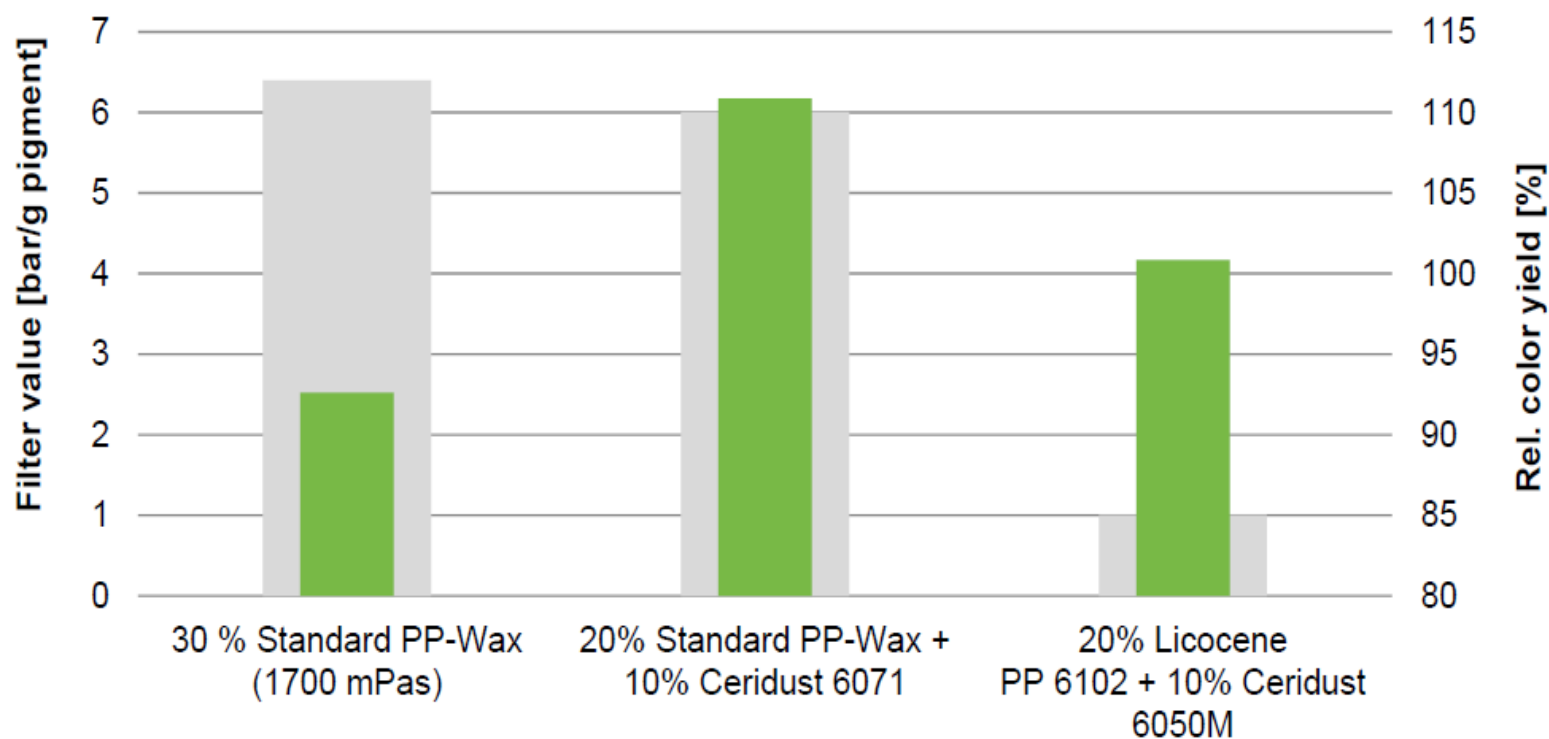
Grafted Licocene PP Grades for Chalk-Filled Polypropylene Compounds - E-Modulus and Elongation

Highest E-modulus and Elongation with grafted Licocene PP grades



PP Filter Test and Color Yield Data

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



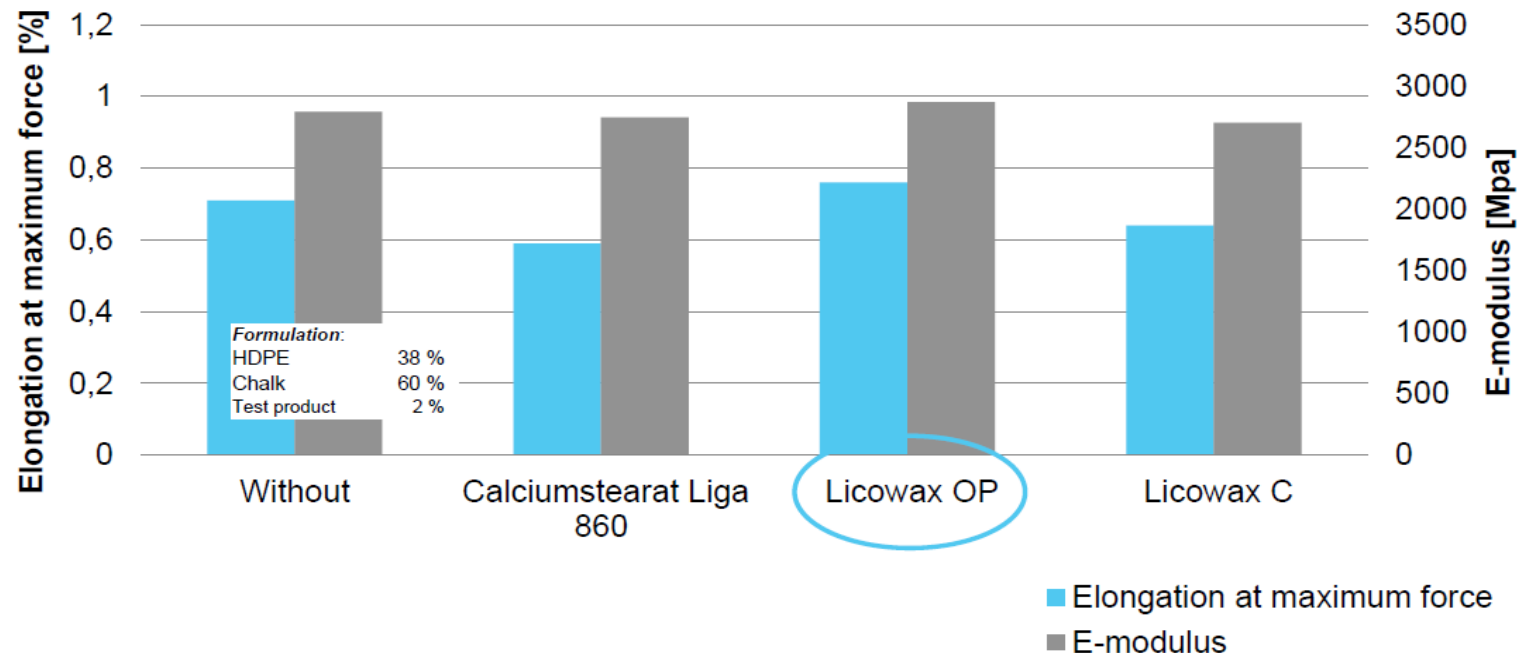
Formulation: 40% Pigm.Red 48:2 (e.g.Lithol Scharlach K 4461); 20 - 30% wax; 30 - 40% PP, MFR 30.
Extruder: ZE25x40D co-rotating; temp. profile: 180-200-190-170-150-170-190-180-175°C;
screw speed: 500 rpm; throughput: 5.0 kg/h; main feed

■ Filter value
■ Color yield

Filled HDPE Physical Testing

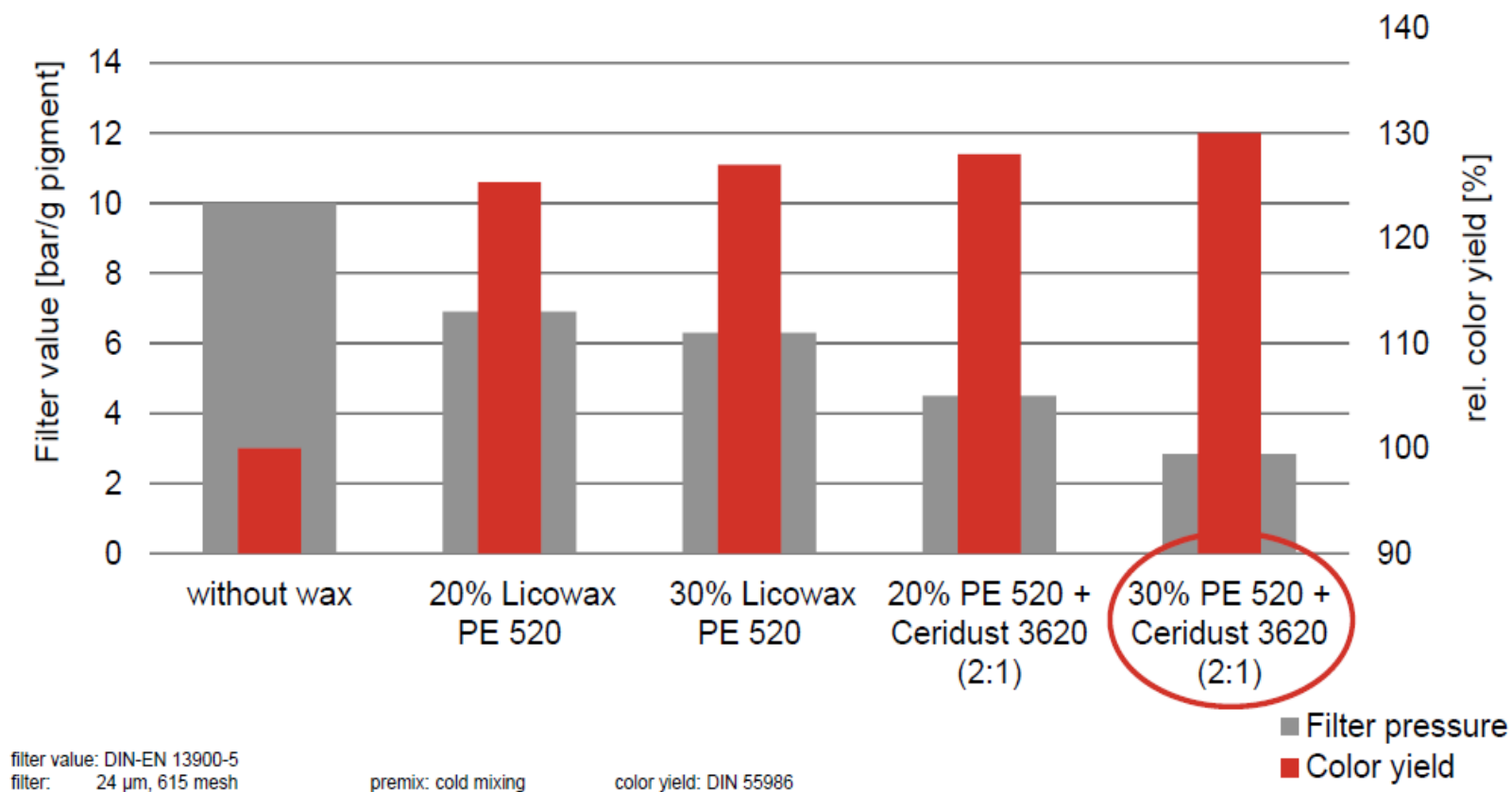
Licowax OP for Chalk-Filled HDPE Compounds - Elongation and E-Modulus

No negative influence of E-modulus and elongation



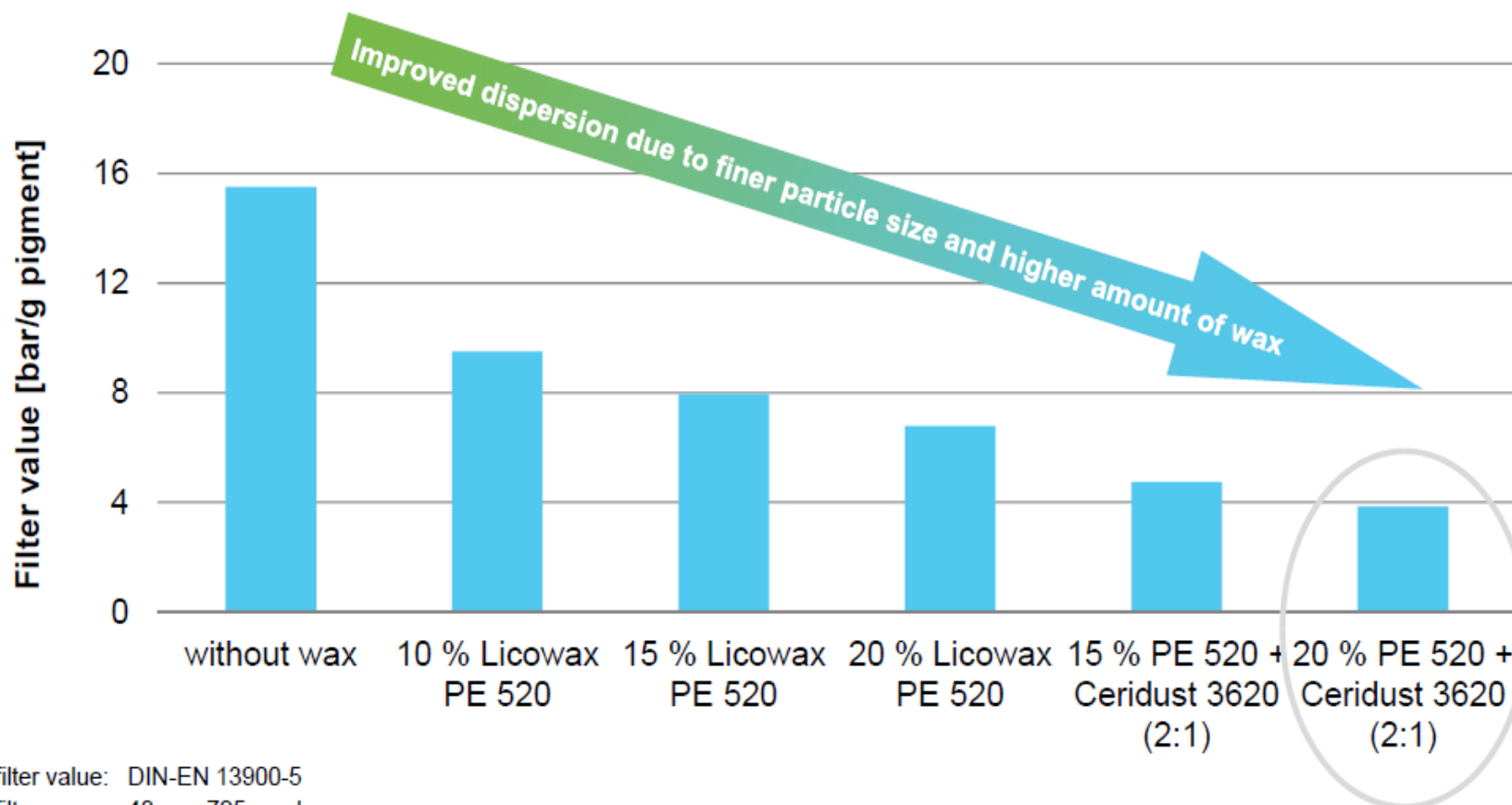
LLDPE MB Filter Test and Color Yield

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



Filter Test – Fast Blue A2R in LLDPE

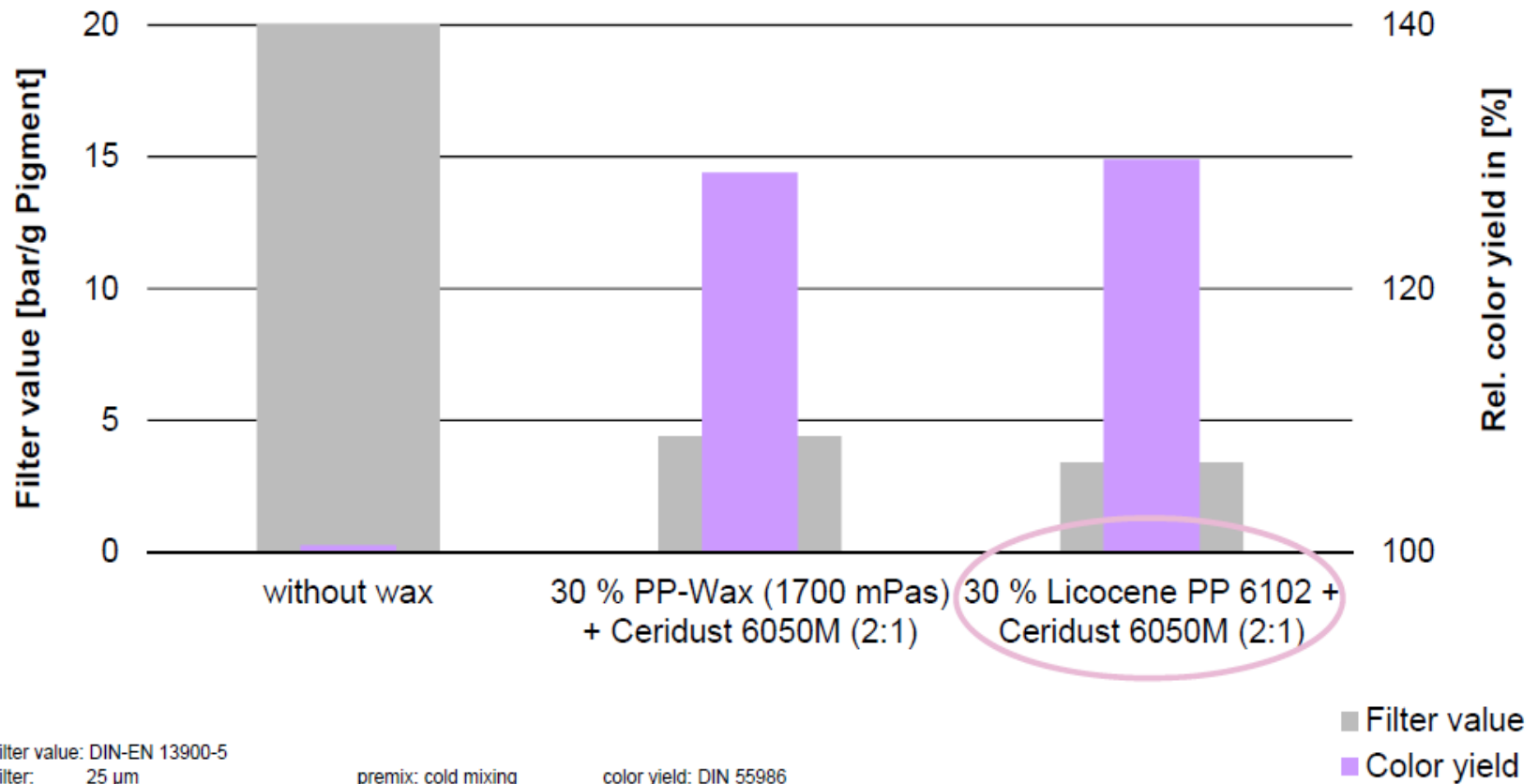
– 30 % Fast blue A2R in LLDPE



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

Effects of Wax on Dispersion and Color yield

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



Summary of Effects of Properly Chosen Waxes

- **Better mold release**
- **Maintains polymer integrity [read viscosity]**
- **Improved melt flow; easier mold fill**
- **Better color consistency; less charring**
- **Reduced volatiles; less mold / vent cleaning**
- **Quicker recrystallization (injection molded crystalline polymers); i.e short cycles**
- **Improved spherulite formation; i.e. better impact physicals**
- **Resin-rich surface with filled compounds; better appearance**
- **Maintains better clarity, where applicable**
- **Improve pigment dispersion and color consistency**

General Wax Recommendations for Olefins

PP fiber masterbatch	Fiber	Dispersion	Licocene PP 6102
PP-filled	Extrusion Injection molding	Dispersion, output, Surface quality, Hydrophobation	Licowax OP Licowax C Licolub FA 1 pwd. veg. based Licocene PP MA 6452 Licocene PP MA 7452
PP-filled, rubber modified	Injection molding	Release effect, Mechanical properties, Paintability	Licolub WE 40
PP-recyclate Filled recylate		Flow properties	Licocene PP 6102 Licowax OP Licocene PP MA 6452 Licocene PP MA 7452
HDPE	Tapes	Slip agent	Licowax C Licolub FA 1 pwd. veg. based
PE-recyclate Filled recylate		Flow properties	Licowax PE 520 Licowax OP Licowax C Licolub FA 1 pwd. veg. based
PE-filled	Extrusion Injection molding	Dispersion, output, Surface quality, Release effect	Licowax OP Licocene PE MA 4351

Troubleshooting – Correcting Common Wax Issues:

Problem	Possible Causes	Remediation
Blacks specks in PE film, or PP extrudate	- excessive die or barrel heat, wax not thermally stable, or other additives not thermally stable and the lube is allowing co-exusion or release from the metal	Use a lube of higher thermal stability; i.e. Licowax OP, PE 520, Licolub WE 40. See Heated Oven results (slides 28,29) for examples.
Pigments chunks or flecks in PE film or film masterbatch	- poor dispersion	Improve masterbatch or compound dispersion with Licowax PE 520 or Licocene PE 4201.
Agglomerates in PP fiber masterbatch	- poor dispersion	Improve dispersion with Licocene PP 6102.
High amp or Torque in highly loaded MB	- melt flow too stiff, colorants not wetted out	Increase melt flow with Licocene PE 520
High Amp or Torque in rubber modified PP	- melt too stiff, metal drag or friction	Increase flow & metal release with Licolub WE 40
Poor adhesion/wetting/compatibility of wood filled olefins - both PP and HDPE Black streaks in extrudate or molded part	- excessive moisture, or insufficient functionality of olefins to bond to hydroxyl-rich surface of cellulose - additive or wax depositing in the die block or manifolds/runner and charring	Improve wetting out and coupling to achieve better tensile and flex-mod with Licocenes PP MA6453, PP MA 7452 Substitute wax with greater thermal stability; i.e. Licowax PE 520, OP or WE 40. If the problem still persists, check other additives, or colorant dispersion.
Mold vents clogging, excessive mold residue, gate "blush"	- insufficient venting, excessive volatilization of wax or additives	Substitute with lower volatility waxes such as montan Licolub WE 40, OP waxes or PE Licowax PE 520 or 190, per chart on slides 25 - 27.
HDPE or PP parts sticking in mold	- insufficient metal	Reduce pull force / mold adhesion with a "slip" like Licowax C. If molding UHMWPE, use Licolube WE 40 for better thermal stability at high temp

QUESTIONS ?

Other Clariant Chemistries for Your Polymers:

Antioxidants (primary): e.g. Hostanox® O, and O 310

Antioxidants (secondary): e.g. Hostanox® P-EPQ

Antistatic Agents: e.g. Hostastat® HS1

Flame Retardants: e.g. Exolit® OP and APP (non-halo)

Light Stabilizers (HALS): e.g. Hostavin® N30

Light Stabilizers (UV Abs'): e.g. Hostavin® VSU

Cited Sources - Bibliography

- *Oxford Dictionary of Chemistry*, 6th Ed. , 2008. Oxford University Press
- *Dictionary of Engineering*, 2nd Ed., 2010. McGraw Hill Publishers
- *Cambridge Learner's Dictionary* with CD ROM, 3rd Ed. 2009 Cambridge University Press
- *New Montan Waxes-General Presentation*, Clariant P&C, 2016. Author: C. Lechner
- *Waxes for Plastics – General introduction*, Clariant P&C, 2010. Author: C. Lechner
- *Waxes- Portfolio, Chemistry & Effects 2*, Clariant P&C, 2016. Author: N. Karche
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