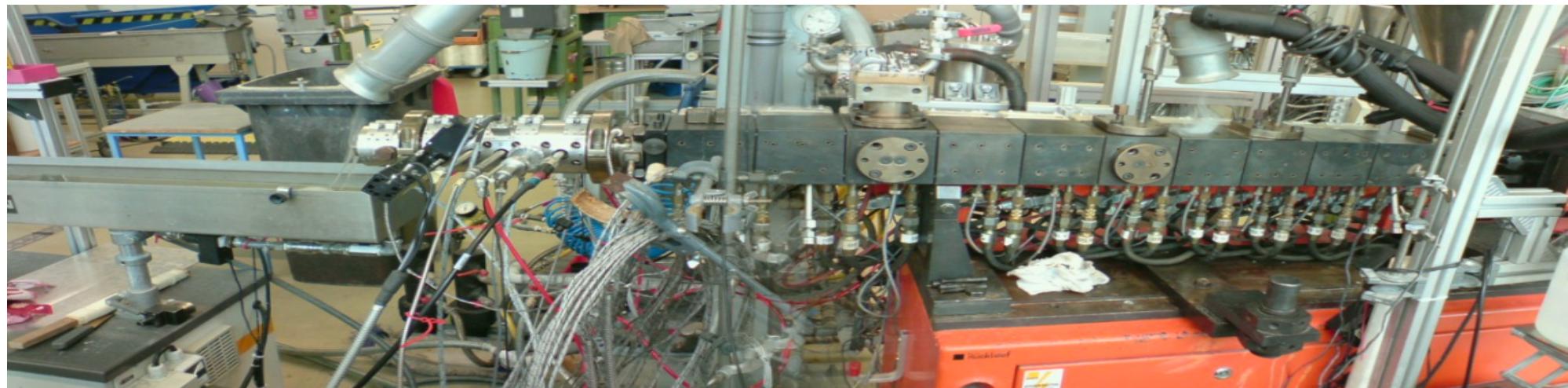


# In- und on-line Prozessüberwachung bei der Polymerverarbeitung und -synthese mit ATR-IR, NIR und RAMAN Spektroskopie

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<sup>2</sup> *LyondellBasell R&D, Ferrara, Italy*



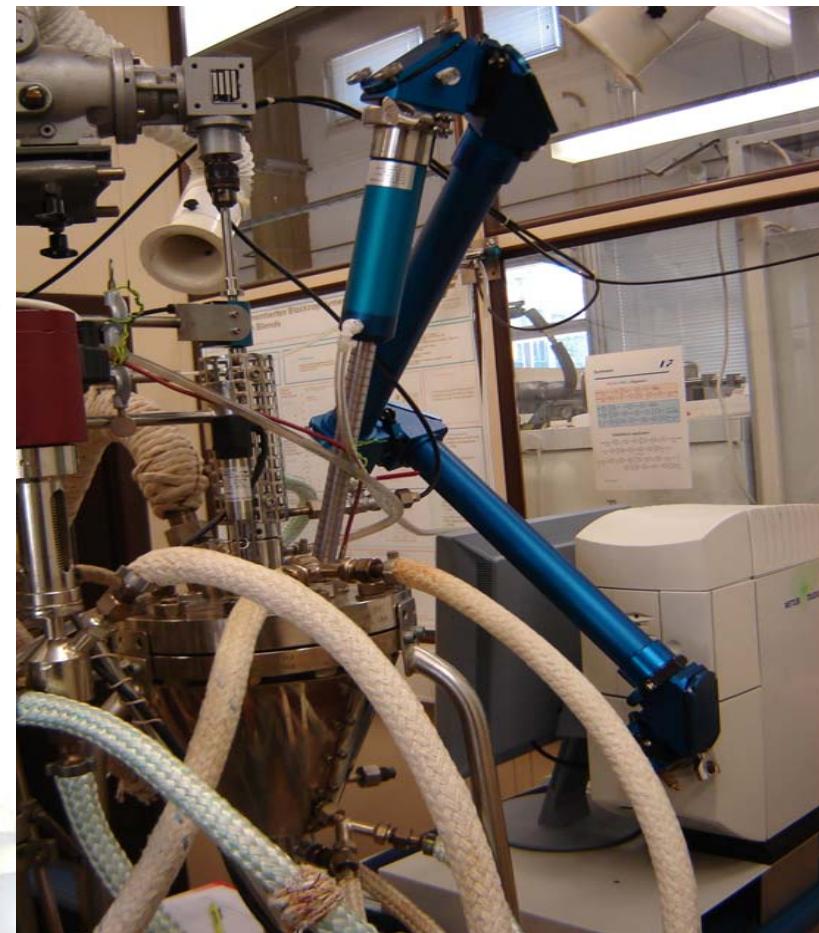
1. Prozessanalytik mit spektroskopischen Methoden in der Polymersynthese und Polymerverarbeitung
2. Methoden und Gerätesysteme
3. Beispiele für Anwendungen zur in-situ Prozessanalytik mit ATR-FTIR und RAMAN Spektroskopie zur Kontrolle von Polymerreaktionen in Lösungen und in Schmelzen
4. Beispiele für Anwendungen zur in-line Prozessüberwachung bei der Extrusion
5. Zusammenfassung

- Sicherung und der Verbesserung der Qualität von Kunststoffen in der polymererzeugenden und polymerverarbeitenden Industrie
  - effektive Prozessführung durch Prozesskontrolle in Echtzeit notwendig
- Fokus dieses Vortrages: FTIR-, NIR- und Raman-Spektroskopie, Ultraschallgeschwindigkeits- und Ultraschalldämpfungsmessungen
  - schnelle, zuverlässige, robuste und zerstörungsfreie Methoden
  - hohen Drücke (400 bar) und hohen Temperaturen (300 °C)
  - Messungen in Reaktoren, Rohrleitungen, Mixern, Extrudern
  - Datenübertragung der gemessenen Daten erfolgt mit Glasfasern bzw. mit Koaxialkabeln, wobei mehrere Messstellen gleichzeitig überwacht werden können
- Anwendungen
  - Chemische Reaktionen, Polymerisationsprozesse und Extrusionsvorgänge überwachen und optimieren
  - Bestimmung der quantitativen Zusammensetzung von Mehrkomponentengemischen (Blends, Composites, Additive)
  - Bestimmung des Umsatzverlaufes von Reaktionen
  - Aussagen zur Morphologie (Teilchengröße und Dispersion)
  - Bestimmung von (End)Produkteigenschaften (Viskosität, mechanische Eigenschaften)
  - Untersuchung des Einflusses der Verarbeitungsbedingungen auf Dispergierprozesse
  - Messungen entlang eines Extruders zur Verfolgung von Misch- und Verteilungsprozessen

## FTIR-ATR Spektroskopie

ATR-IR Spektrometer: ReactIR 4000 (Mettler Toledo)  
4000 - 650 cm<sup>-1</sup>, MCT Detektor

Sensoren: Silizium und Diamant Kristalle



## NIR Spektroskopie

NIR Prozess-Spektrometer SentroProc 1030 – 1660 nm und 1350 – 2037 nm

Sensoren: ½" NIR Sonden für Transmissions und diffuse Reflexionsmessungen (300°C, 150 bar)

## RAMAN Spektroskopie

RAMAN Spektrometer:

HoloProbe (KOSI)

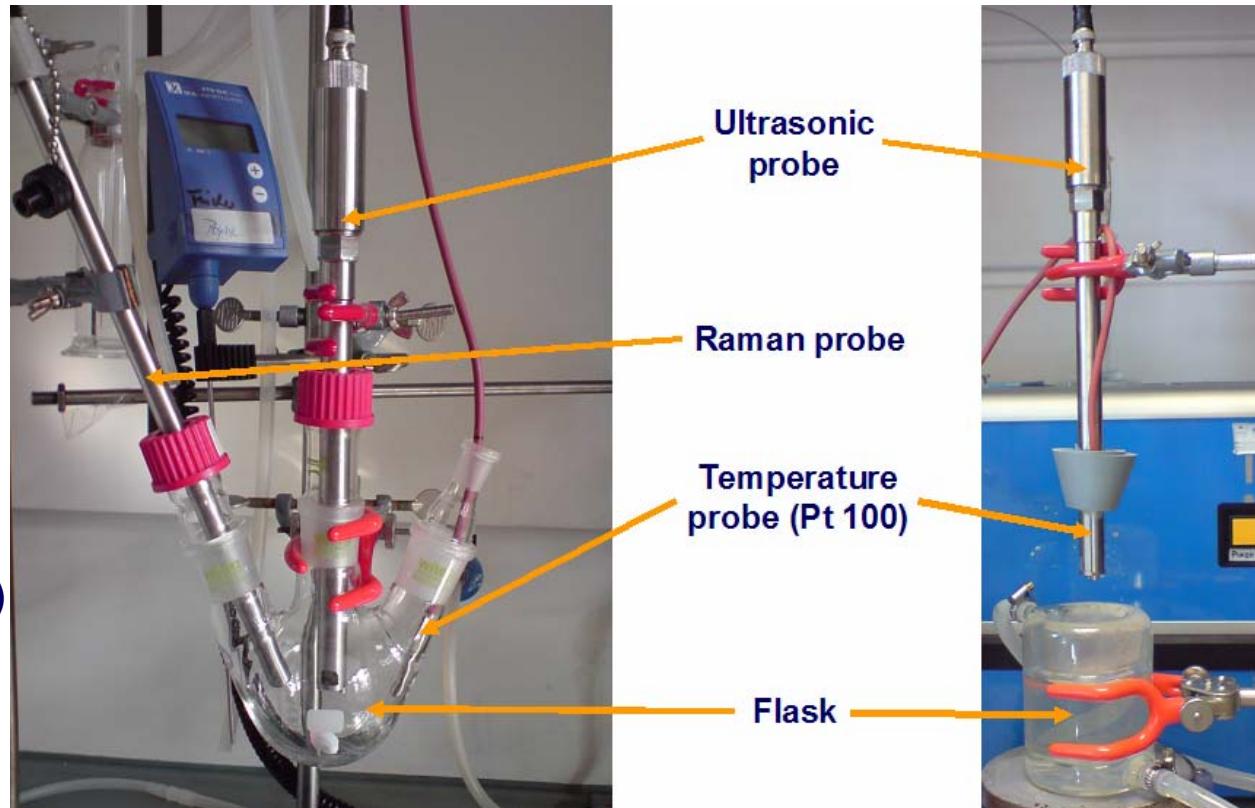
3500 – 150 cm<sup>-1</sup>, CCD Detektor,

Laseranregung: 785 nm

Sensoren:

verschiedene RAMAN Sonden

(1/2" Extruder, short and long focus)



## Ultraschallmessungen

(Ultraschallgeschwindigkeit, Ultraschalldämpfung)

Ultraschall-Transducer: Piezoelektrisch, Anregungsfrequenz: 10 MHz, Nadelimpuls

(U = -40 V, t = 50 ns)

Pulser: US Ultratek PCIPR300

Receiver: Acqiris DP235 (time resolution: 10 ns)

Ultraschalldämpfungsspektren: FFT, Spektralbereich: 3 - 12 MHz

## TEM

- sample preparation: cryo ultracutting by Leica Ultracut UCT, layer thickness approx. 80 nm
- TEM: JEM 2010, 200 keV

## SEM

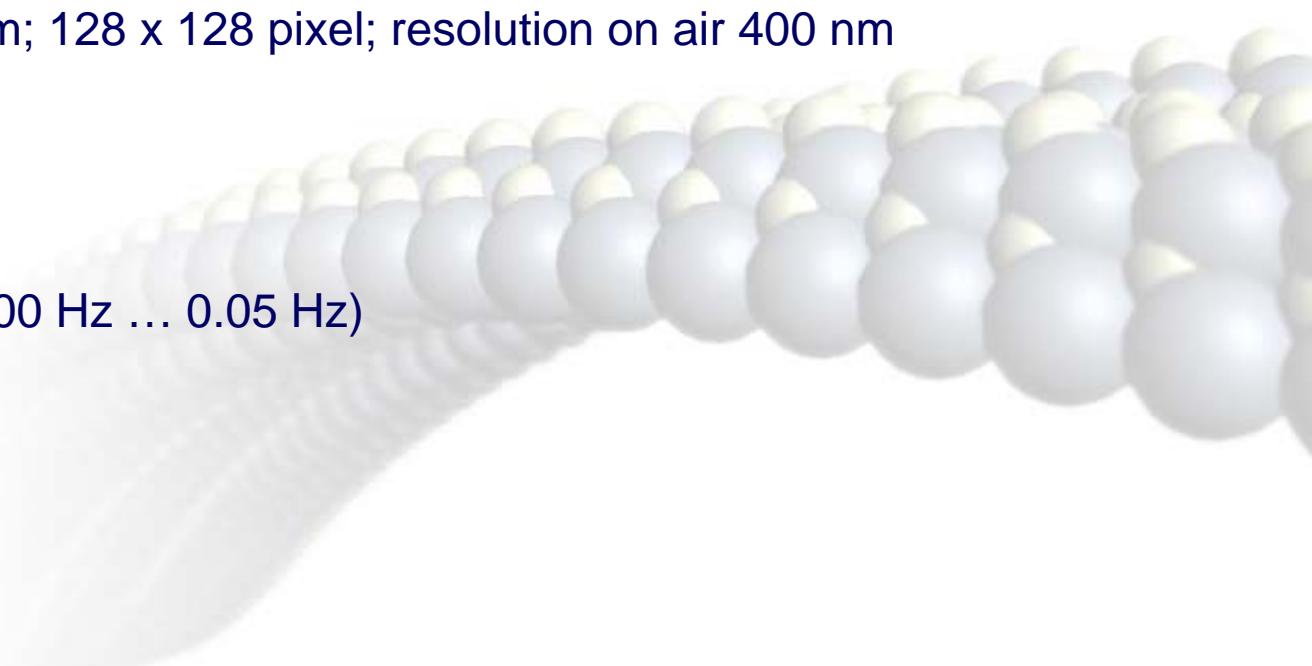
- sample preparation: brittle fractured samples at low temperature, sputtered with 5 nm Pt
- SEM: Ultra Plus Zeiss, Germany, 3kV detector: SE2

## RAMAN-Imaging

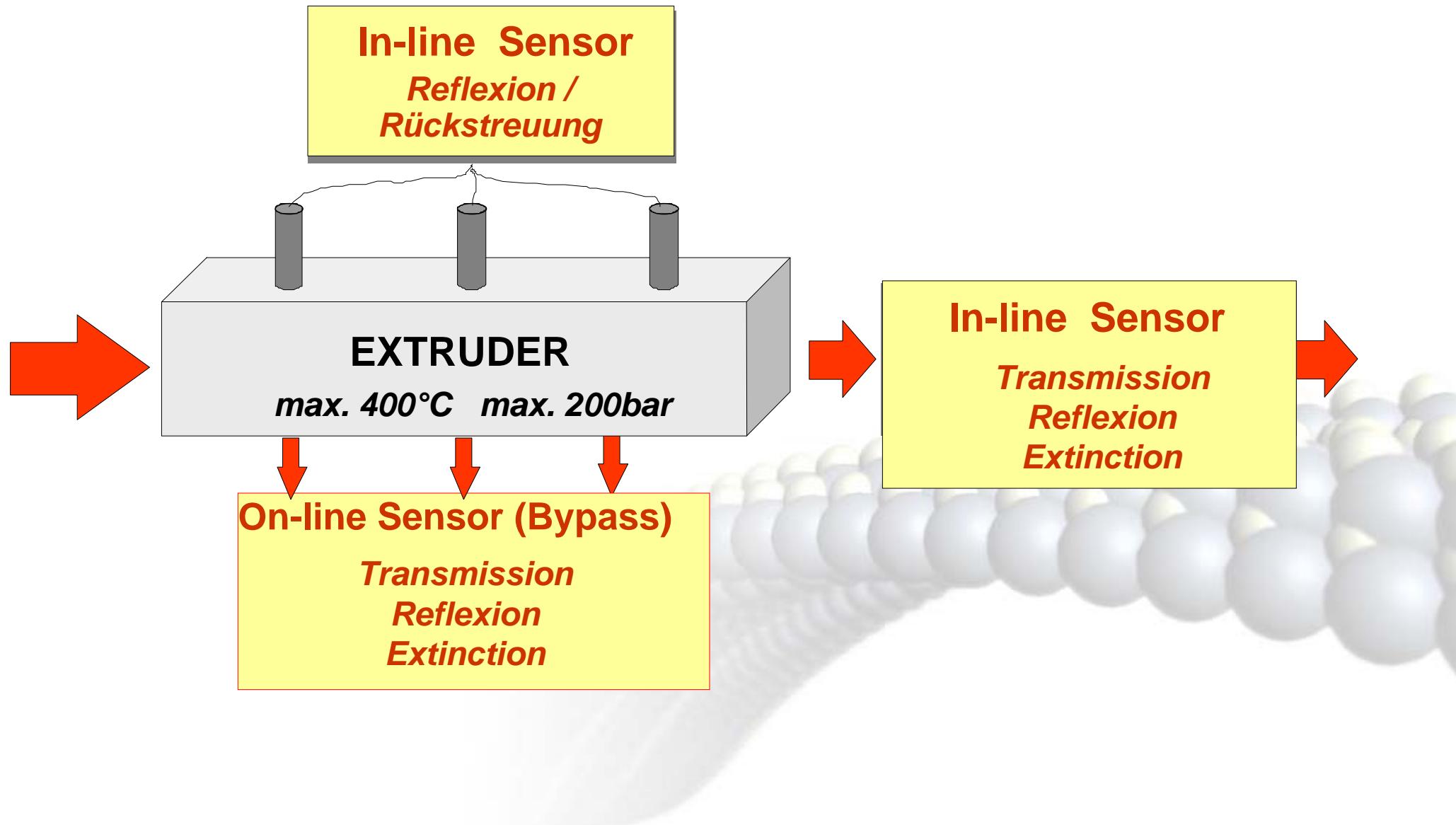
- Alpha300 R, Confocal Raman Microscope, WITec GmbH Ulm, Germany
- Nd:YAG laser 532 nm, 100x objective
- scan range xy-scan: 25 x 25 µm; 128 x 128 pixel; resolution on air 400 nm
- 62 ms / spectrum

## Rheology

- ARES rheometer - 240 °C
- dynamical frequency sweep (100 Hz ... 0.05 Hz)



## Extrusion: mögliche Messstellen entlang des Extruders für Messadapter mit verschiedenen Sensoren

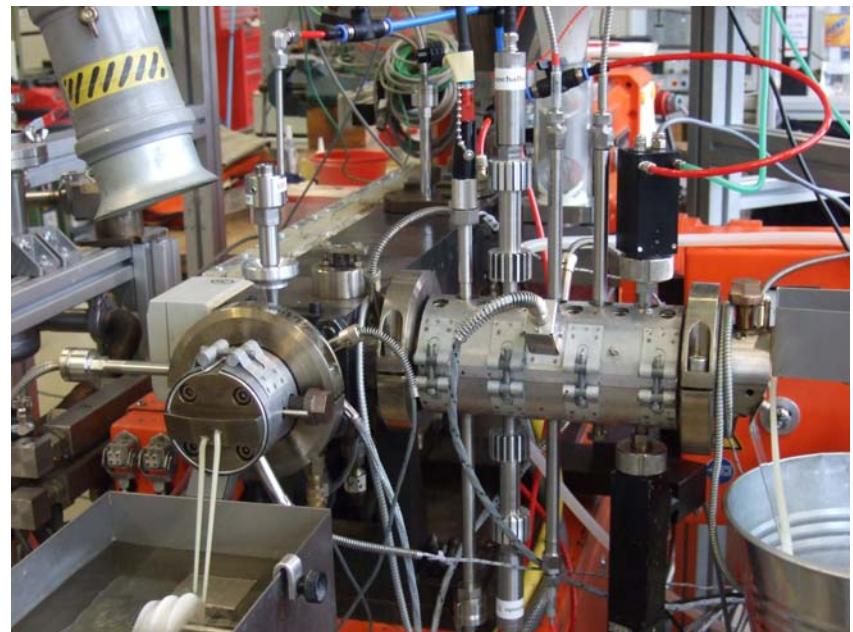


## On-line system

extracts the sample continuously (bypass) and presents it to the instrument

**advantage:** separated from the process stream, so temperature and pressure can be controlled

**disadvantage:** time lag between sampling and measurement



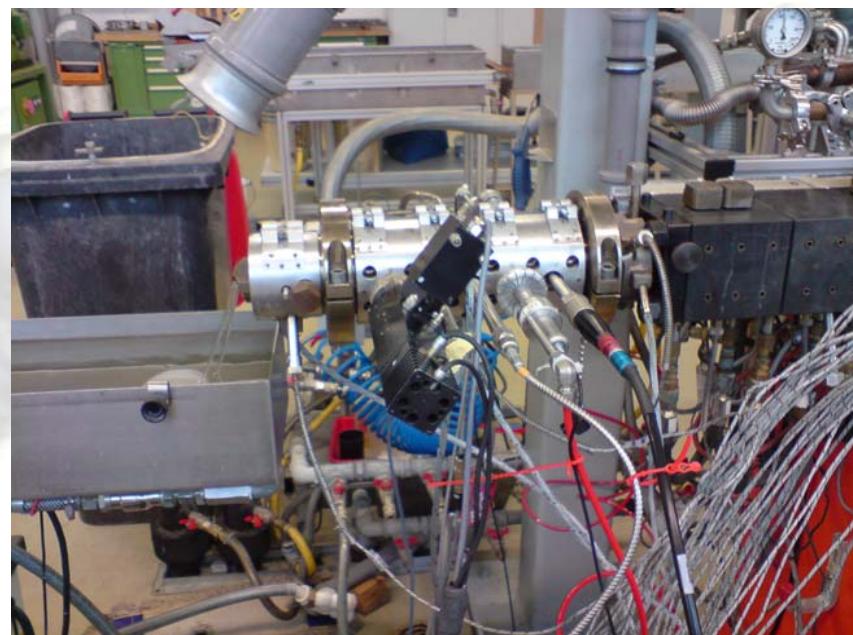
## In-line system

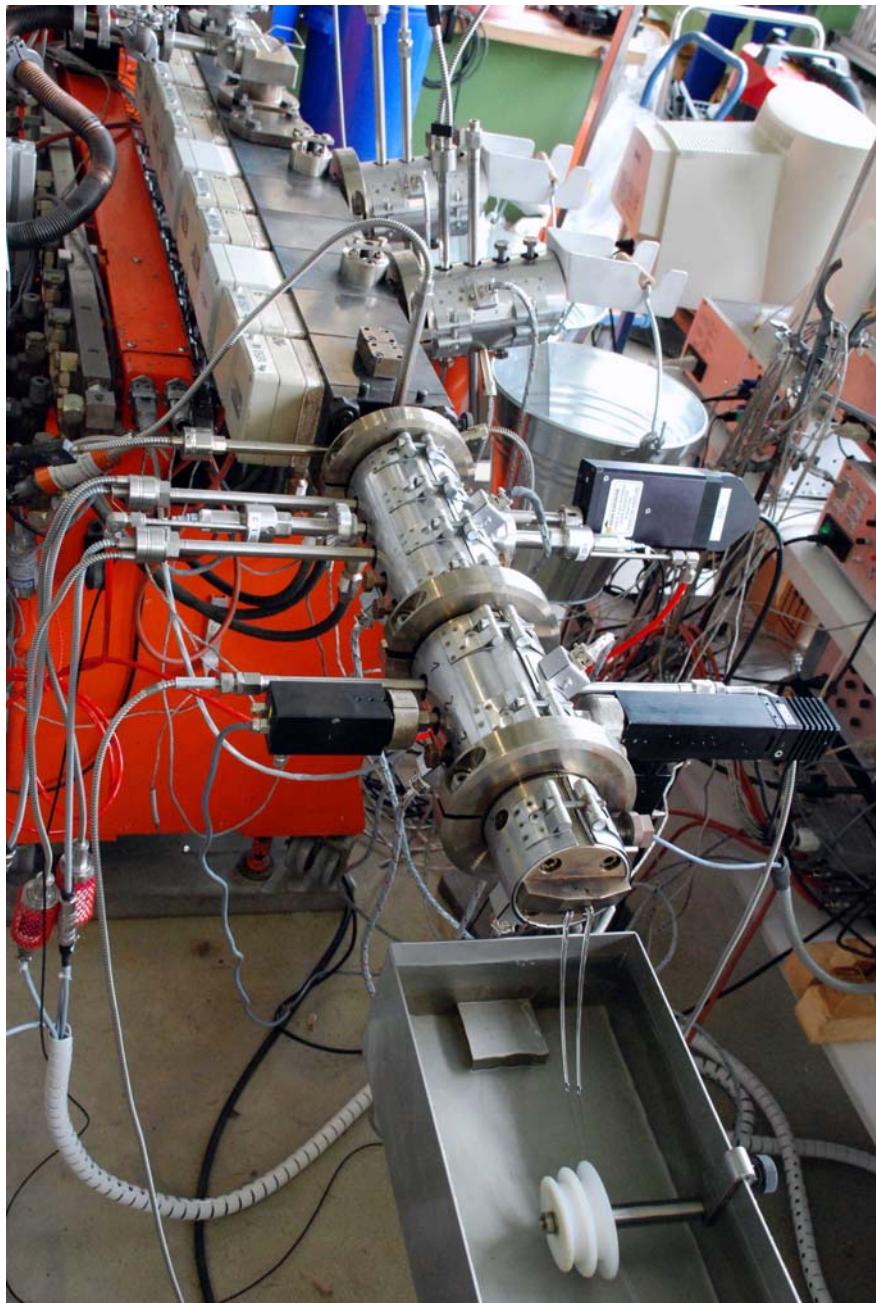
measurement is done directly in the process line

**advantage:** no sampling delays

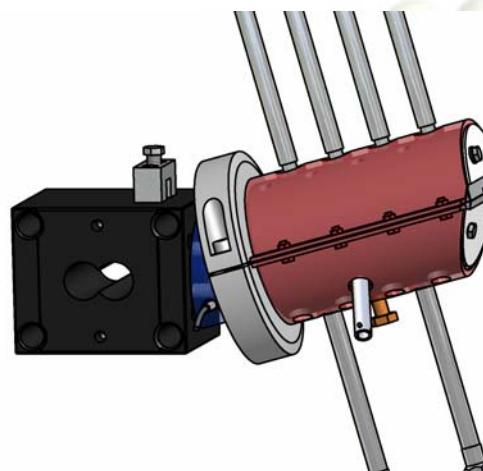
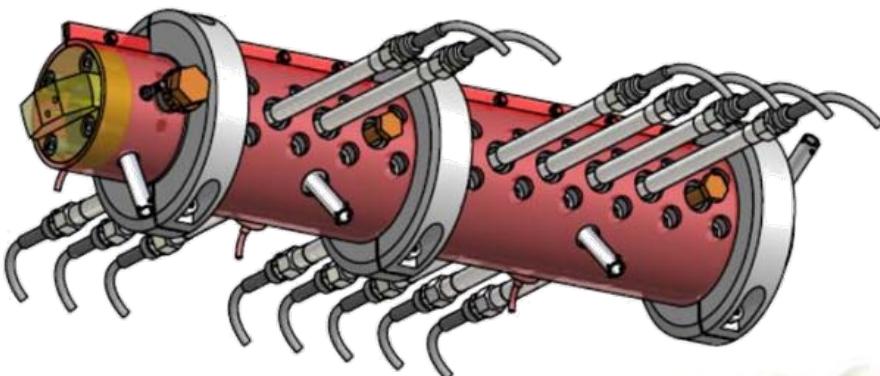
**disadvantage:**

- measurement is limited by temperature and pressure variations
- the probe can interfere with the main process stream





Extruder with end adapters at the die and two side outlet adapters and sensors for **Ultrasonic, NIR, UV/VIS, Raman, Light extinction**, pressure and temperature measurements



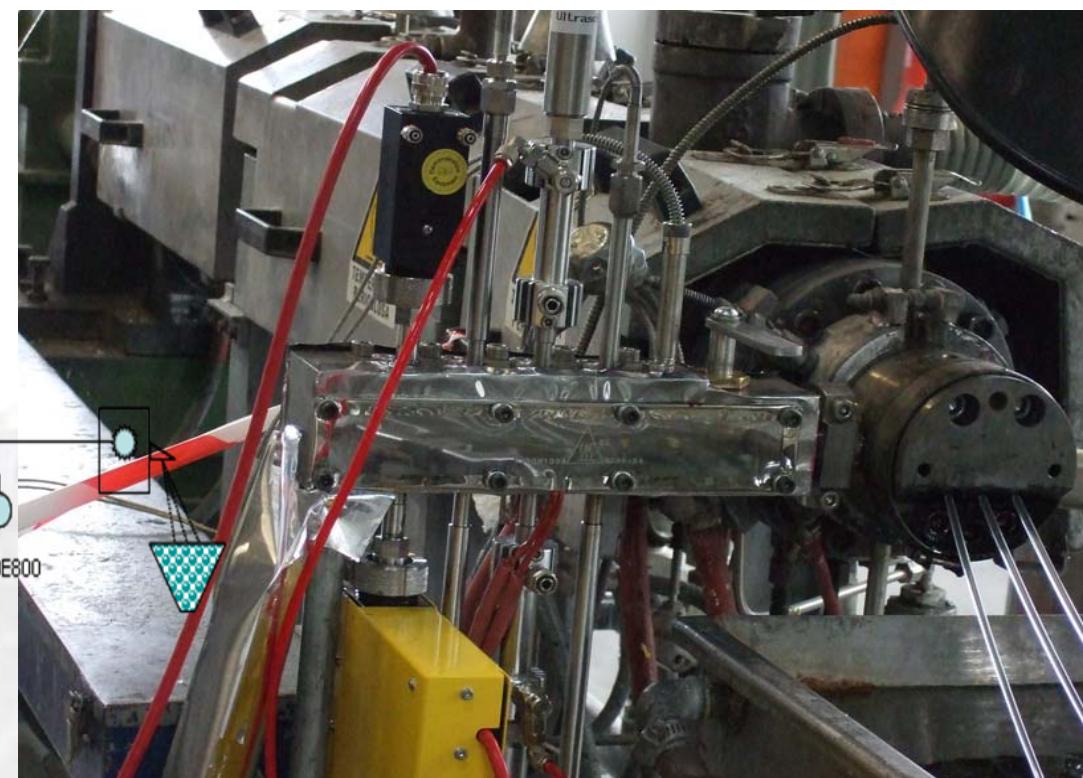
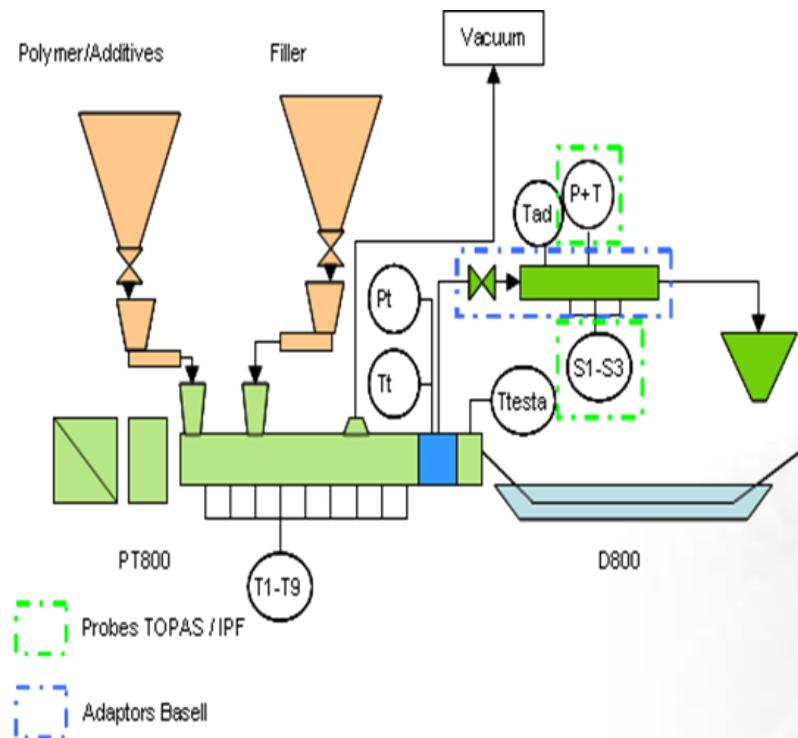
## Measuring adapters

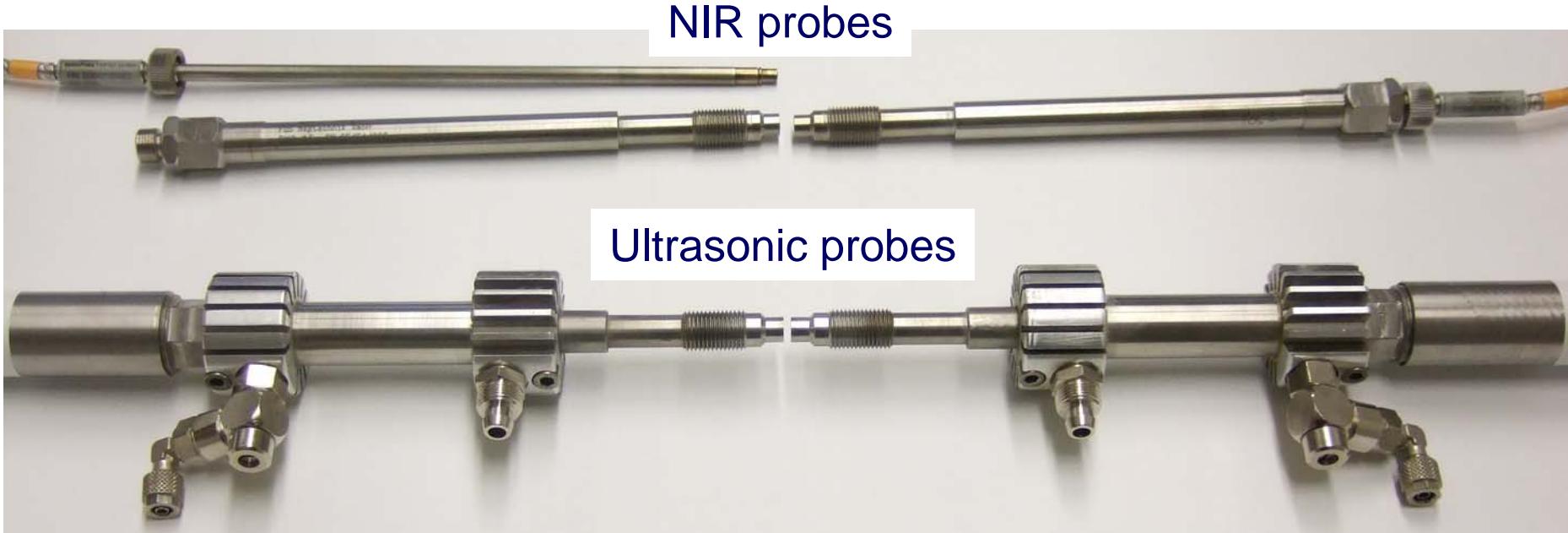
- Up to 16 (in-line) and 7 (on-line) different probes (NIR, US, UV/Vis, Raman)
- 1/2 " industry standard

## Industrial Extruder

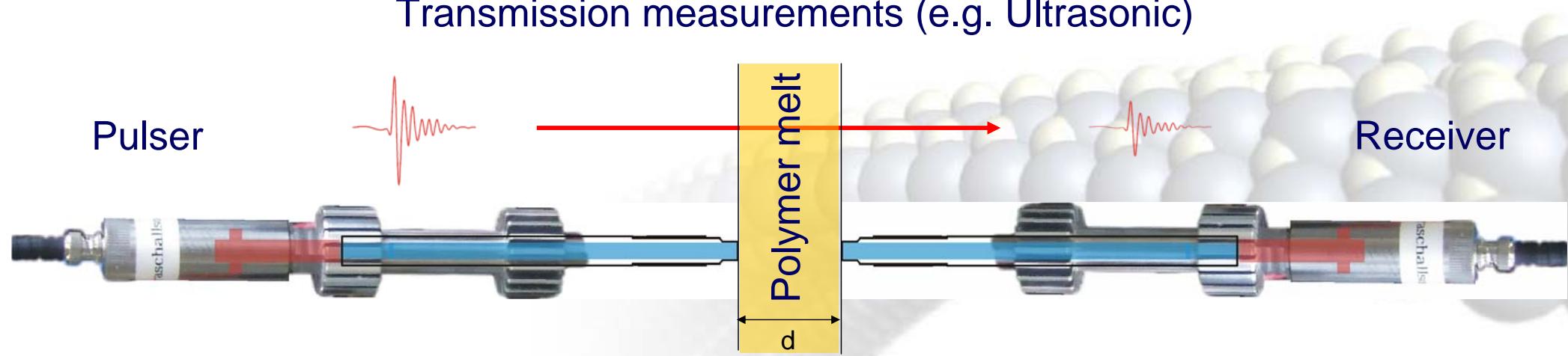
Maris TM45 twin-screw extruder, T = 180 - 210 °C , N = 150 - 300 rpm, Q = 35 - 70 kg/h

Configuration of extruder Maris 45 for scale up with on-line bypass adapter and sensors for NIR, Ultrasonic, light extinction, pressure and temperature





Transmission measurements (e.g. Ultrasonic)



## Multivariate Daten Analyse (Chemometrie) – Grundprinzipien

Reduction of variables to principle components (PC)

Algebra of matrices

Simplification of measured information, reduction to spectral changes

Finding a model to describe the system without input of information

Application to real parameters

Chemometrics: Principal Component Regression PCA  
Partial Least Squares PLS

**n dimensional**  
**range of measured**  
**variables or spectra**

$n \gg m$

**m dimensional**  
**range of PC**

## Multivariate Daten Analyse (Chemometrie) – Grundprinzipien

Calibration

In-line measured  
values (spectra)  
with known  
properties



Off-line measured  
values (particle size,  
mechanical properties,  
shear thinning exp.)

Model  
(PC)

Validation

In-line measured  
values (spectra)  
with unknown  
properties



Model  
(PC)

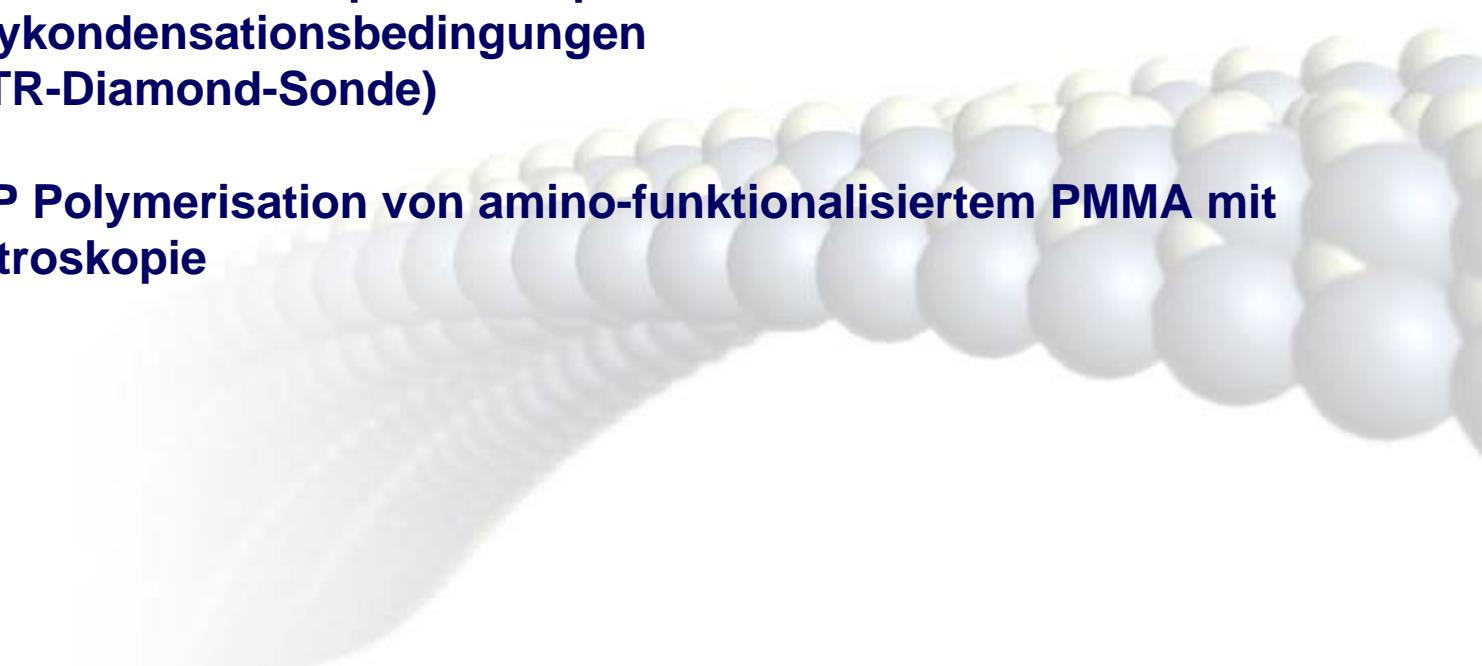


real time  
predicted values  
(particle size,  
mechanical properties,  
shear thinning exp.)

Validation is comparison between  
with off-line measured values  
real time predicted values

### 3. Beispiele für Anwendungen zur in-situ Prozessanalytik mit ATR-FTIR und RAMAN Spektroskopie zur Kontrolle von Polymerreaktionen in Lösungen und in Schmelzen

- # **Monitoring der Modifizierung von OH Endgruppen von hochverzweigten poly(urea-urethanen) mit Isocyanaten mit ATR-IR Spektroskopie (ATR-Si-Sonde)**
- # **Überwachung der Polykondensation von Polyethylenterephthalat (PET) in einem Rührautoklaven mit ATR-IR Spektroskopie mit dem Ziel der Optimierung der Polykondensationsbedingungen (Hochtemperatur ATR-Diamond-Sonde)**
- # **Verfolgung der ATRP Polymerisation von amino-funktionalisiertem PMMA mit in-line RAMAN Spektroskopie**





**ATR-IR spectrometer:** ReactIR 4000 (Mettler Toledo)  
4000 - 650 cm<sup>-1</sup>, MCT detector

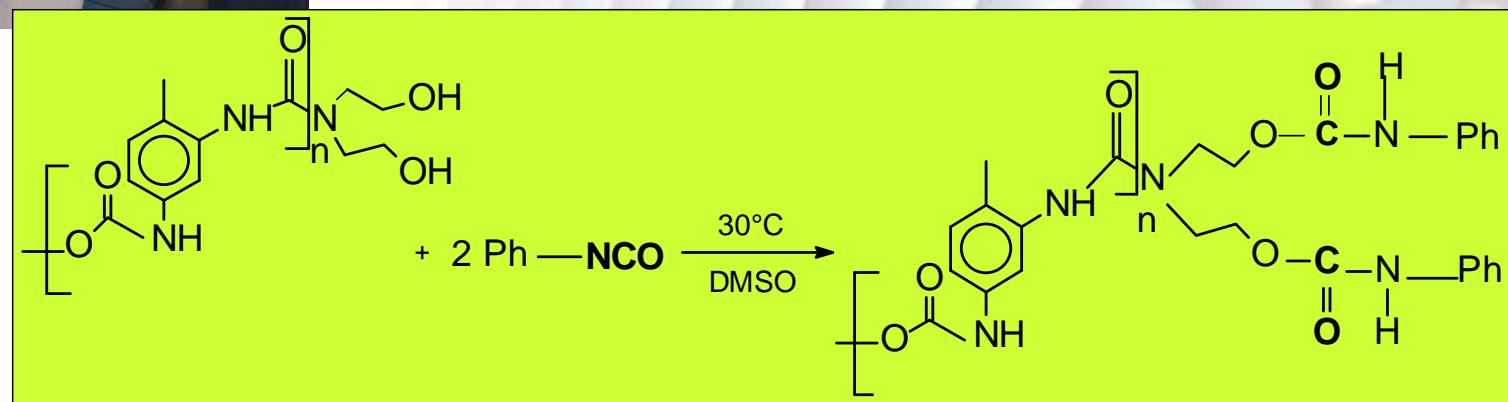
**Probe:** ATR (SiComp) probe  
gold coated moveable tubes as lightpipe

**Conditions:**

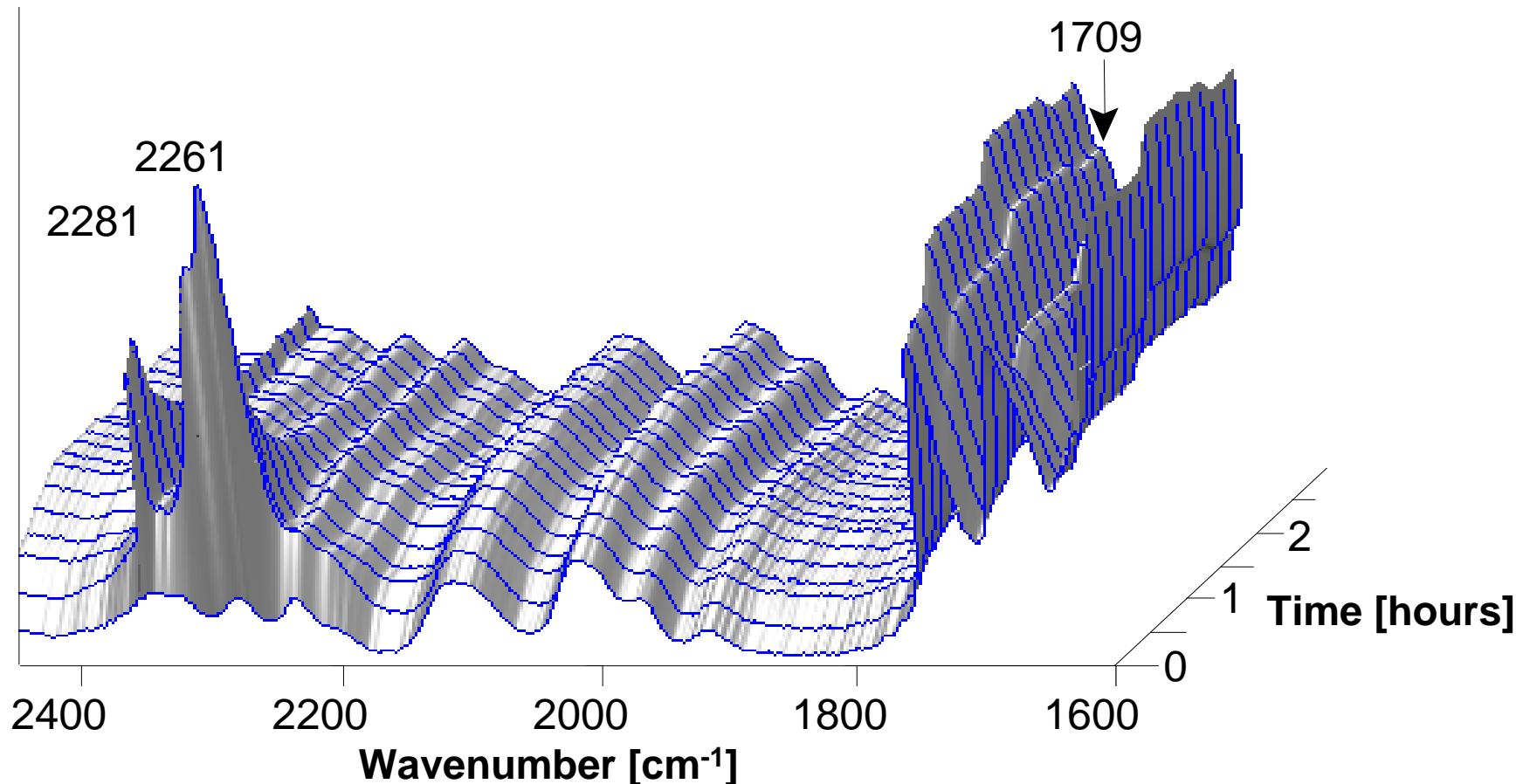
250 ml flask under Argon  
reaction time: 3 hours

room temperature

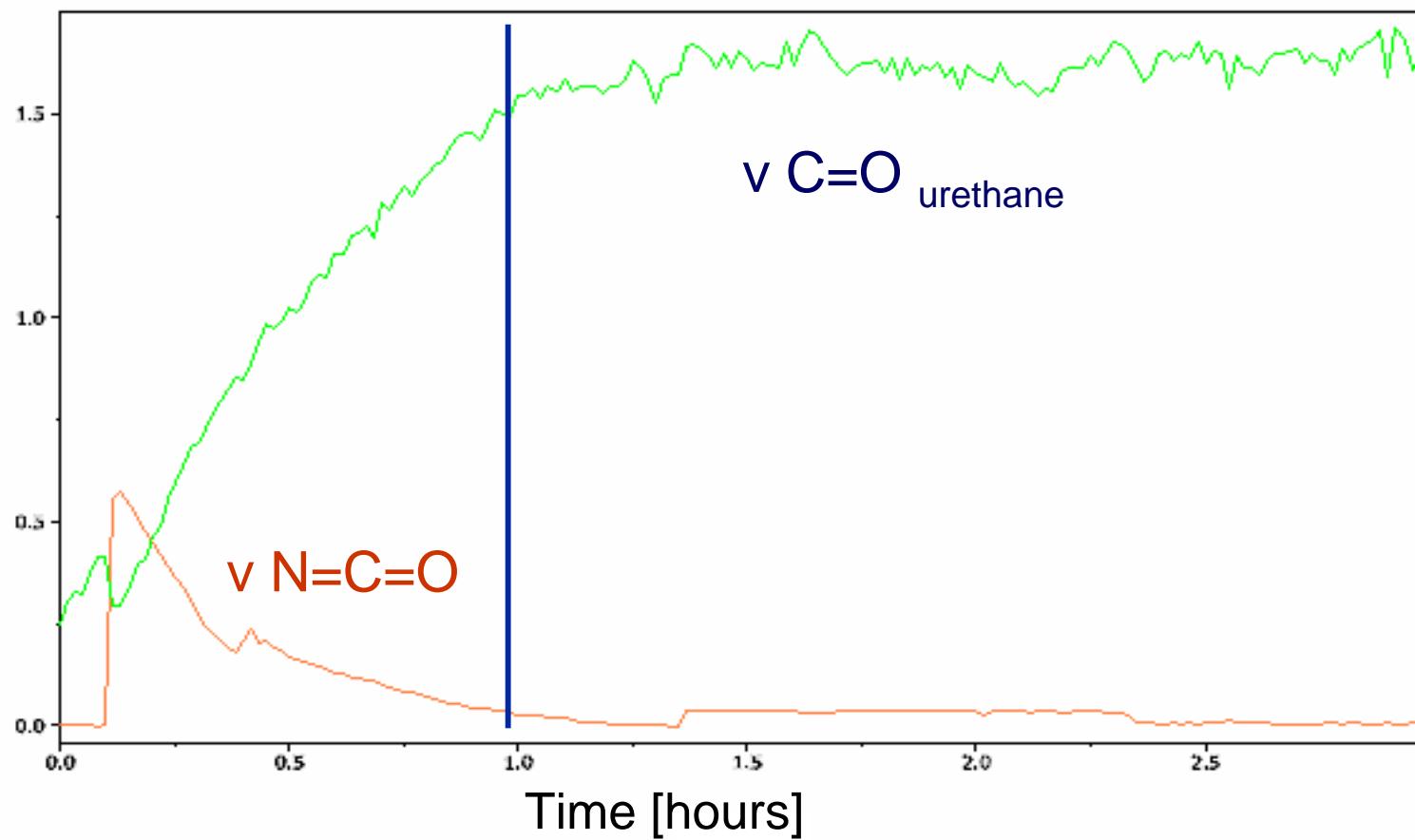
time: every minute an IR  
spectrum



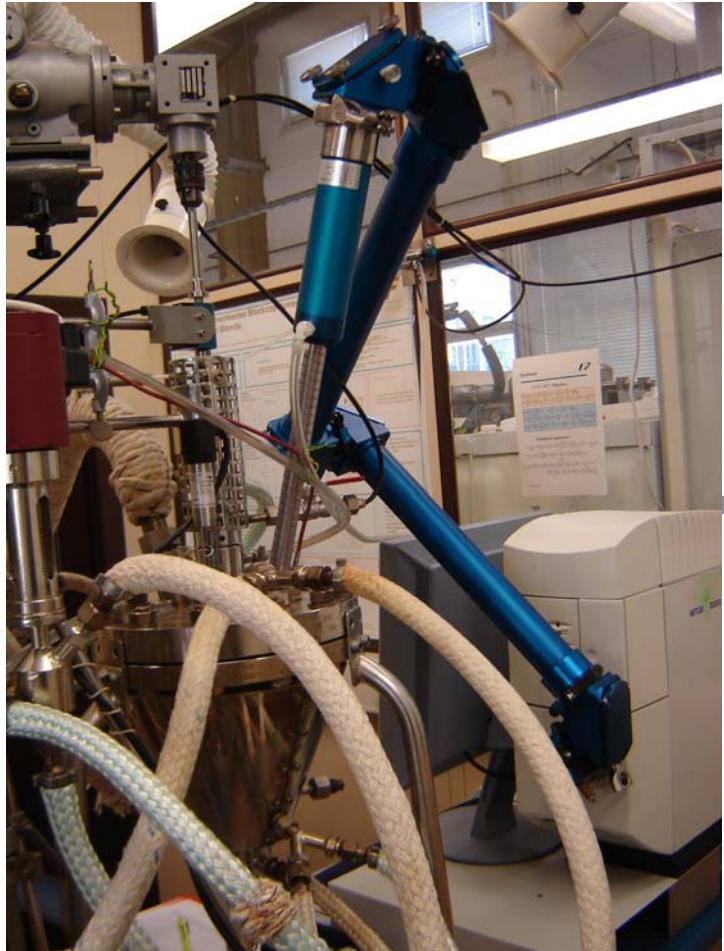
Detection of the degradation of NCO at  $2281 + 2261 \text{ cm}^{-1}$   
Creation of the urethane endgroup C=O at  $1709 \text{ cm}^{-1}$



We could detect that the reaction was terminated after 1 hour



Collaboration with T. Long, Virginia Tech



## Aims:

- Adaption of the ATR-FTIR equipment to the 2 L-stirring autoclave
- Real-time plots of the FTIR-spectra during polycondensation reaction at 280°C
- Optimization of the polycondensation conditions by the received information

## Conditions:

2.3 L-stainless steel stirring autoclave with vacuum line

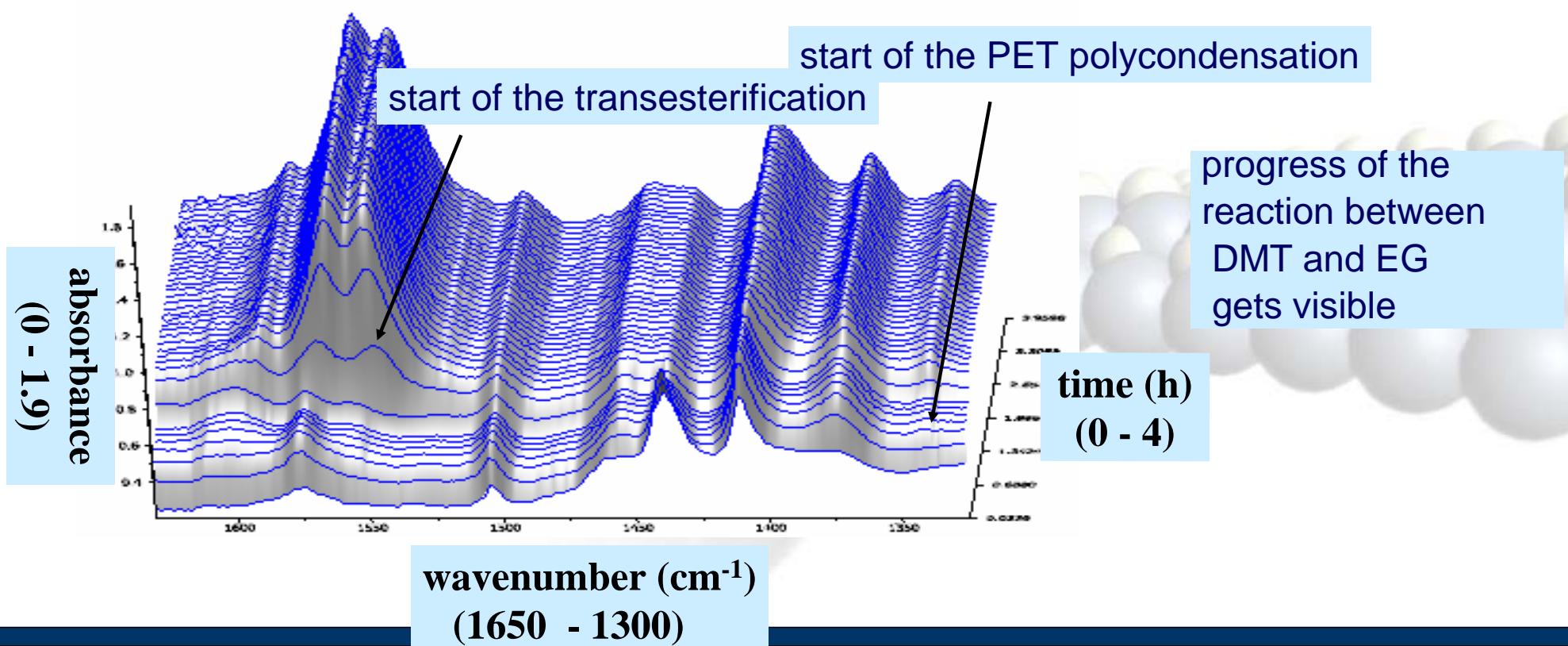
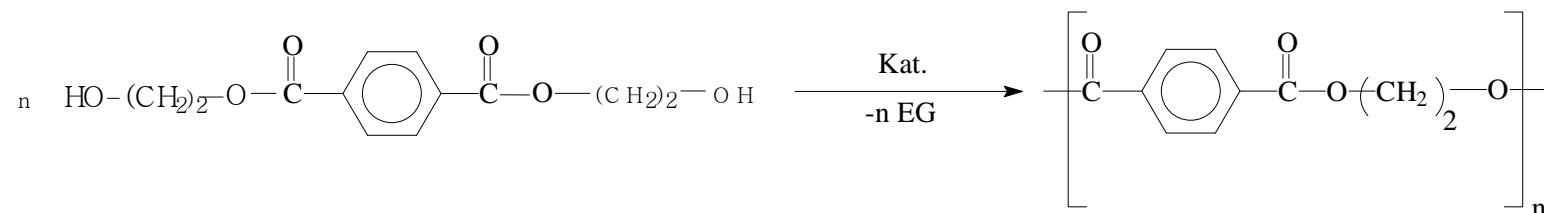
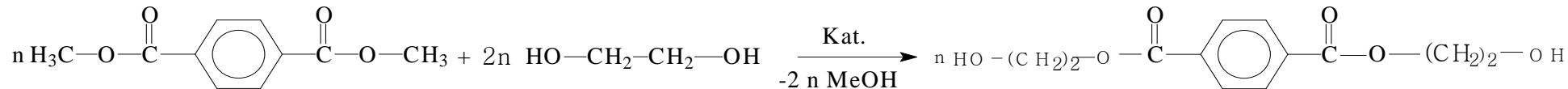
High temperature diamond ATR probe

reaction time: 7 hours (45 min at 200 °C, 60 min at 260 °C, 415 min at 280 °C)

Temperature: 280°C

time: every 4 minutes an IR spectrum (150 scans)

# Überwachung der Polykondensation von Polyethylenterephthalat (PET) in einem Röhrautoklaven mit ATR-IR Spektroskopie



aim of this synthesis was the creation of a reactive polymer for a consecutive application in cross linkable polymer blends

the **AtomicTransferRadicalPolymerisation** was carried out in solution at 25 °C in a flask

the kinetics of the polymerization was studied in anisole by in-line RAMAN spectroscopy and off-line  $^1\text{H}$  NMR spectroscopy

measure time: every two minutes a RAMAN spectrum

the probe was connected via optical fibers with the RAMAN spectrometer

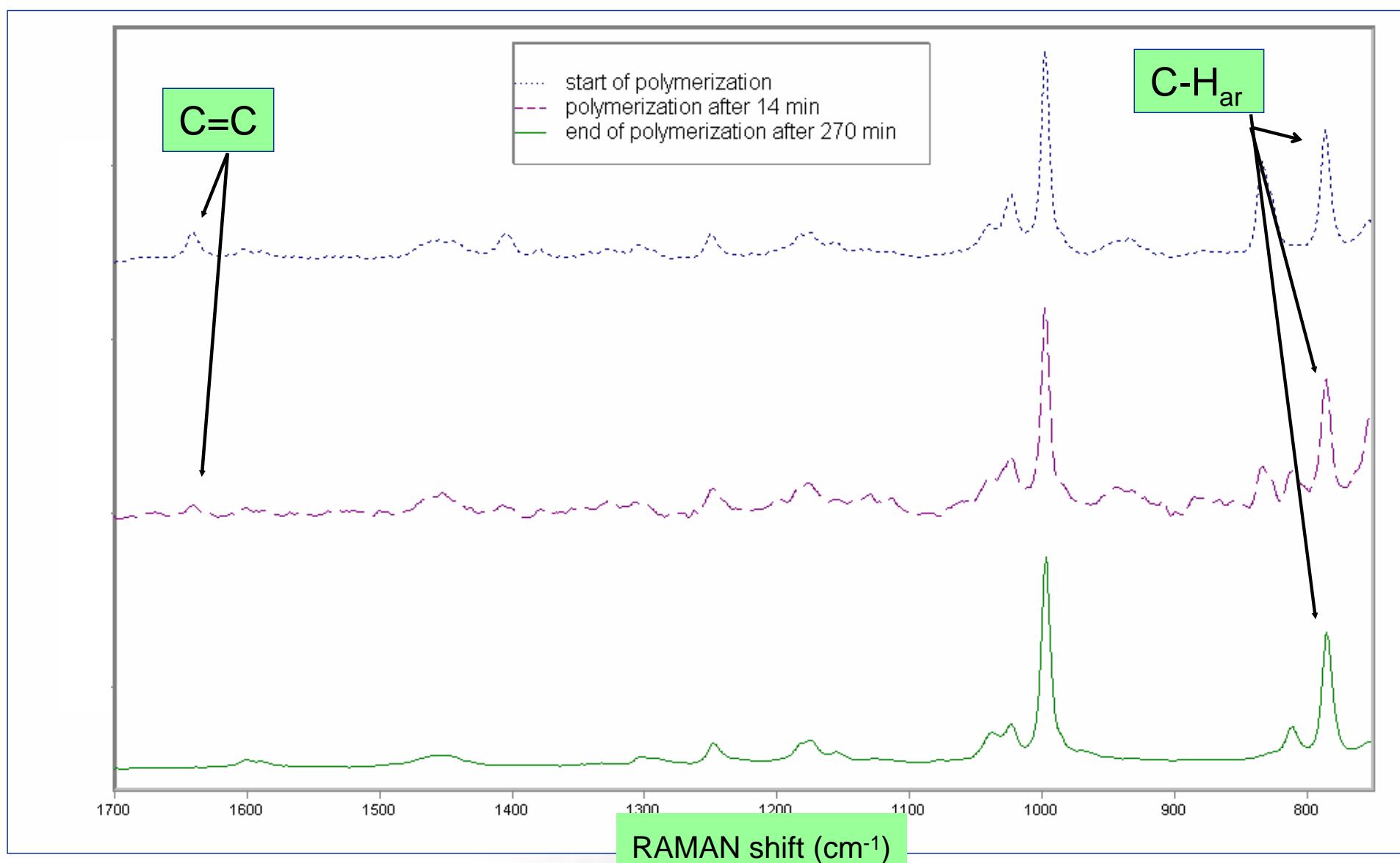
RAMAN spectrometer: HoloProbe (Kaiser Optical Systems)

laser: 785 nm

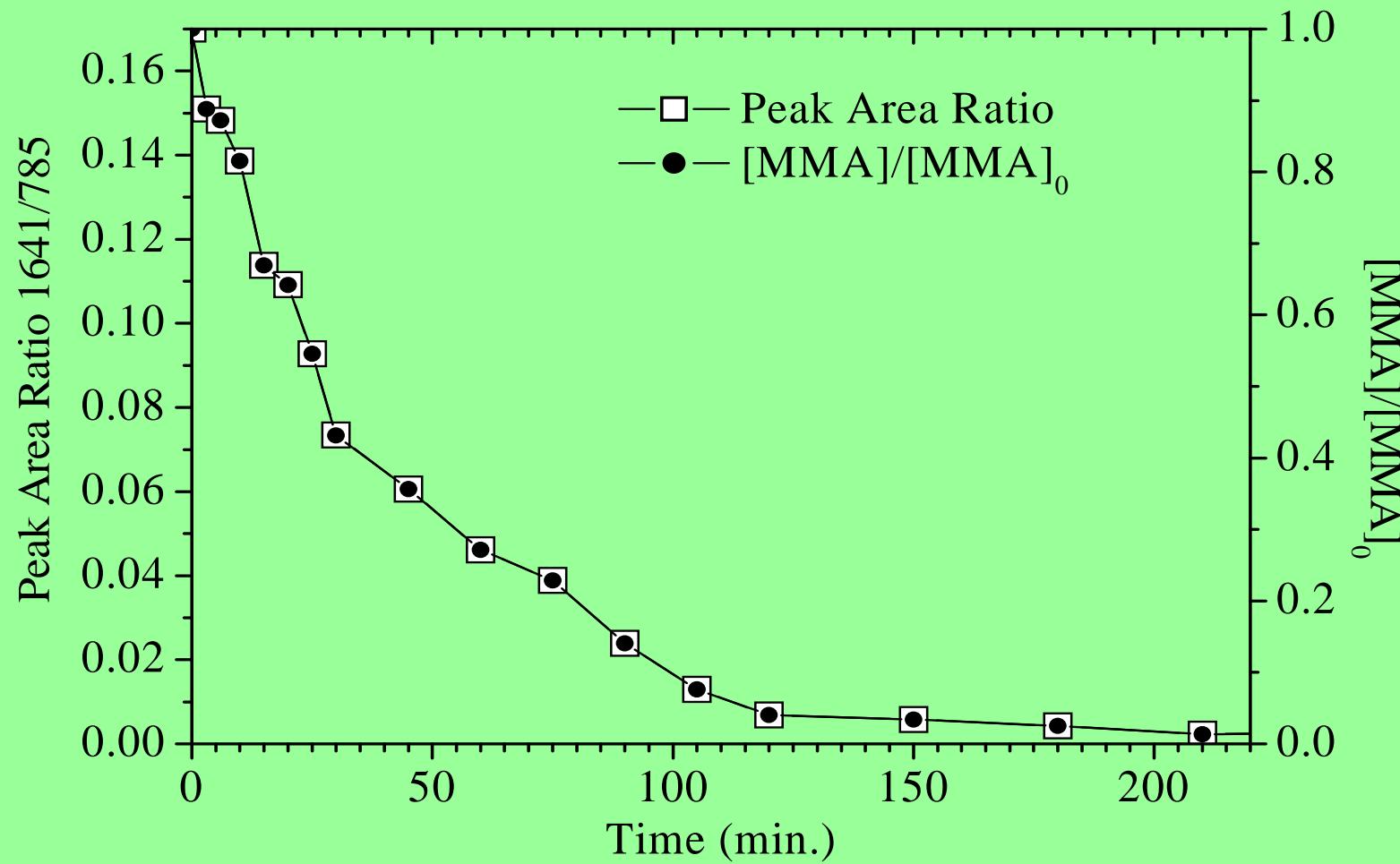
detector: CCD camera

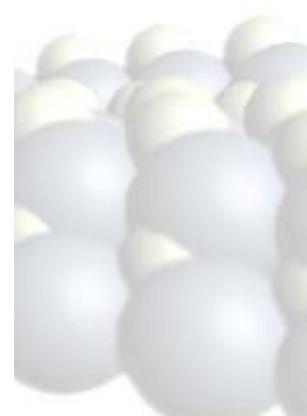
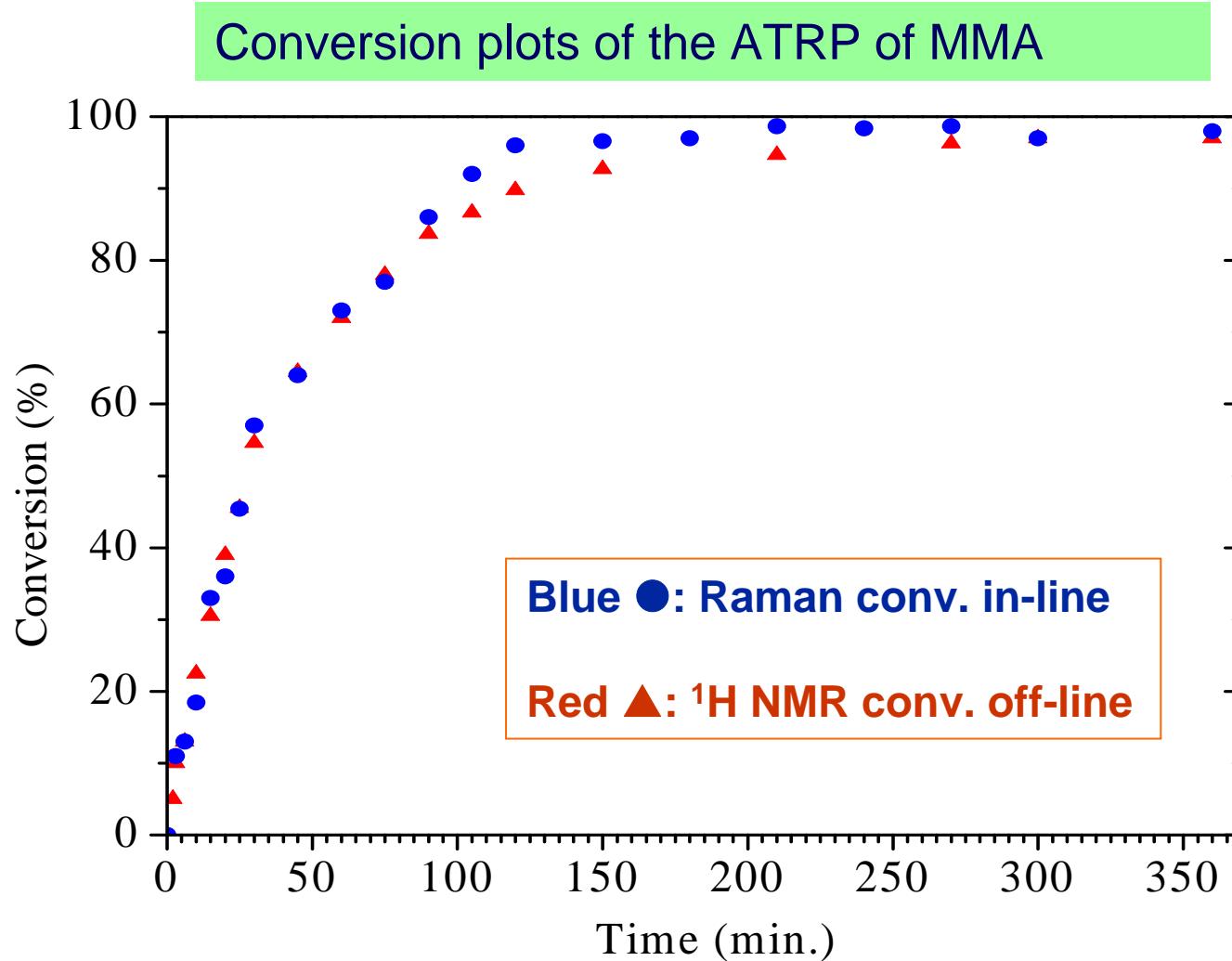
resolution: 4  $\text{cm}^{-1}$

# Verfolgung der ATRP Polymerisation von aminofunktionalisiertem PMMA mit in-line RAMAN Spektroskopie



# Verfolgung der ATRP Polymerisation von aminofunktionalisiertem PMMA mit in-line RAMAN Spektroskopie





## 4. Beispiele für Anwendungen zur in-line Prozessüberwachung bei der Extrusion

- # Bestimmung des Rest-Gehaltes und der Nachweisgrenze von Vinylacetat in PP/EVA-Blends mit in-line RAMAN
- # Bestimmung der Partikelgröße von SiO<sub>2</sub>- und Talkum-Partikeln in PP mit NIR, RAMAN und Ultraschalldämpfung
- # Quantitative Vorhersage der Dispersion von Nanofüllstoffen (modifizierte Schichtsilikate) in PP und PA6 mit NIR und Ultraschallmessungen bei variierenden Prozessbedingungen
- # Bestimmung des Exfolierungsgrades von PP Nanocompositen zur Untersuchung des Dispersionsprozesses entlang des Extruders mit NIR und Ultraschallmessungen bei variierenden Prozessbedingungen
- # Scale-up (Faktor 10) mit NIR und Ultraschallmessungen auf einen Industrie-Extruder zur Kontrolle der Dosierung und des Exfolierungsgrades für PP Nanocomposite
- # Monitoring der Reaktiv-Extrusion zur in-situ Generierung von nanoskaligen TiO<sub>2</sub>-Partikeln in PP

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Aim was the determination of the efficiency of Raman-spectroscopy for the quantitative limit of detection and the accuracy of the VA-content in real time during extrusion.

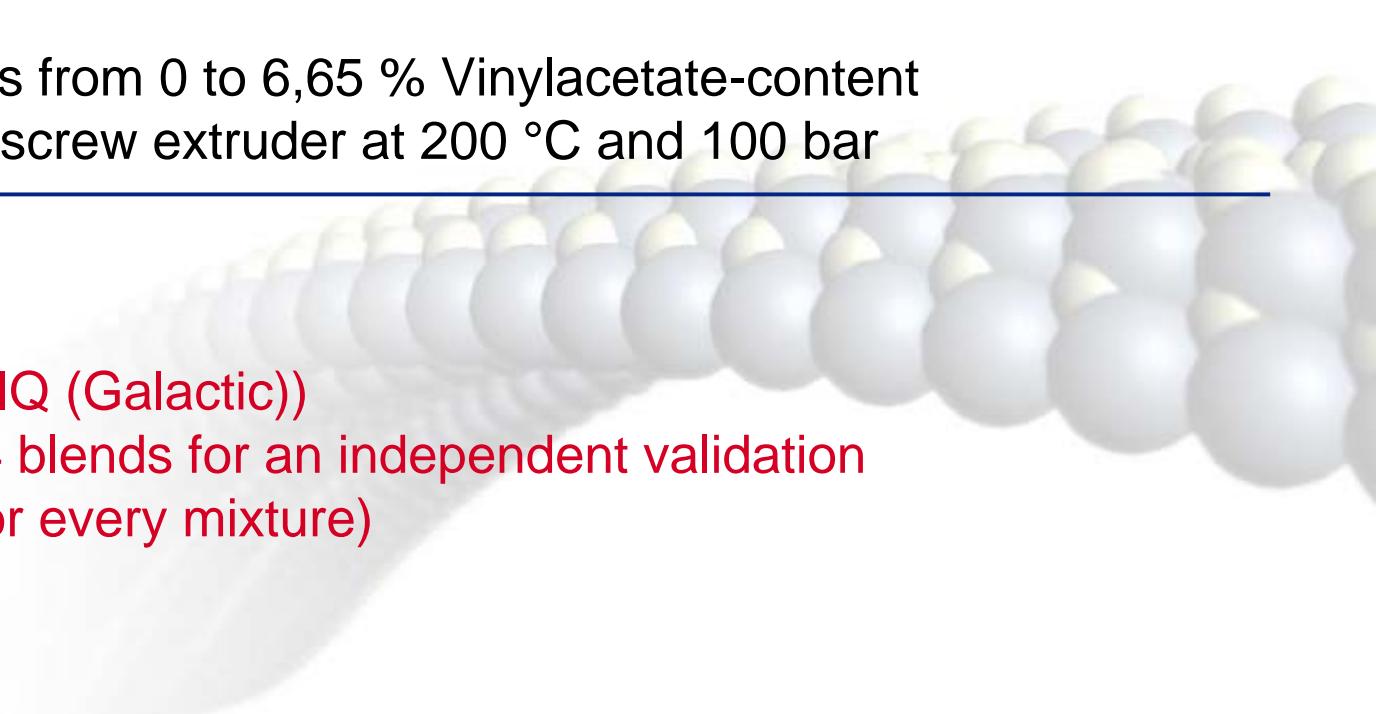
Raman high temperature and high pressure probe  
measure time: 300 ms/scan, 40 scans  
fibers: 50 µm (from laser) and 100 µm (to CCD-detector)

24 different PP/EVA blends from 0 to 6,65 % Vinylacetate-content were extruded in a single screw extruder at 200 °C and 100 bar

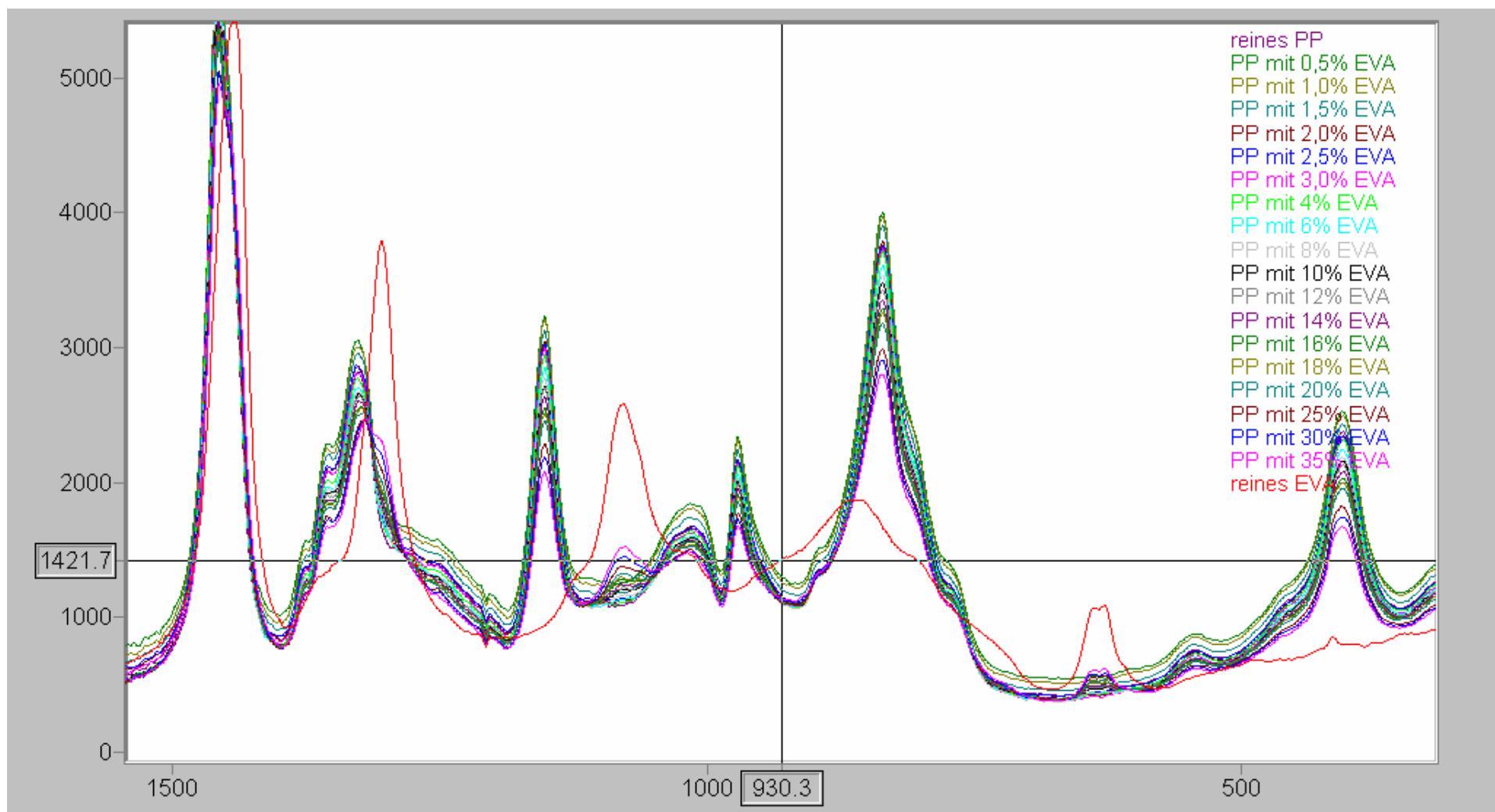
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## Chemometrics

PLS (GRAMS32/PLSplus/IQ (Galactic))  
20 blends for calibration, 4 blends for an independent validation  
240 Spectra (10 spectra for every mixture)

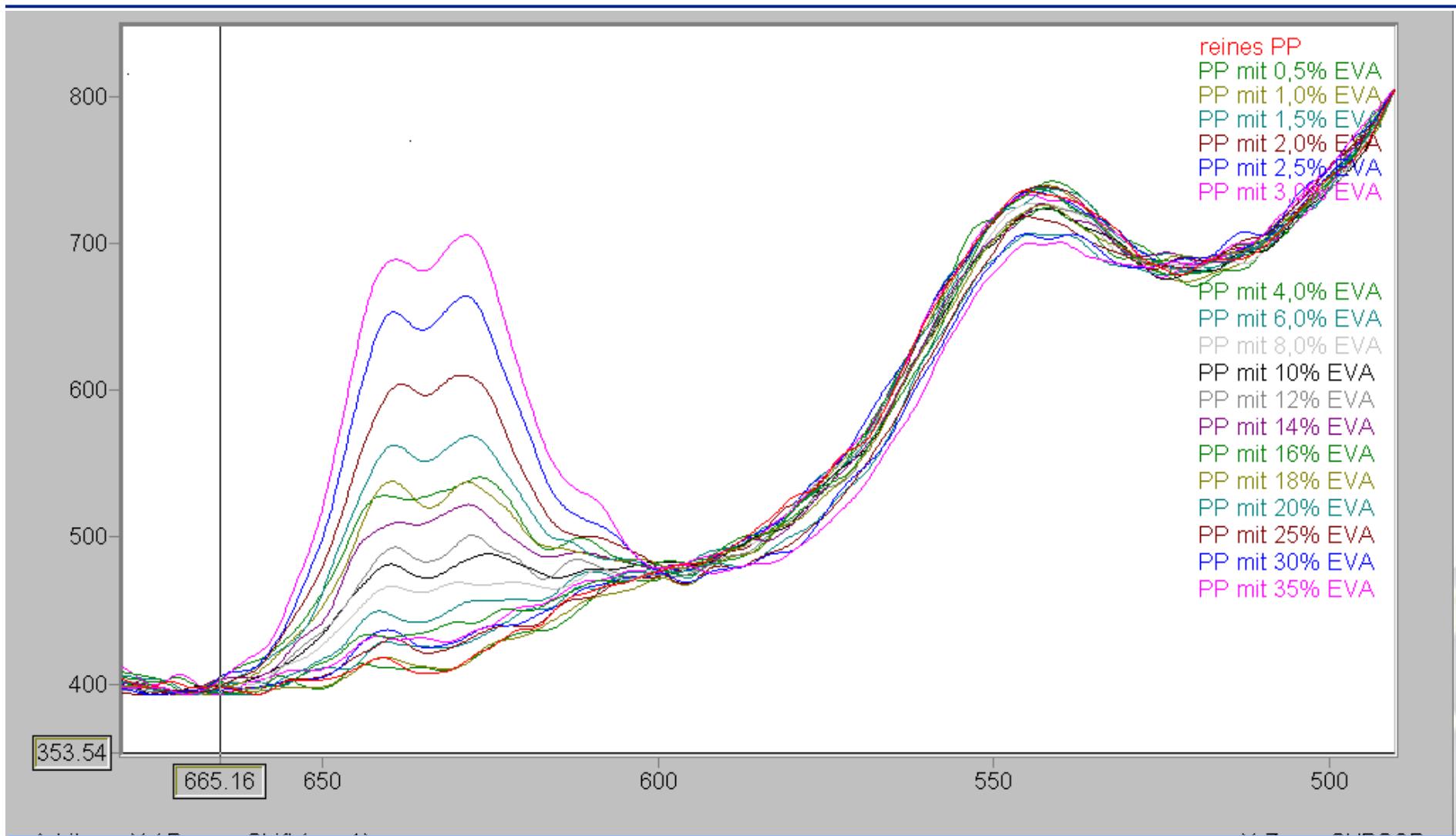


# Bestimmung des Rest-Gehaltes und der Nachweisgrenze von Vinylacetat in PP/EVA-Blends mit in-line RAMAN



**PP/EVA - in-line RAMAN-Spectra**

# Bestimmung des Rest-Gehaltes und der Nachweisgrenze von Vinylacetat in PP/EVA-Blends mit in-line RAMAN



PP/EVA - in-line RAMAN-Spectra (630 cm<sup>-1</sup>)

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## Quantitative Analysis

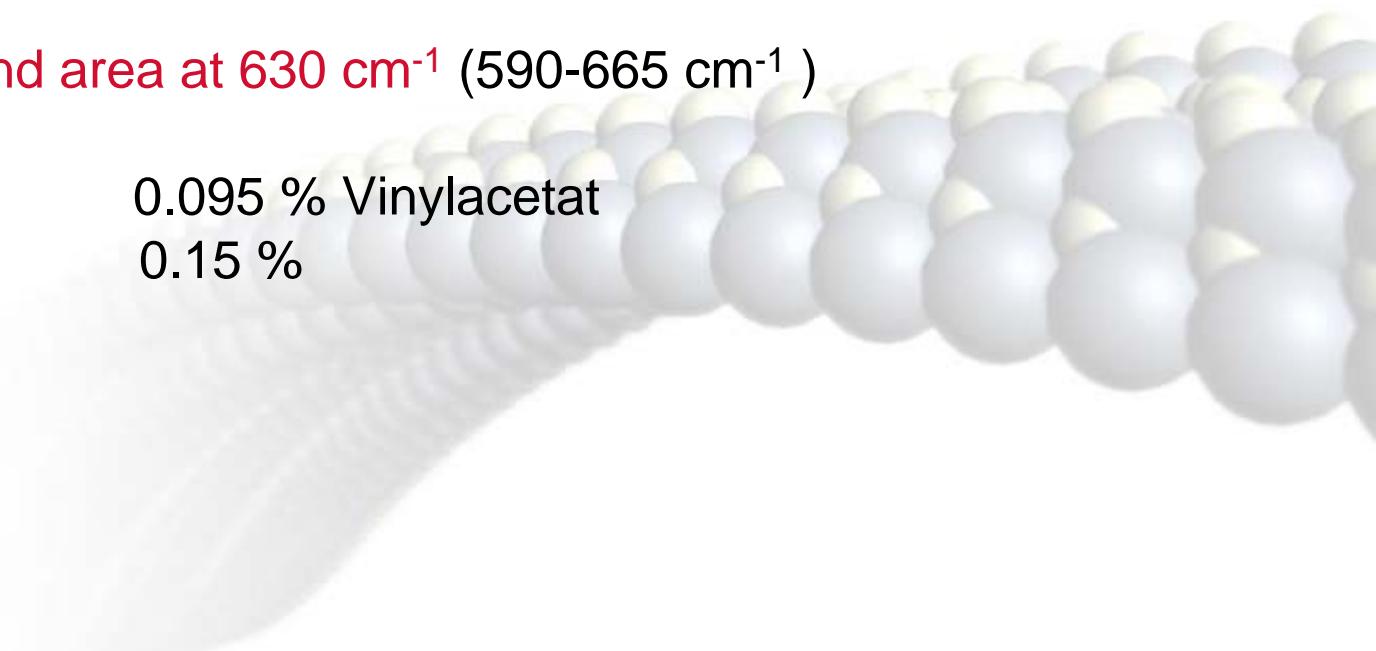
### I. Chemometrics – PLS (350 bis 1600 cm<sup>-1</sup>)

R<sup>2</sup> : 0,9937 (3 components)  
detection limit: 0.19 % Vinylacetate  
accuracy: 0.2 %

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### II. Integration of the band area at 630 cm<sup>-1</sup> (590-665 cm<sup>-1</sup>)

detection limit: 0.095 % Vinylacetat  
accuracy: 0.15 %



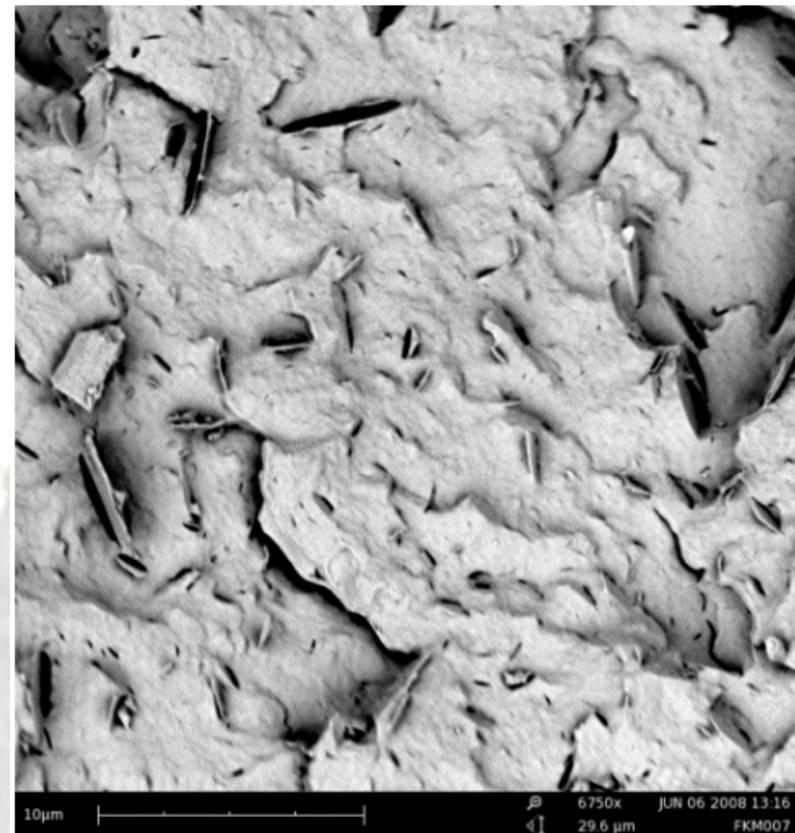
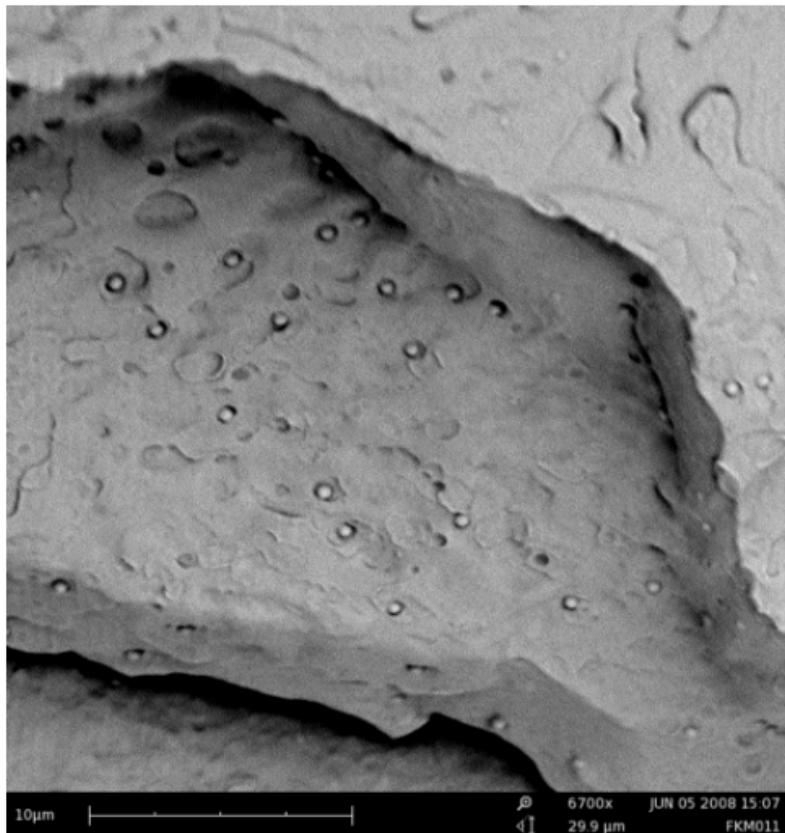
## Off-line determination of particle size by SEM (PA6-NC)

Determination of the particle size for three different SiO<sub>2</sub> and one talcum particles.

SiO<sub>2</sub> -1: 0.25 µm, SiO<sub>2</sub> - 2: 0.60 µm, SiO<sub>2</sub> - 3: 0.08 - 0.2 µm, Talcum: 1.0 - 2.1 µm

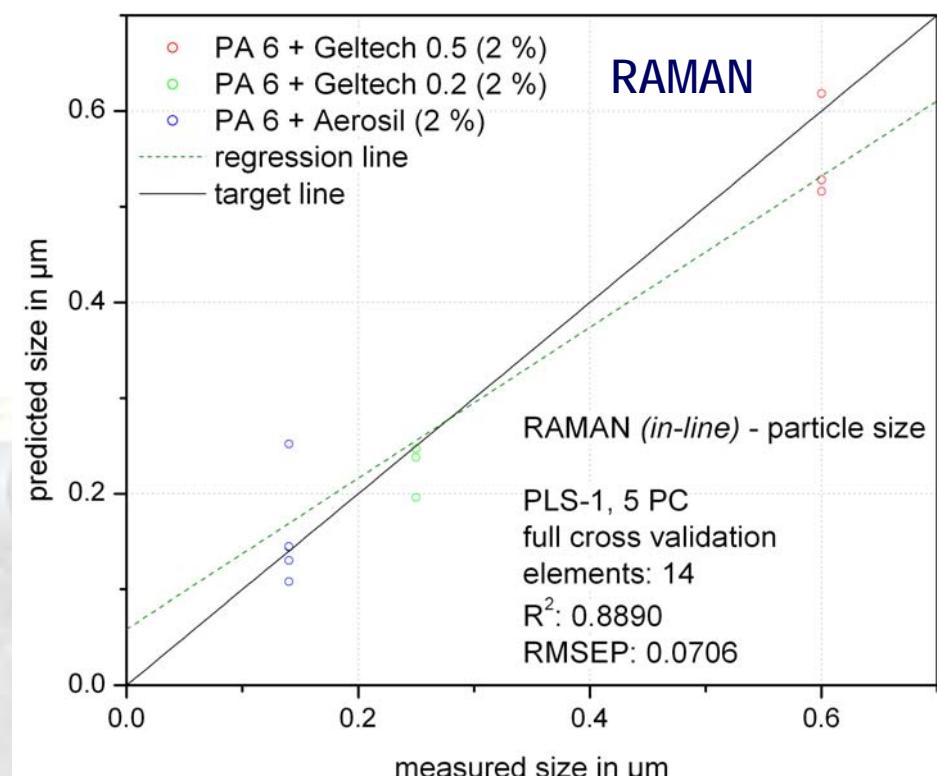
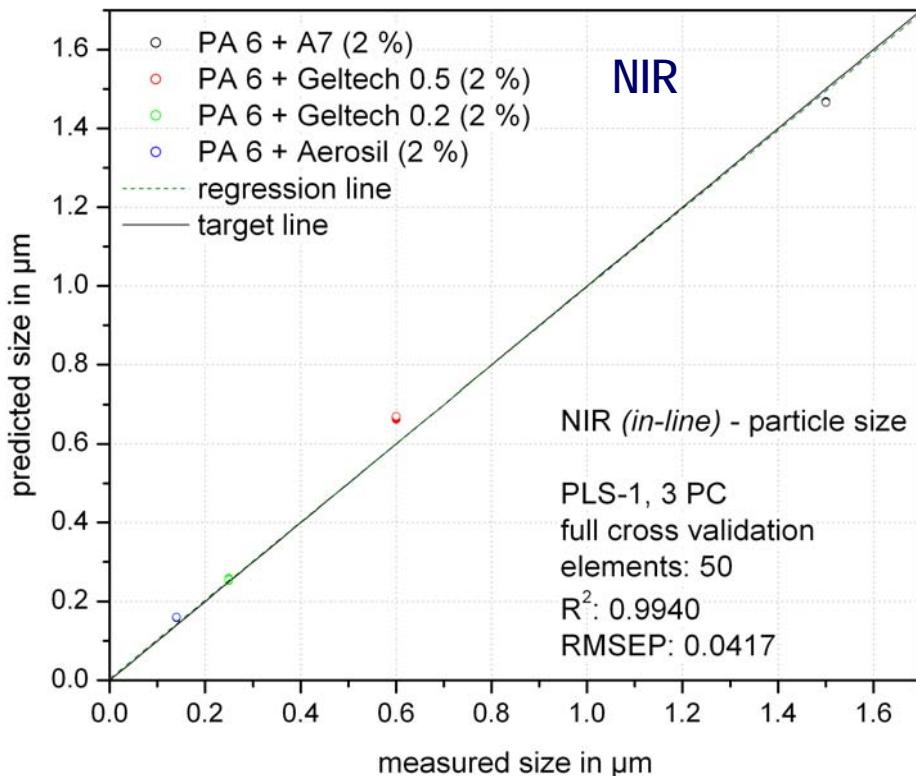
SiO<sub>2</sub> (0.6 µm) / PA6

Talcum / PA 6



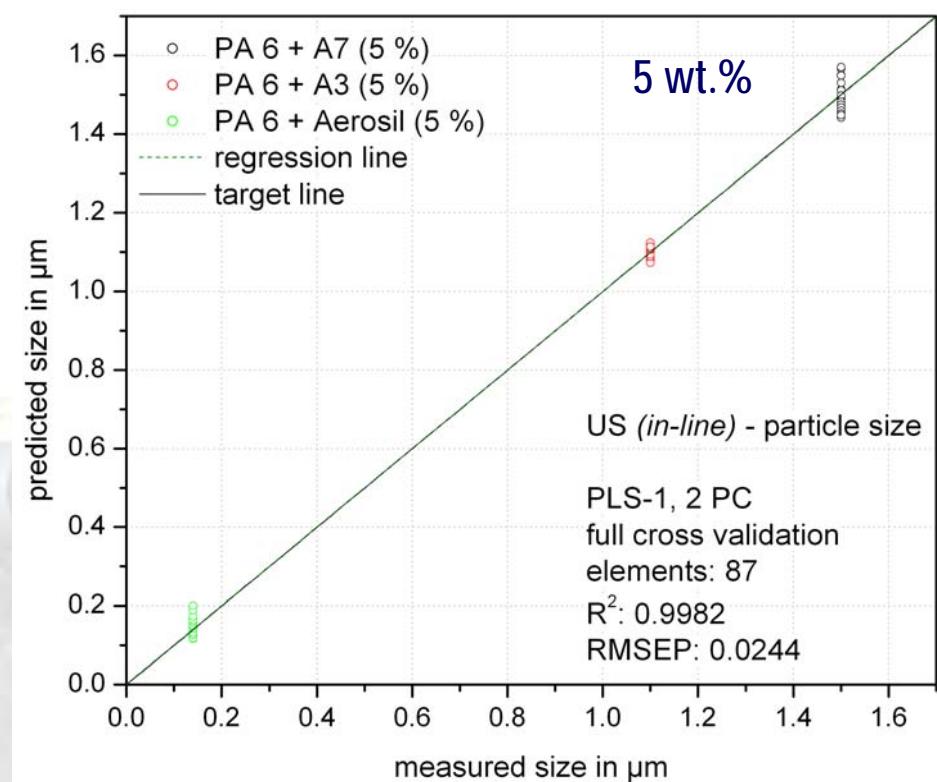
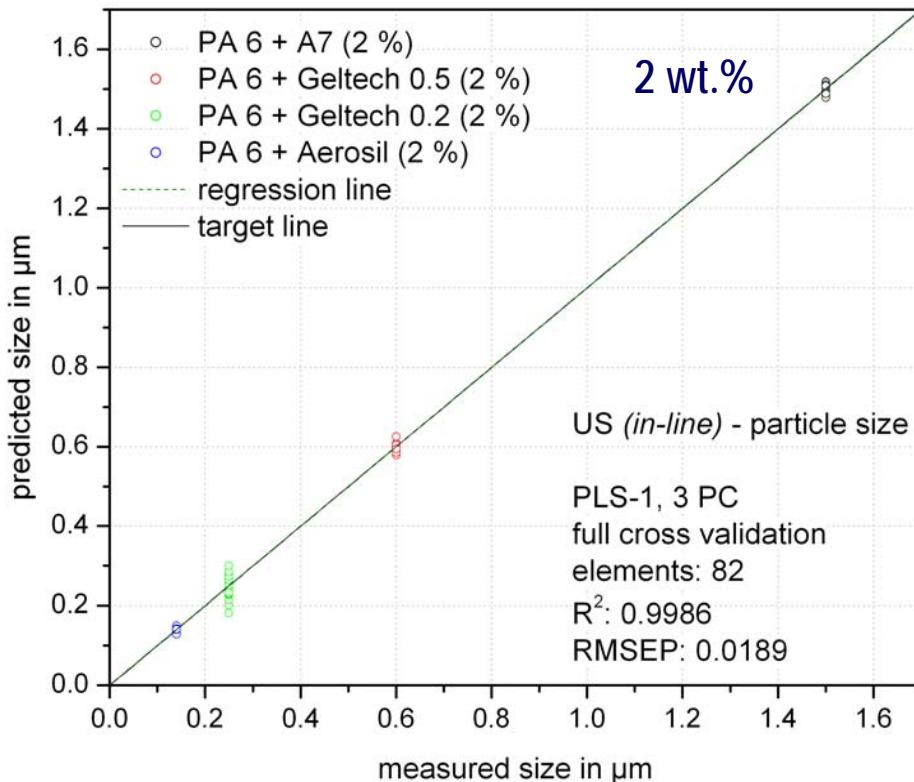
## In-line prediction of particle size by NIR and RAMAN using off-line SEM results

- Determination of the particle size for three different SiO<sub>2</sub> and one talcum particles.  
SiO<sub>2</sub> -1: 0.250 µm, SiO<sub>2</sub> -2: 0.600 µm, SiO<sub>2</sub> -3: 0.08 - 0.2 µm, Talcum: 1.0 - 2.1 µm
- x-axis: measured particle size by SEM
- y-axis: calculated particle size with chemometrics (program UNSCRAMBLER)



## In-line prediction of particle size by Ultrasonic using off-line SEM results

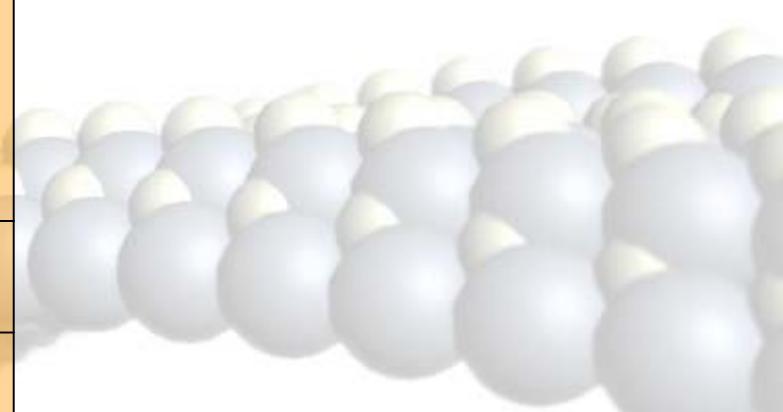
- Determination of the particle size for three different SiO<sub>2</sub>, and talcum particles and two nanofillers.  
SiO<sub>2</sub> -1: 0.250 µm, SiO<sub>2</sub> -2: 0.600 µm, SiO<sub>2</sub> -3: 0.08 - 0.2 µm, Talcum: 1.0 - 2.1 µm
- x-axis: measured particle size by SEM
- y-axis: calculated particle size with chemometrics (program UNSCRAMBLER)



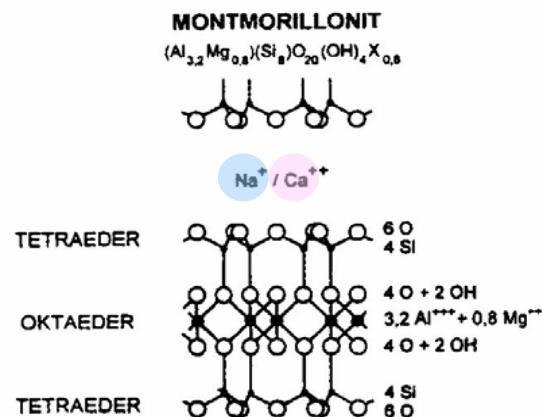
Calculated particle sizes from NIR and Ultrasonic measurements  
Comparison with SEM/TEM data for defined nanoparticles in PA-6

particles (5 wt.%)	av. particle size (SEM) [nm]	calc. particle size (US) [nm] $R^2 = 0.997$
SiO <sub>2</sub> (Aerosil R9200)	140	160 ± 40
Talcum A3	1100	1099 ± 25
Talcum A7	1500	1500 ± 60

particles (2 wt%)	av. particle size (SEM) [nm]	calc. particle size (NIR) [nm] $R^2 = 0.999$	calc. particle size (US) [nm] $R^2 = 0.998$
SiO <sub>2</sub> (Geltech 0.5)	600	665 ± 4	600 ± 30
SiO <sub>2</sub> (Geltech 0.2)	250	256 ± 5	240 ± 60
SiO <sub>2</sub> (Aerosil R9200)	140	160 ± 2	138 ± 12
Talcum A7	1500	1466 ± 4	1500 ± 20

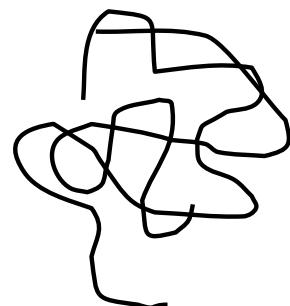


## 2:1 Clay minerals



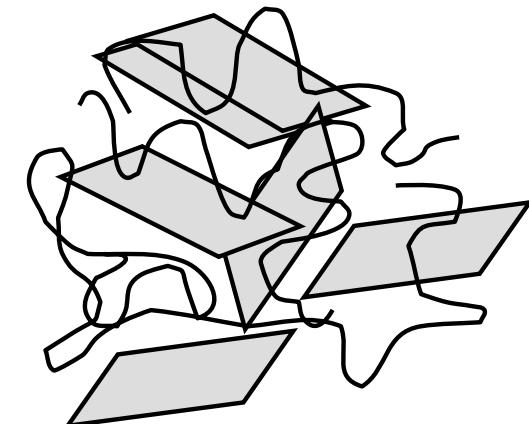
## Polymer

+



## Nanocomposite

exfoliated



dispersion  
degree of intercalation/exfoliation

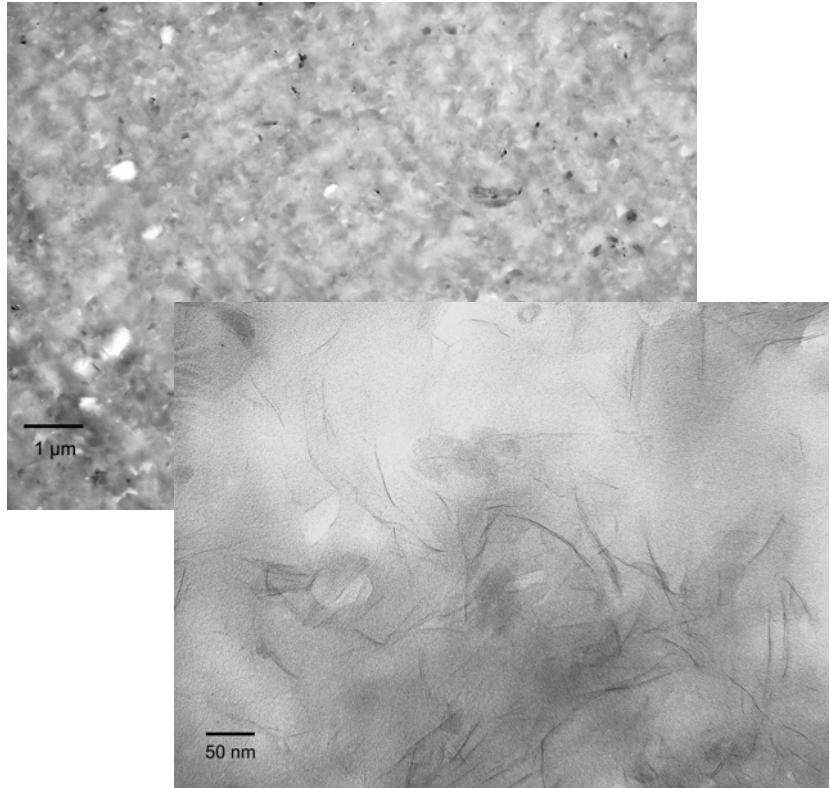
intercalated

Information necessary to control the properties

- content/concentration
- particle size
- dispersion (degree of intercalation/exfoliation) of the nanofiller in the polymer matrix
- mechanical properties

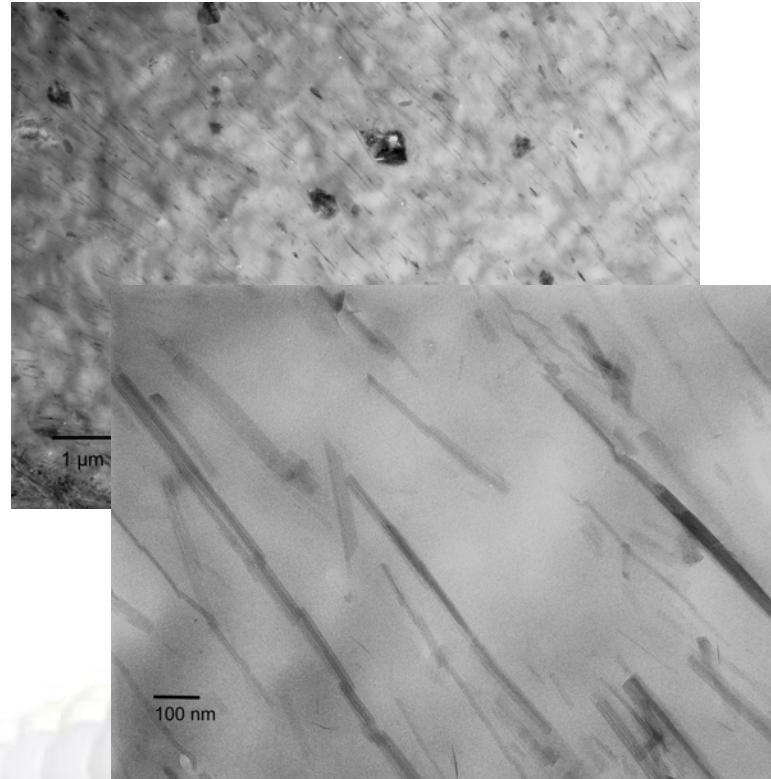
Controlling of the process by real time analysis of these parameters secures the quality of the product and saves time, costs and raw material

PA6 with 7.5 wt% layered silicate  
- montmorillonite (Nanofil)



- nanofiller montmorillonite homogeneous distributed,
- no agglomerates visible,
- layer silicates are exfoliated

PA6 with 5 wt% needle silicate  
- Sepiolite



- nanofiller homogeneous distributed,
- agglomerates visible,
- needle silicates are partially intercalated

Aus Scherviskositätsmessungen wird der Scherverdünnungskoeffizient berechnet. Dieser ist ein Maß für den Grad der Exfolierung der Schichtsilikate im Polymer. Der Grad der Exfolierung dient als Messgröße für die Dispersion.

## Bestimmung des Exfolierungsgrades für 5 PA6 Nanokomposite mit unterschiedlich modifizierten Schichtsilikaten

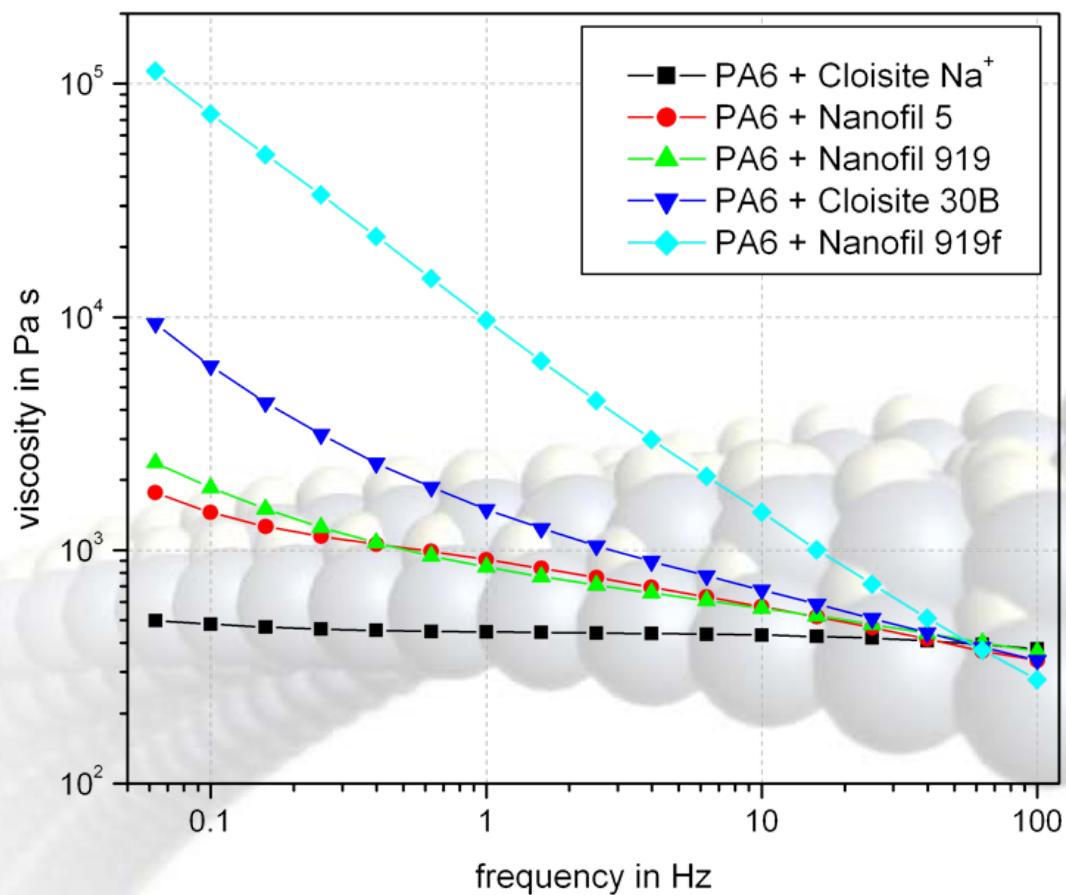
- experiment: ARES rheometer - 240 °C, dynamical frequency sweep (100 Hz ... 0.05 Hz)
- calculation of the shear thinning exponents  $n$  at low frequencies from the shear viscosity  $\eta$
- high shear thinning coefficient: good exfoliation
- low shear thinning coefficient: bad exfoliation

$$\eta \sim \omega^n$$

*n*... Shear thinning exponent

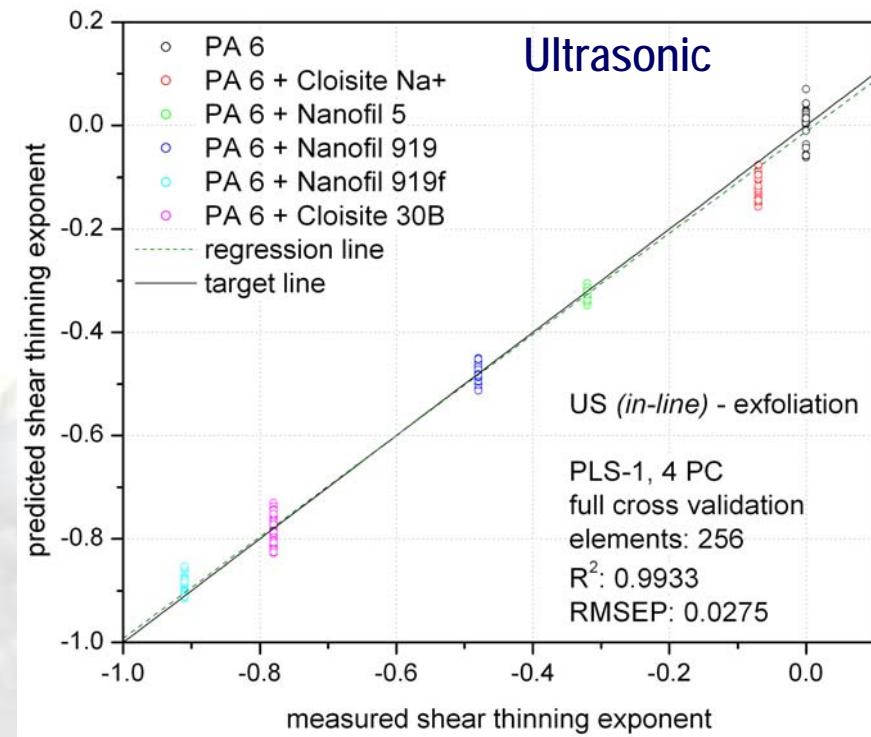
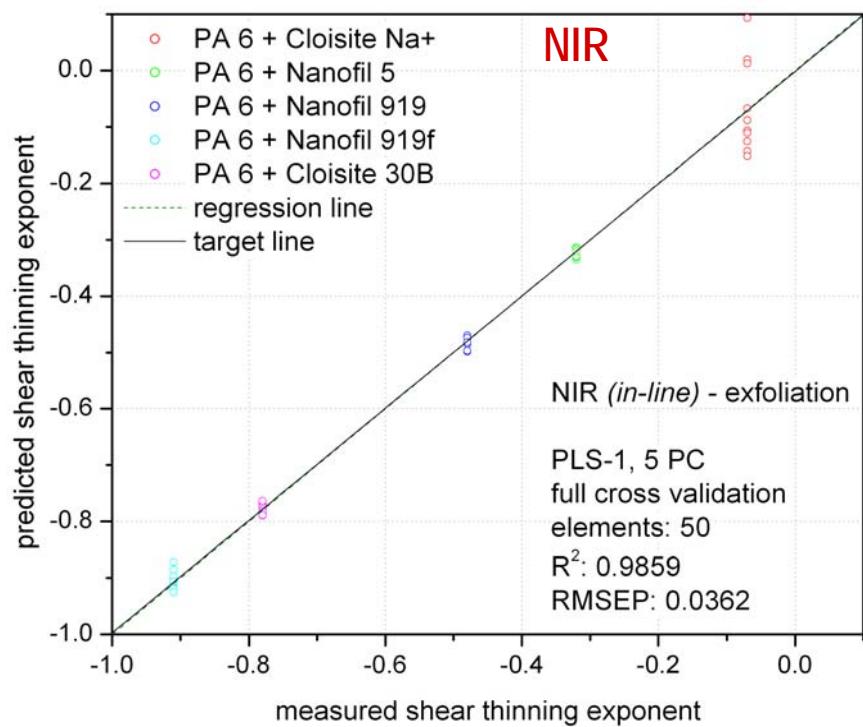
R. Wagener et al., *Polymer* (2003)

nanocomposite	exponent
PA 6 + Cloisite Na+	0.07(2)
PA 6 + Nanofil 5	0.32(3)
PA 6 + Nanofil 919	0.48(4)
PA 6 + Nanofil 919f	0.91(5)
PA 6 + Cloisite 30B	0.78(4)



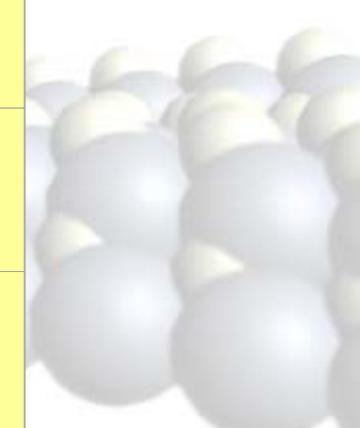
## In-line prediction of shear thinning exponents by **NIR** and Ultrasonic using off-line shear viscosity

- determination of the degree of intercalation/exfoliation
- x-axis: measured shear thinning exponents (ARES)
- y-axis: calculated shear thinning exponents with chemometrics (program UNSCRAMBLER)



- calculated degree of intercalation/exfoliation of 5 different MMT's from **NIR** and **Ultrasonic** data in comparison to calculated shear thinning exponents of rheologic shear thinning measurements
- both methods have a very good correlation to the experimental shear thinning exponent !

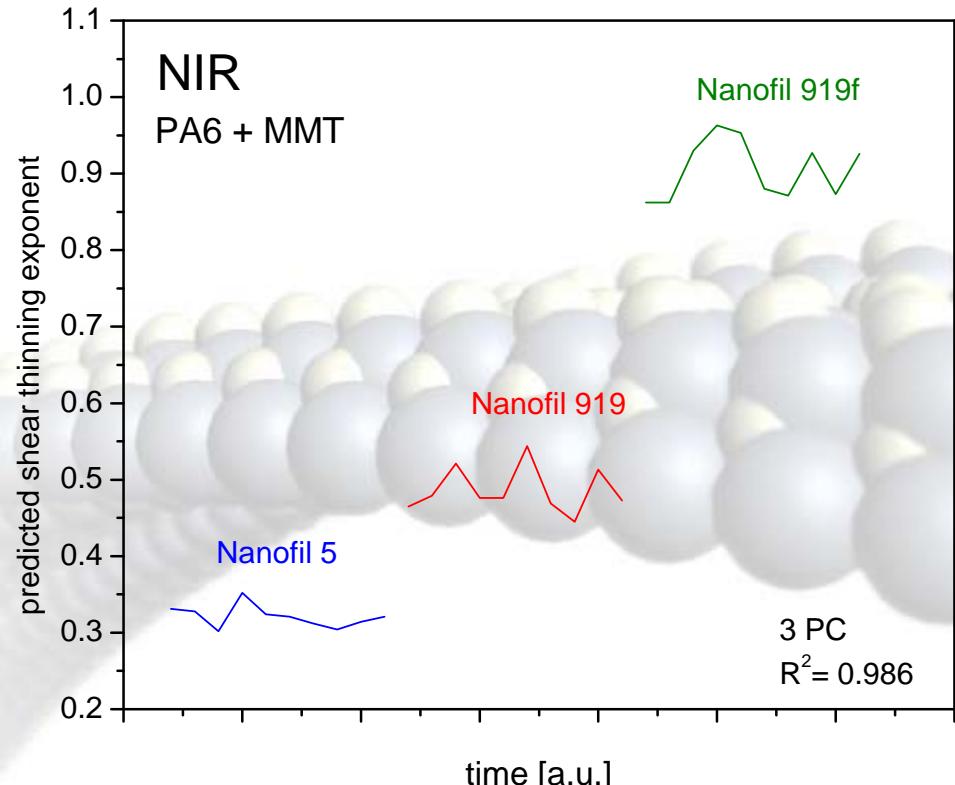
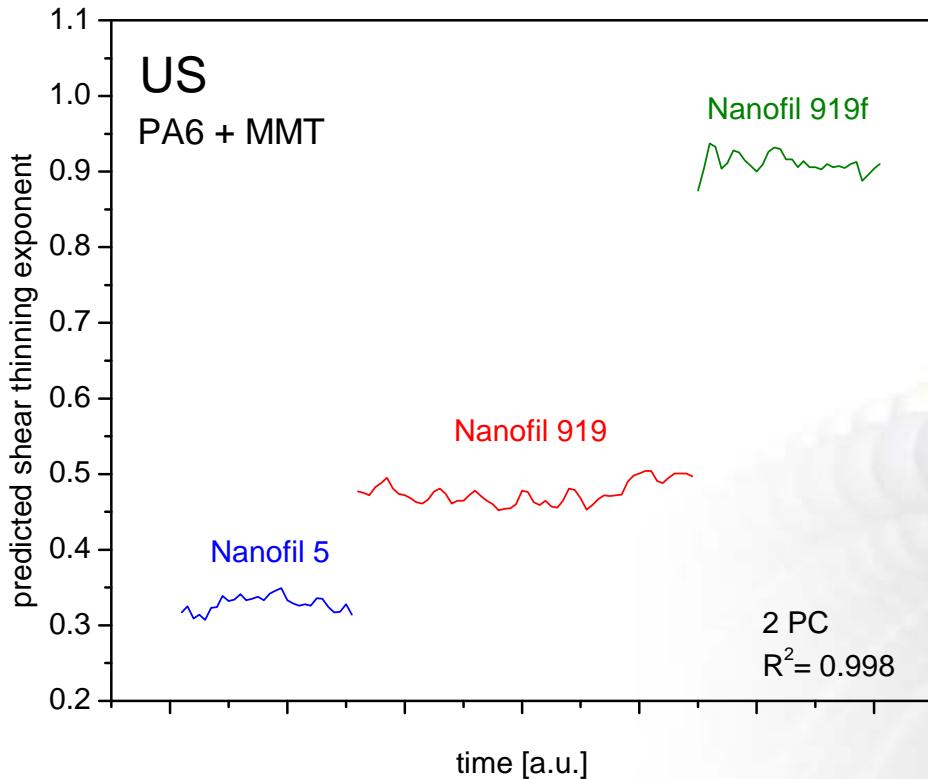
matrix	nanofiller	mass conc.	Meas. shear thinning exp. (ARES)	Calc. shear thinning exp. <b>NIR</b>	Calc. shear thinning exp. <b>US</b>
PA6	<b>"Nanofil 919 f"</b> <i>chem. modified MMT</i>	5%	-0.91	<b>-0.87 – -0.93</b>	<b>-0.85 – -0.92</b>
PA6	<b>"Nanofil 919"</b> <i>chem. modified MMT</i>	5%	-0.48	<b>-0.47 – -0.50</b>	<b>-0.45 – -0.51</b>
PA6	<b>"Nanofil 5"</b> <i>chem. modified MMT</i>	5%	-0.32	<b>-0.31 – -0.34</b>	<b>-0.30 – -0.35</b>
PA6	<b>"Cloisite 30 B"</b> <i>chem. modified MMT</i>	5%	-0.78	<b>-0.76 – -0.79</b>	<b>-0.73 – -0.83</b>
PA6	<b>"Cloisite Na+"</b> <i>unmodified MMT</i>	5%	-0.07	<b>+0.09 – -0.15</b>	<b>-0.07 – -0.16</b>



## Monitoring of shear thinning exponents by NIR and Ultrasonic measurements

Best exfoliation behavior (highest STE) for Nanofil919f, good exfoliation for Nanofil919, lowest exfoliation for Nanofil5 in PA6

- Determination of the degree of intercalation/exfoliation for PA6 with different MMT
- x-axis: time
- y-axis: predicted shear thinning exponents calculated by multivariate data analysis using in-line measurements

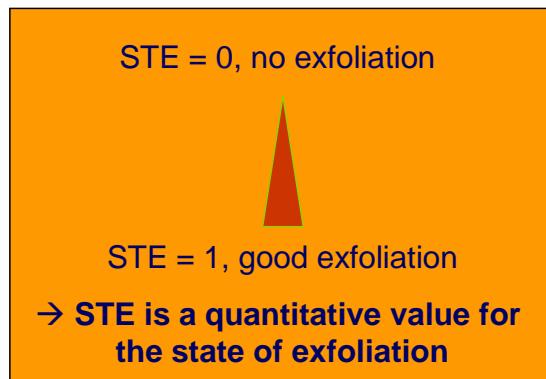


## Determination of the degree of intercalation/exfoliation of mPP/C20A with 3 different screw speeds

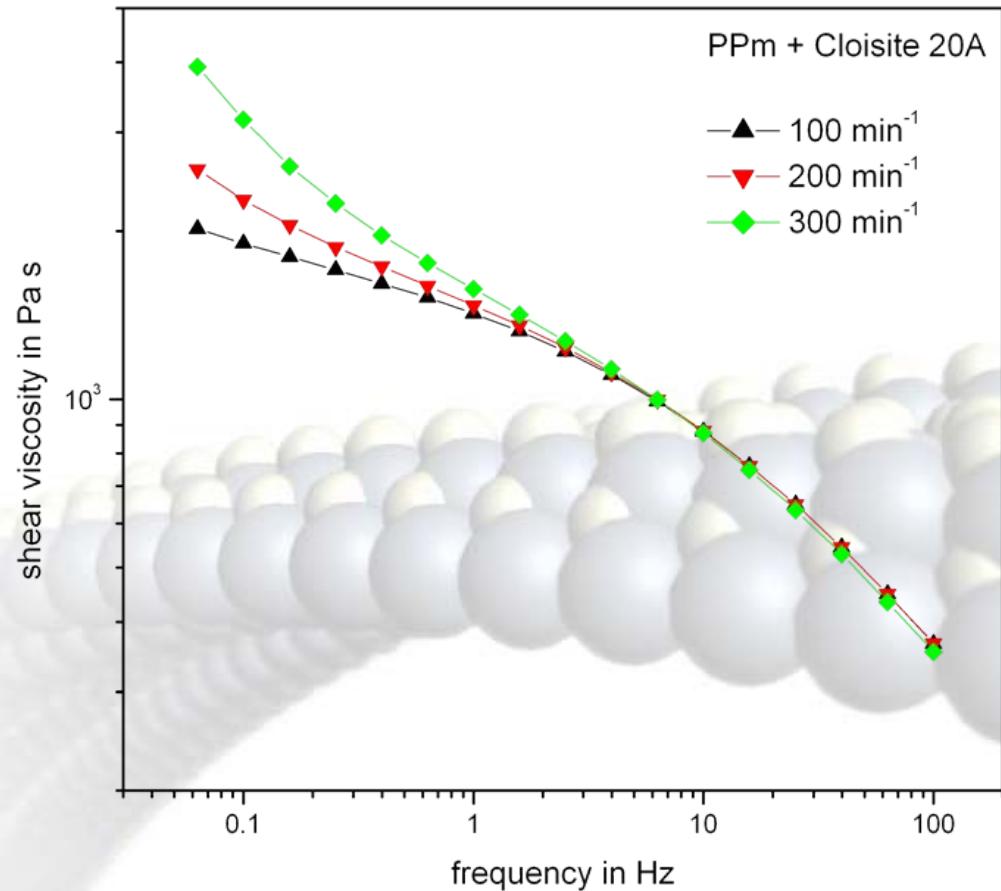
- Experiment: ARES rheometer - 220 °C, dynamical frequency sweep (100 Hz ... 0.05 Hz)
- Calculation of the shear thinning exponents  $n$  at low frequencies from the shear viscosity  $\eta$   
(precondition: same nanofiller content, 5 wt.% inorganic content)

$$\eta \sim \omega^n$$

n... Shear thinning exponent (STE)  
 $\eta$ ... shear viscosity  
 $\omega$ ... frequency

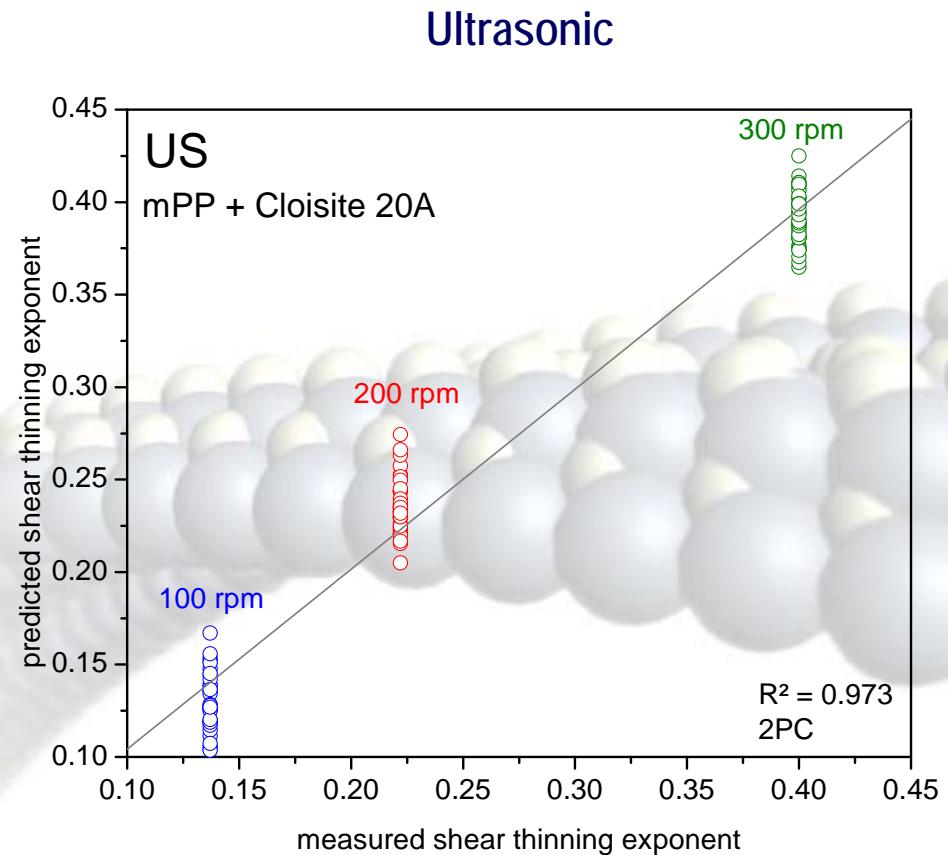
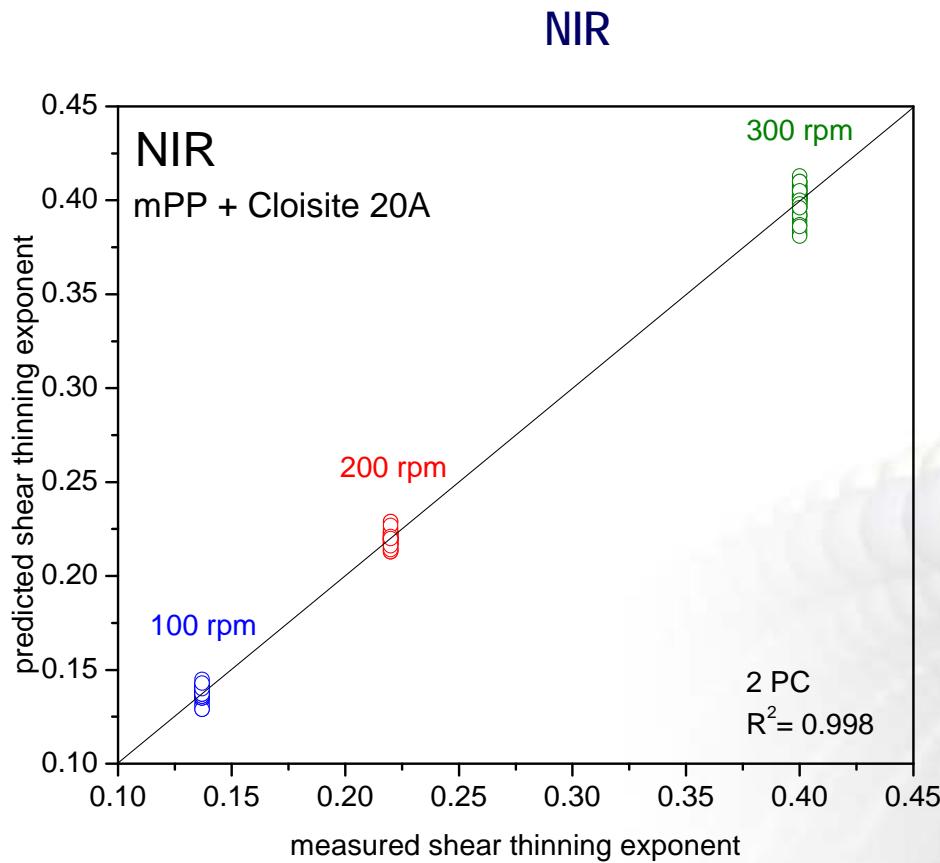


nanocomposite	exponent
PPm + Cloisite 20A (100 rpm)	$0.14 \pm 0.02$
PPm + Cloisite 20A (200 rpm)	$0.22 \pm 0.03$
PPm + Cloisite 20A (300 rpm)	$0.40 \pm 0.05$



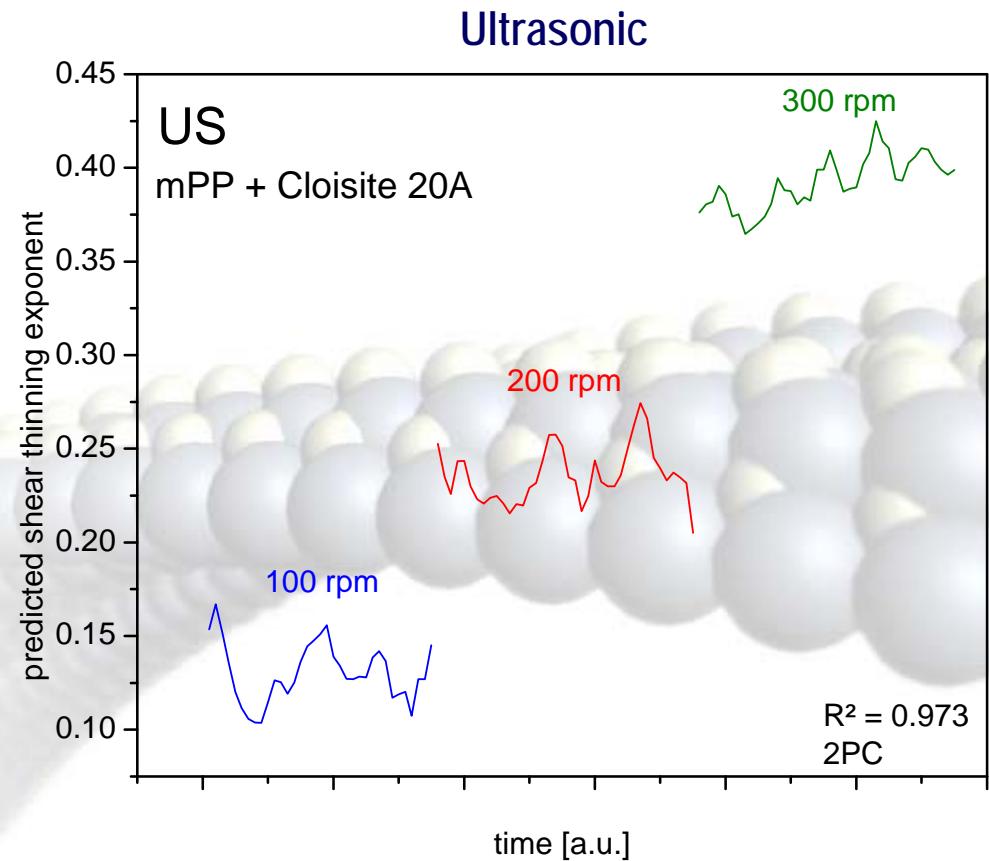
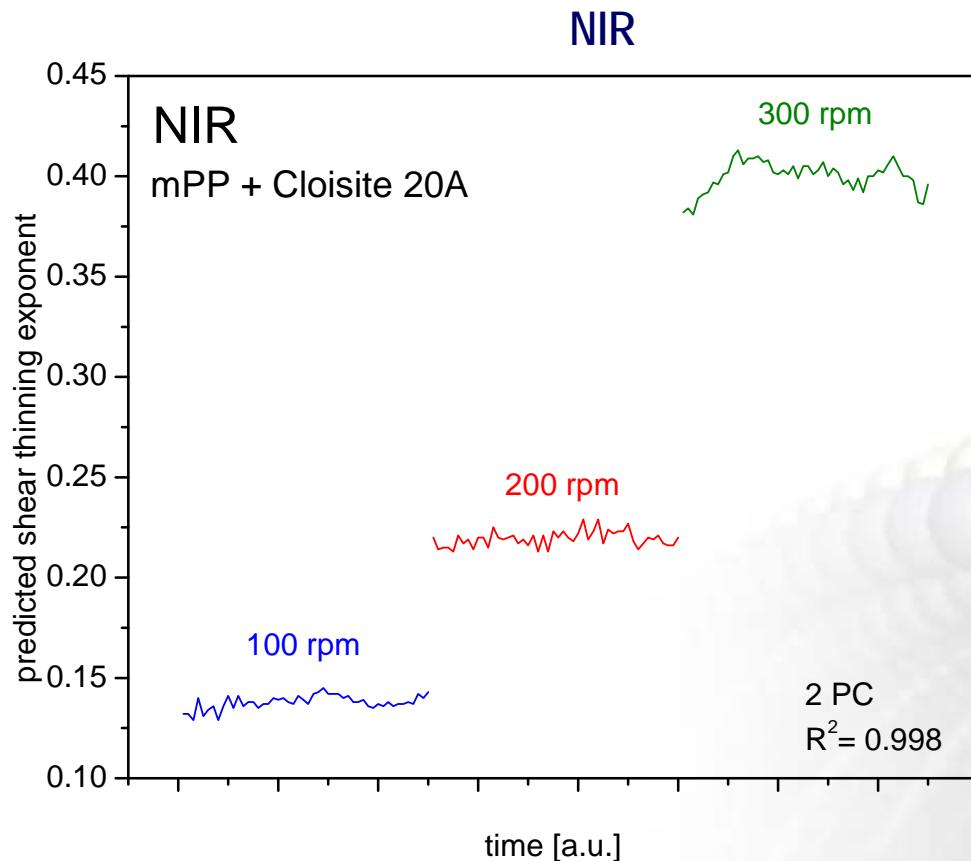
## Prediction of shear thinning exponents by NIR and Ultrasonic using off-line shear thinning exponents Increasing degree of exfoliation at increasing screw speeds

- Determination of the degree of intercalation/exfoliation for modified PP with MMT C20A at different screw speeds
- x-axis: off-line measured shear thinning exponents (ARES)
- y-axis: calculated shear thinning exponents by multivariate data analysis using in-line measurements



Monitoring of shear thinning exponents by NIR and Ultrasonic  
Increasing degree of exfoliation at increasing screw speeds

- Determination of the degree of intercalation/exfoliation for modified PP with MMT C20A at different screw speeds
- x-axis: time
- y-axis: predicted shear thinning exponents calculated by multivariate data analysis using in-line measurements



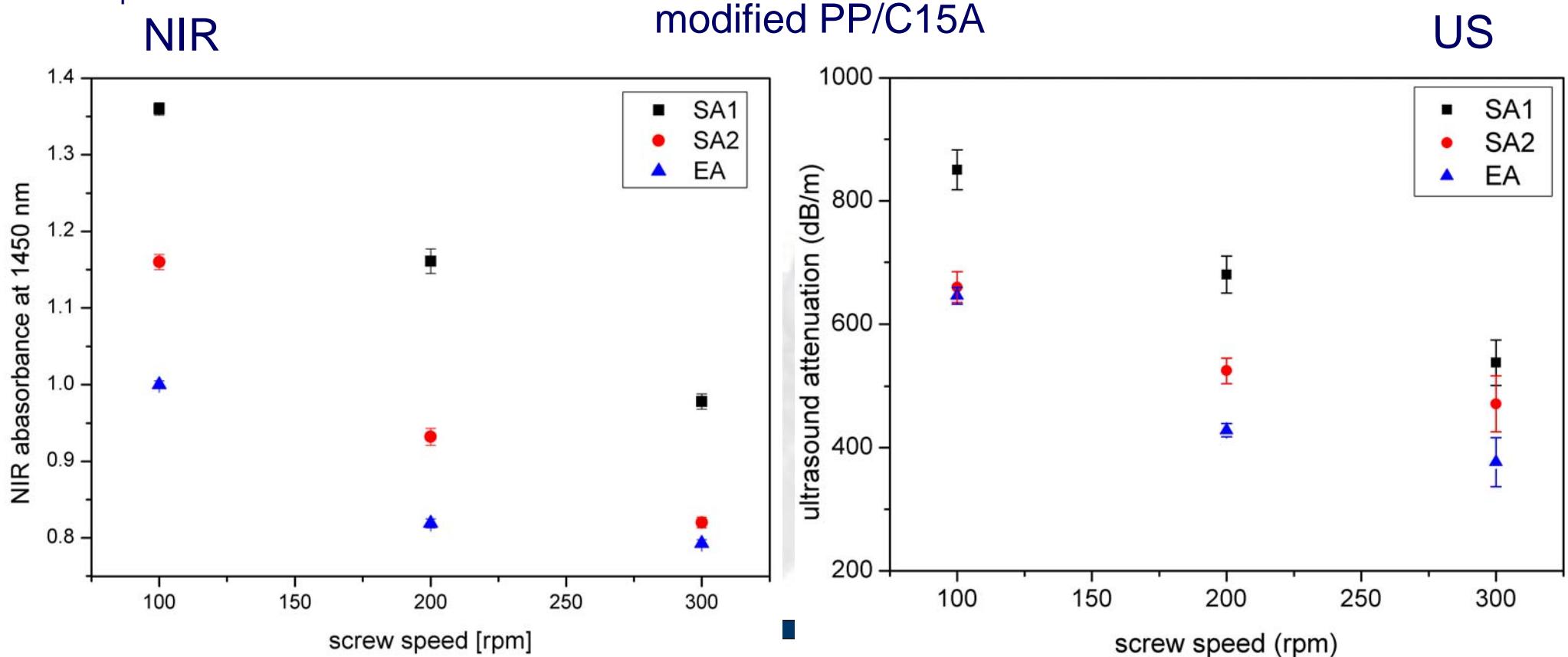
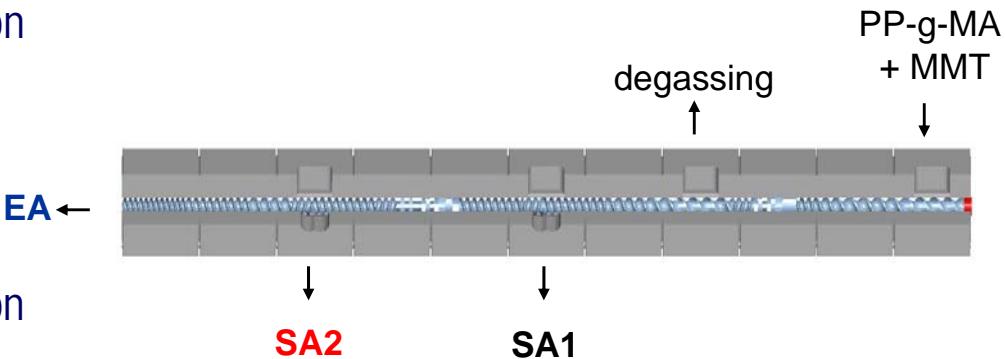
# Untersuchung des Dispersionsprozesses von PP Nanocompositen entlang des Extruders mit NIR und Ultraschallmessungen

- decreasing of NIR- absorption and Ultrasound attenuation along the extruder (SA1, SA2, EA)

→ improvement of the exfoliation state

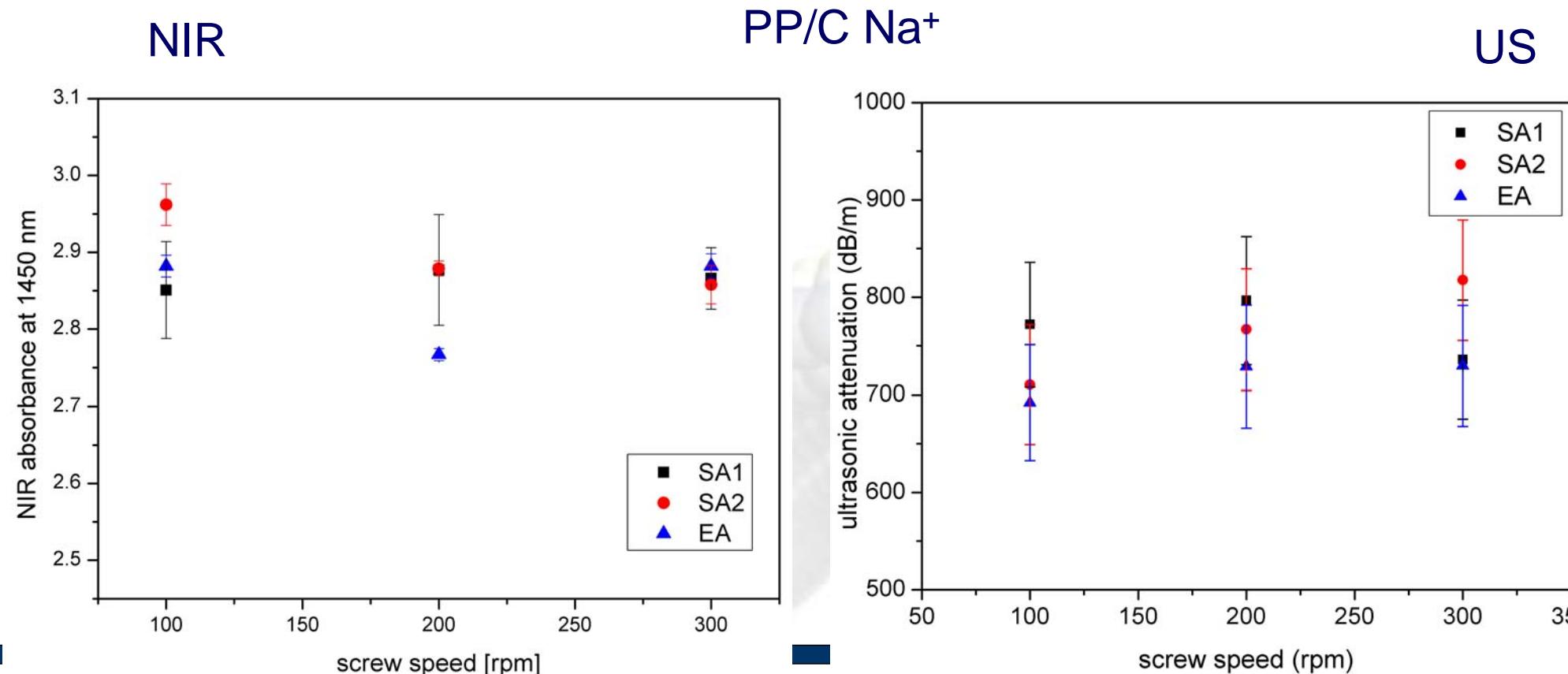
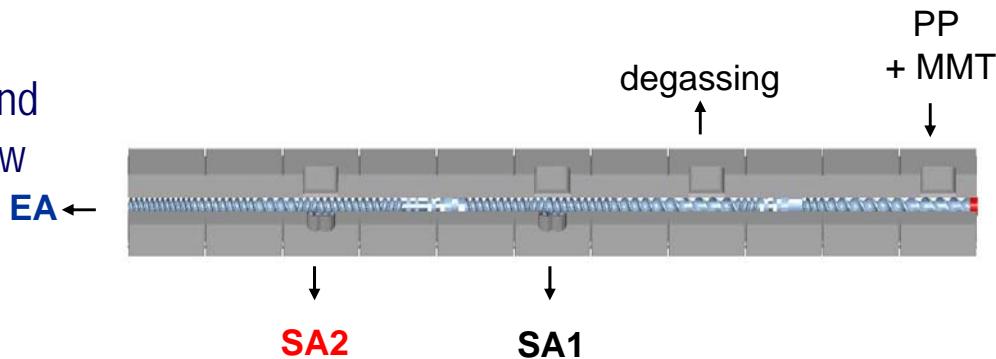
- decreasing of NIR- absorption and Ultrasound attenuation with increasing screw speed (100 → 300 rpm)

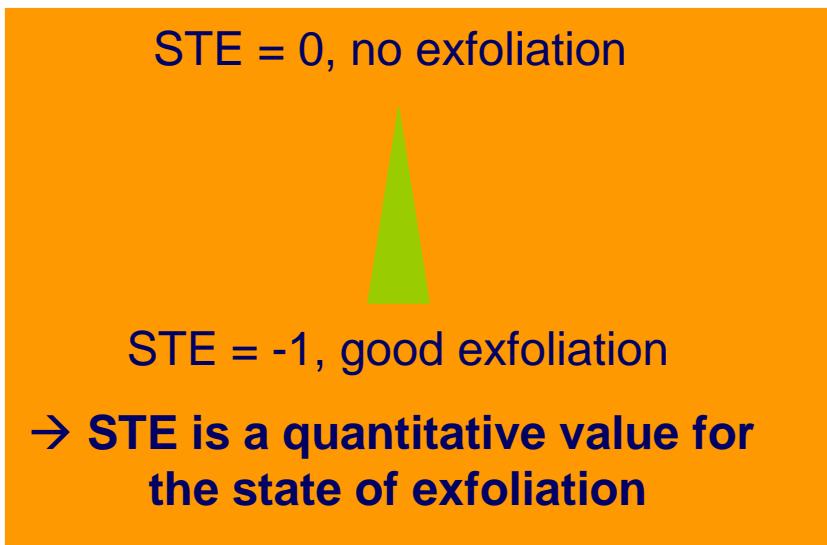
→ improvement of the exfoliation state



# Untersuchung des Dispersionsprozesses von PP Nanocompositen entlang des Extruders mit NIR und Ultraschallmessungen

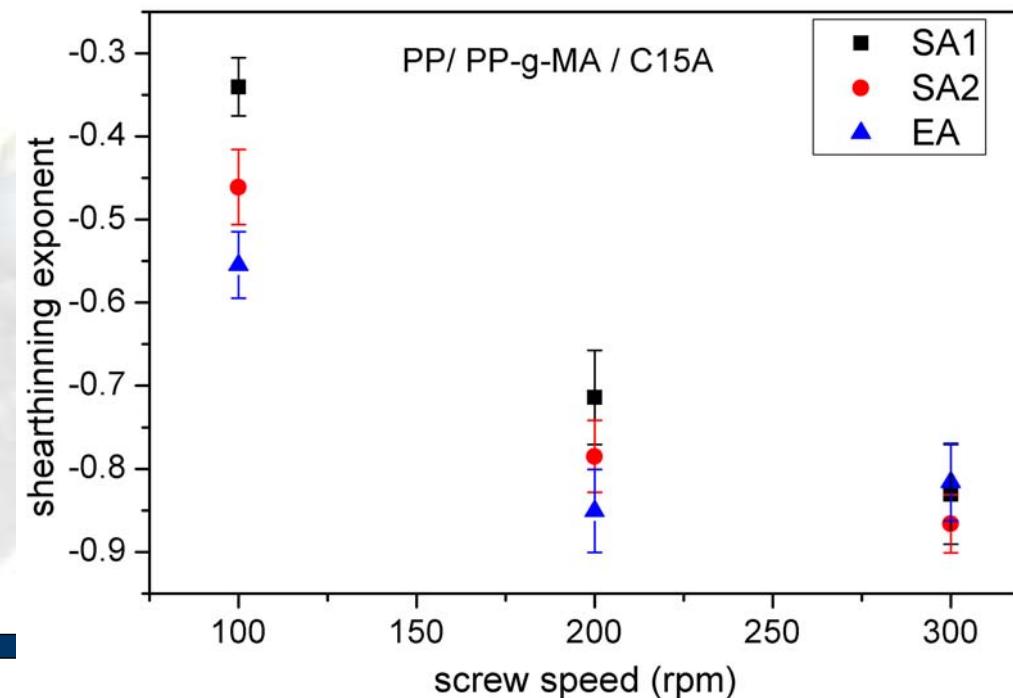
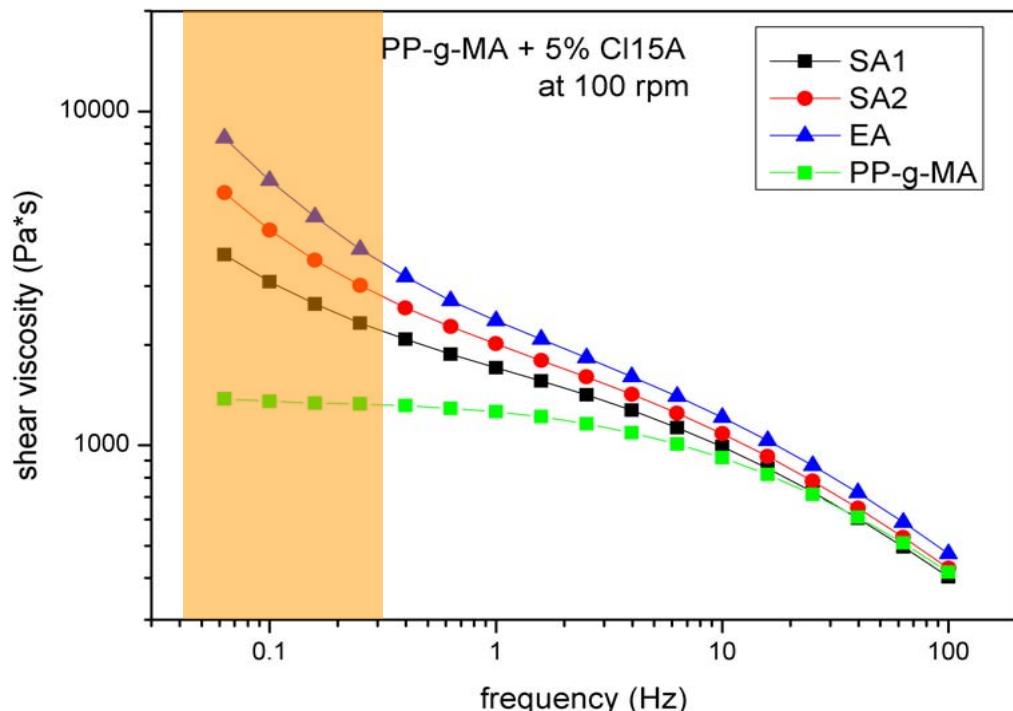
- no significant changes of NIR- absorption and Ultrasound attenuation along the extruder and with increasing screw speed
- no exfoliation of the layered silicates



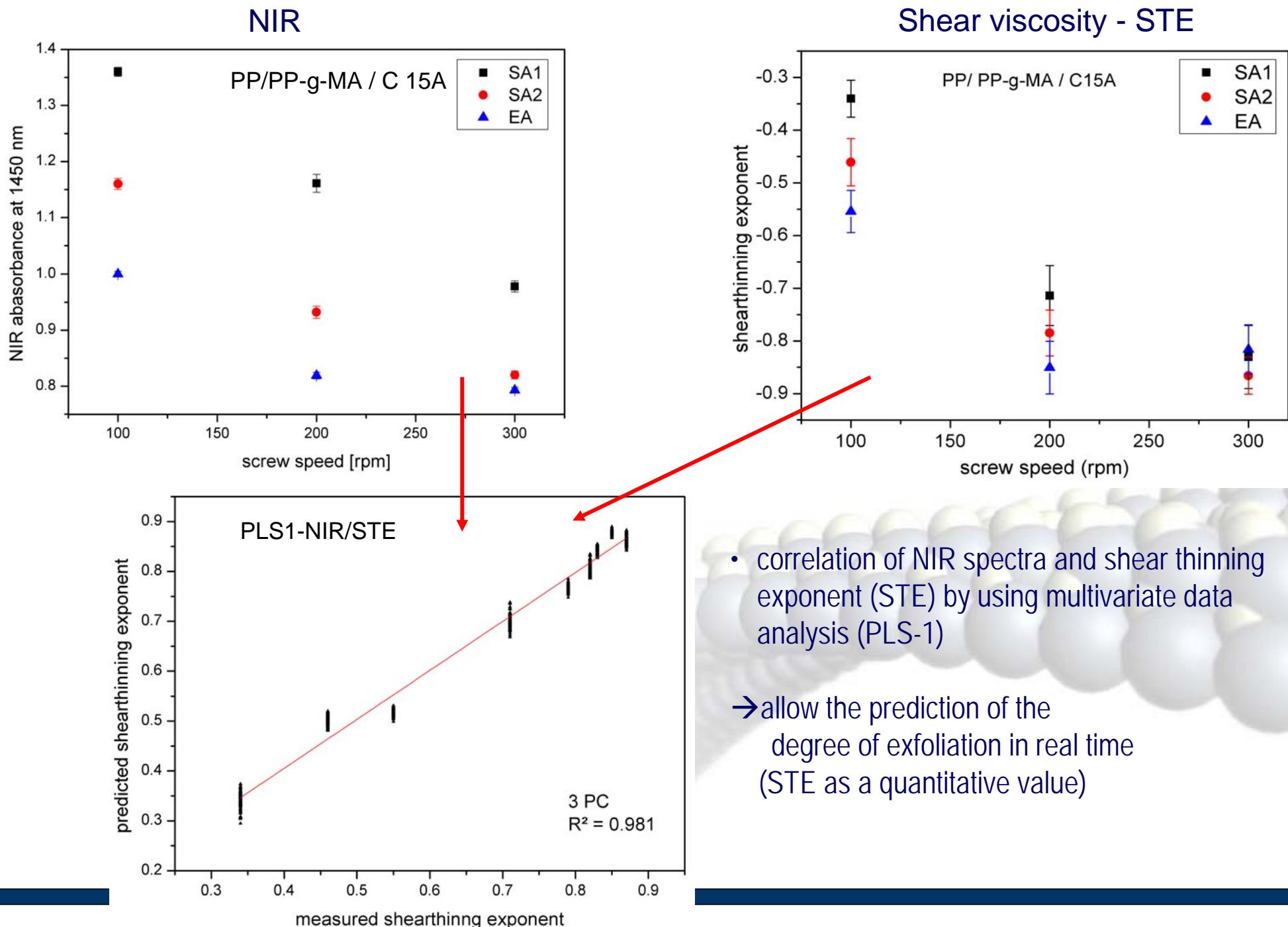


- increasing of the shear thinning exponent along the extruder and with increasing of the screw speed

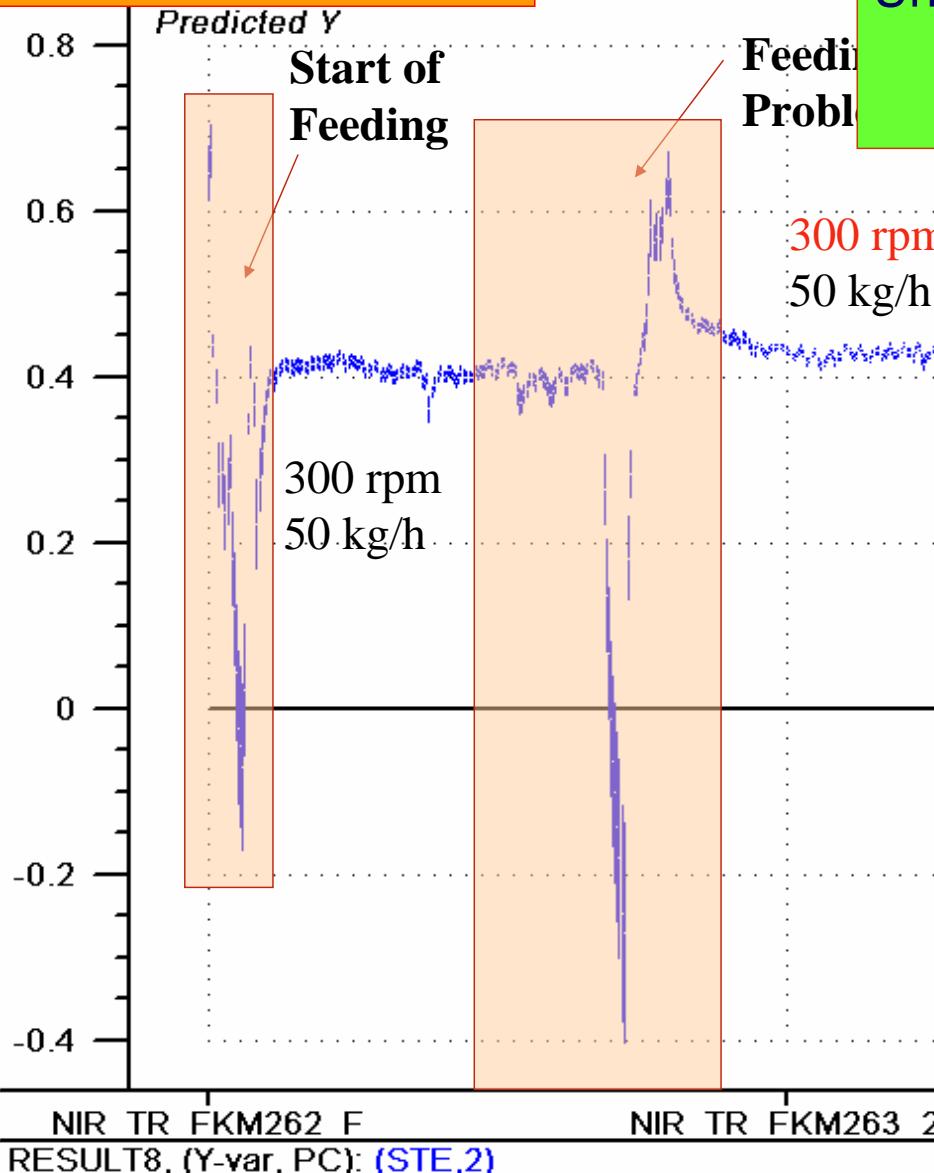
→ improvement of the exfoliation state



# Korrelation der in-line NIR Spektren und der Scherverdünnungskoeffizienten



**shear thinning exponent**



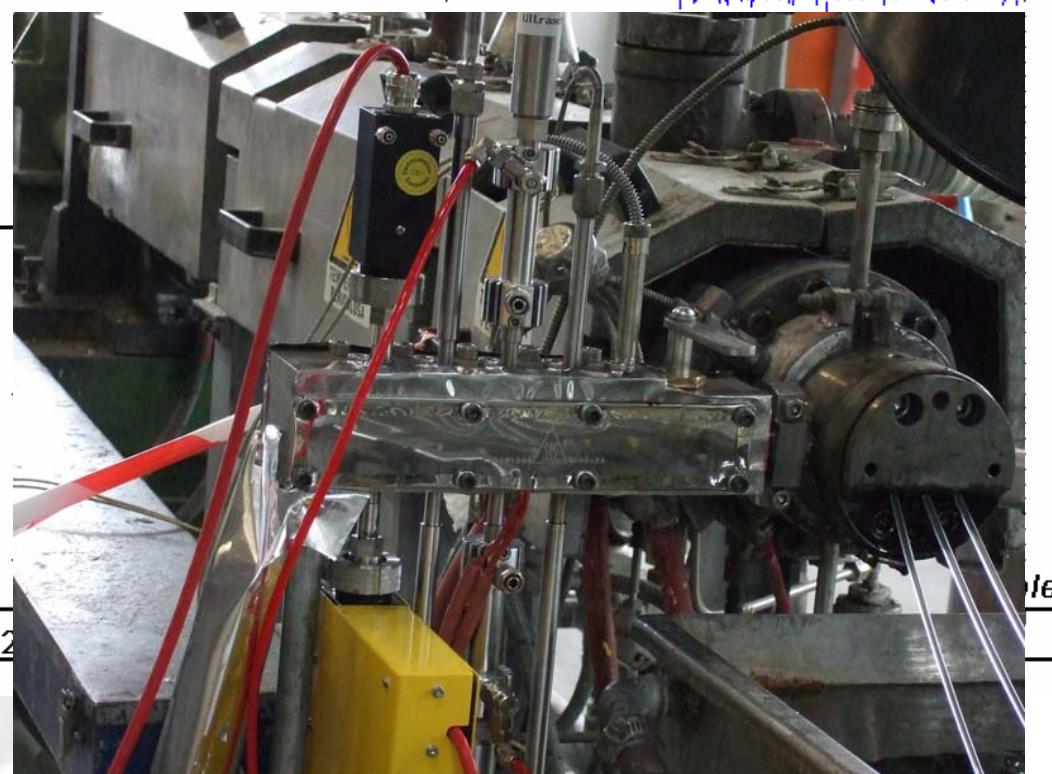
Unterschiedliche Drehzahl

Temperatur konstant  
konstanter Durchsatz

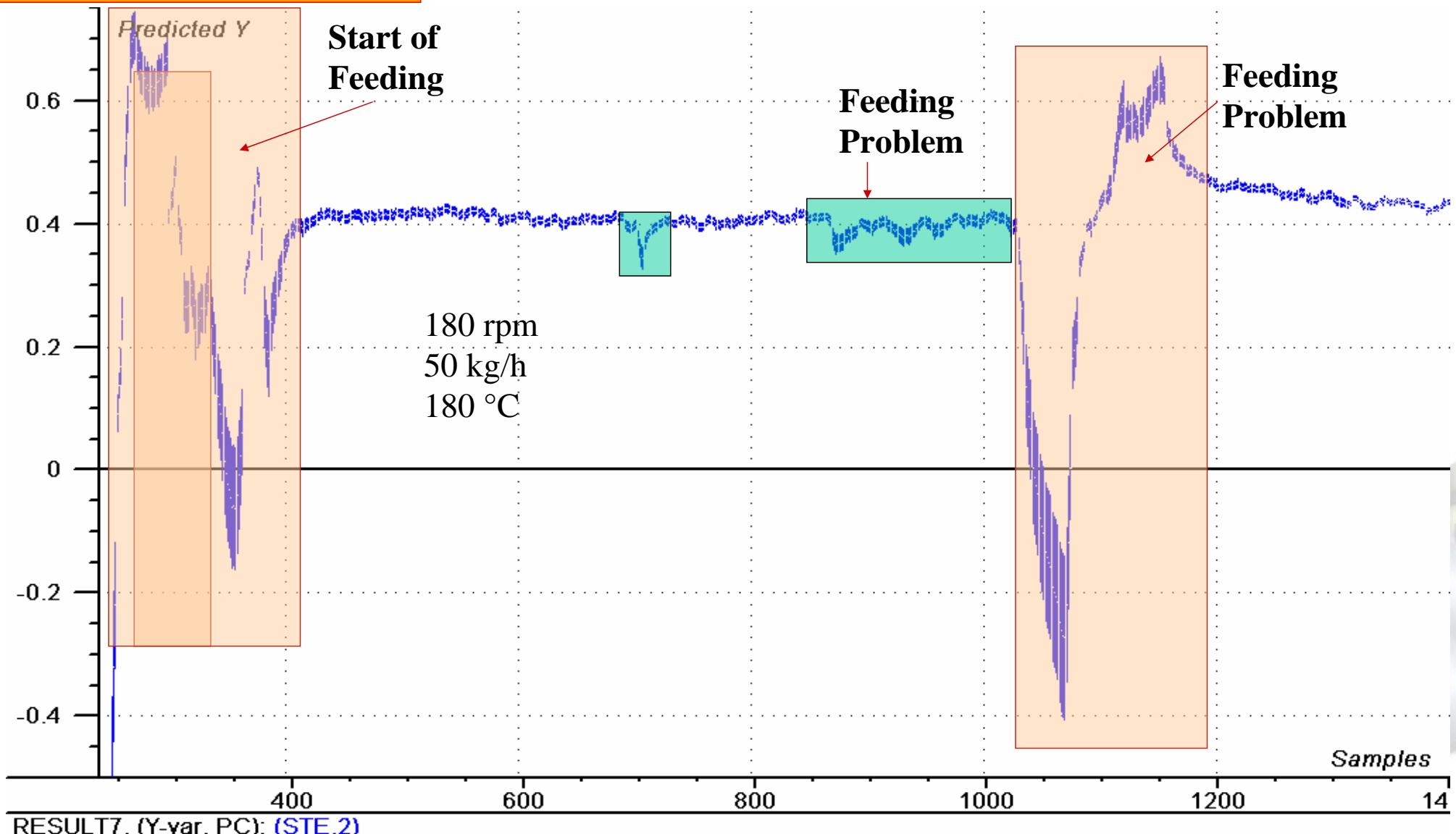
300 rpm  
50 kg/h

230 rpm  
50 kg/h

180 rpm  
50 kg/h

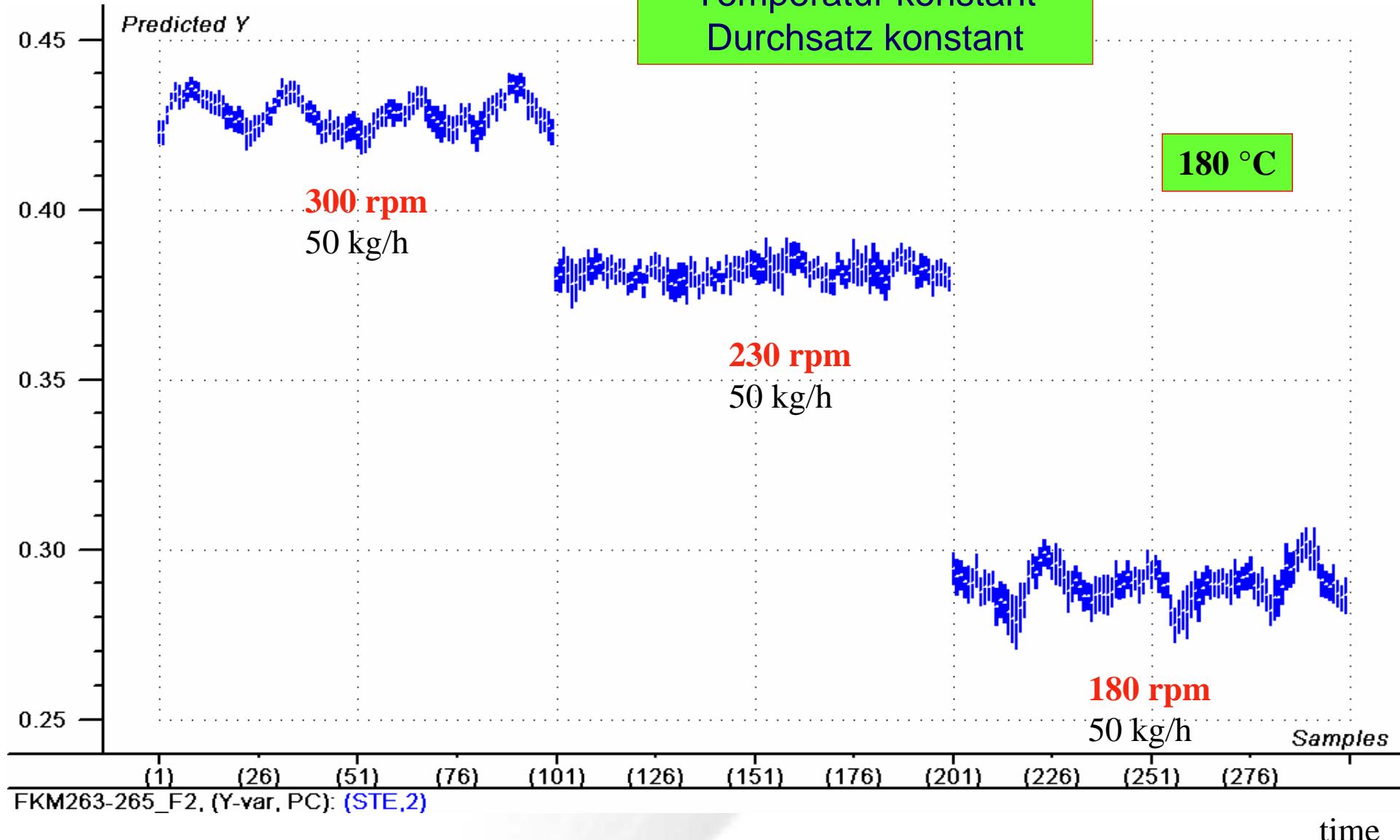


## shear thinning exponent

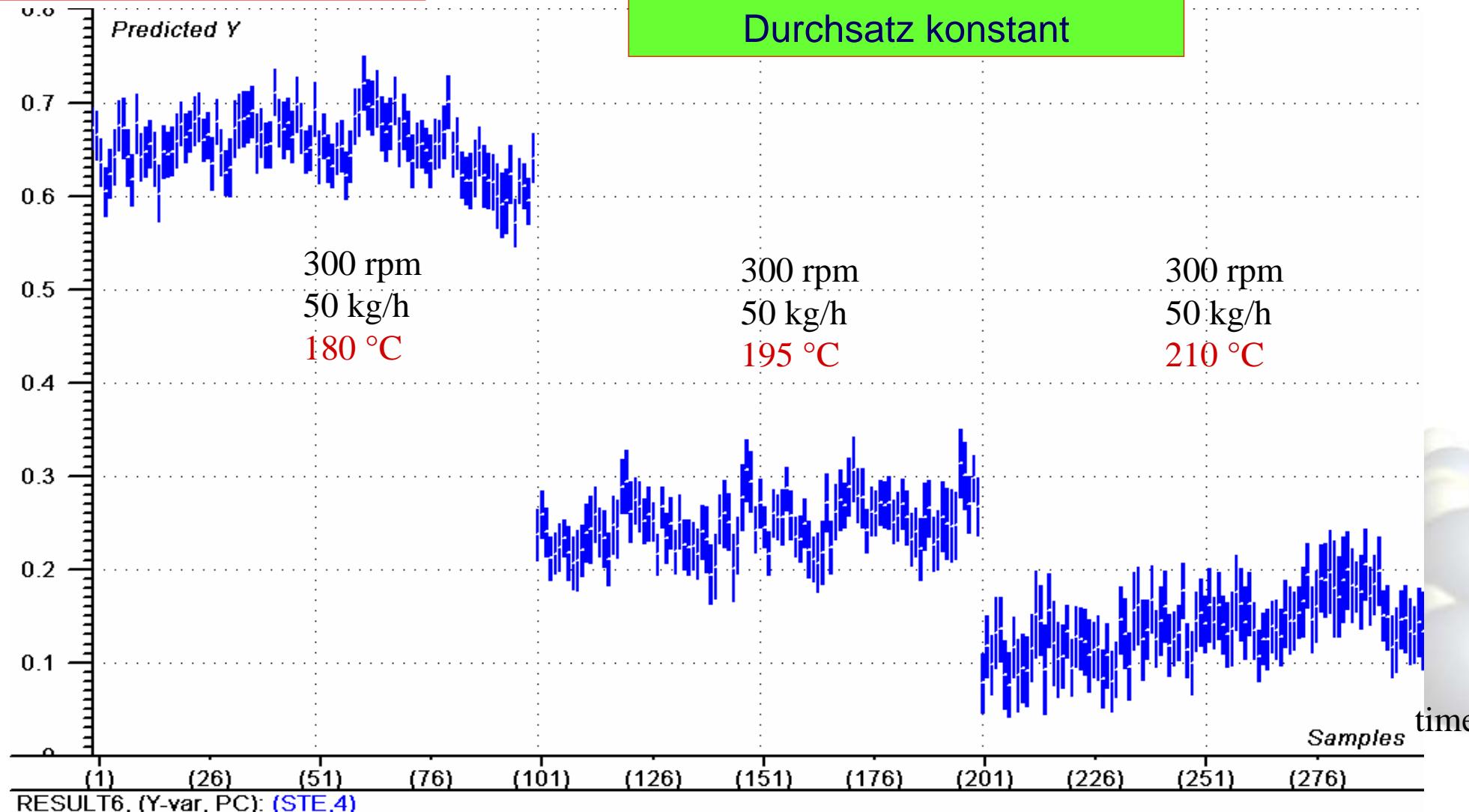


**shear thinning exponent**

Unterschiedliche Drehzahl  
Temperatur konstant  
Durchsatz konstant

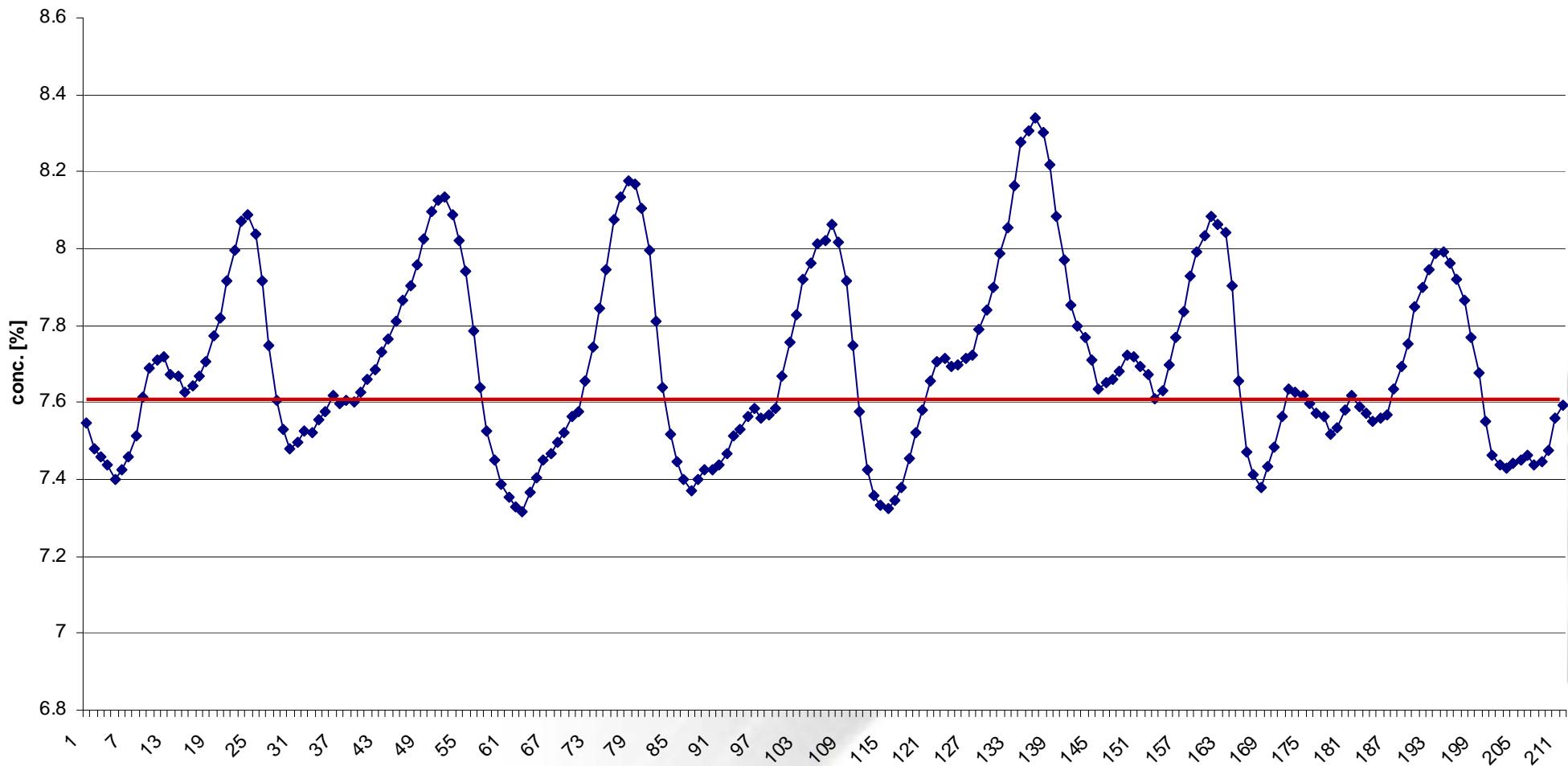


**shear thinning exponent**

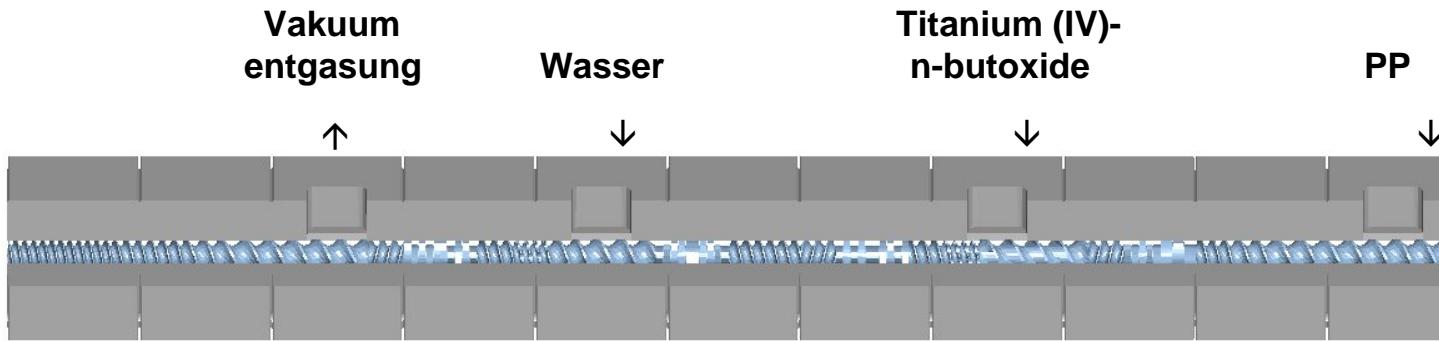
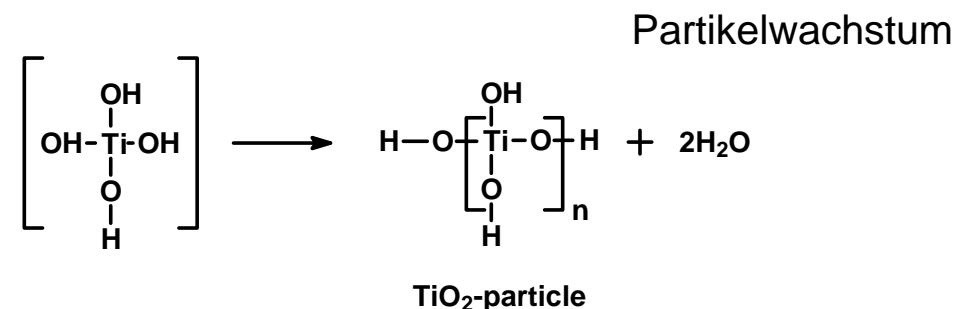
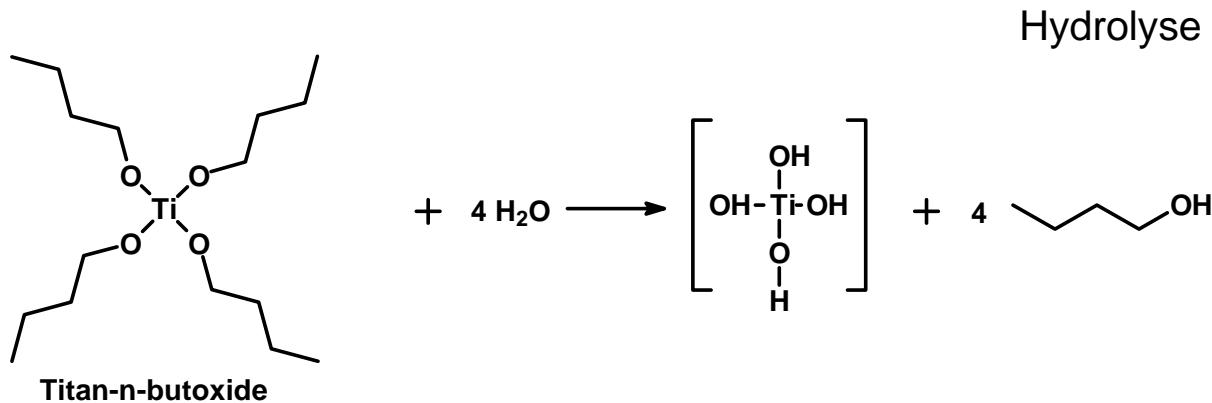


Nachweis periodischer Fluktuationen (Kalibrierung in Konzentrationseinheiten)  
im Extrusionsprozess, verursacht durch instabile Dosierung

predicted concentration C20A

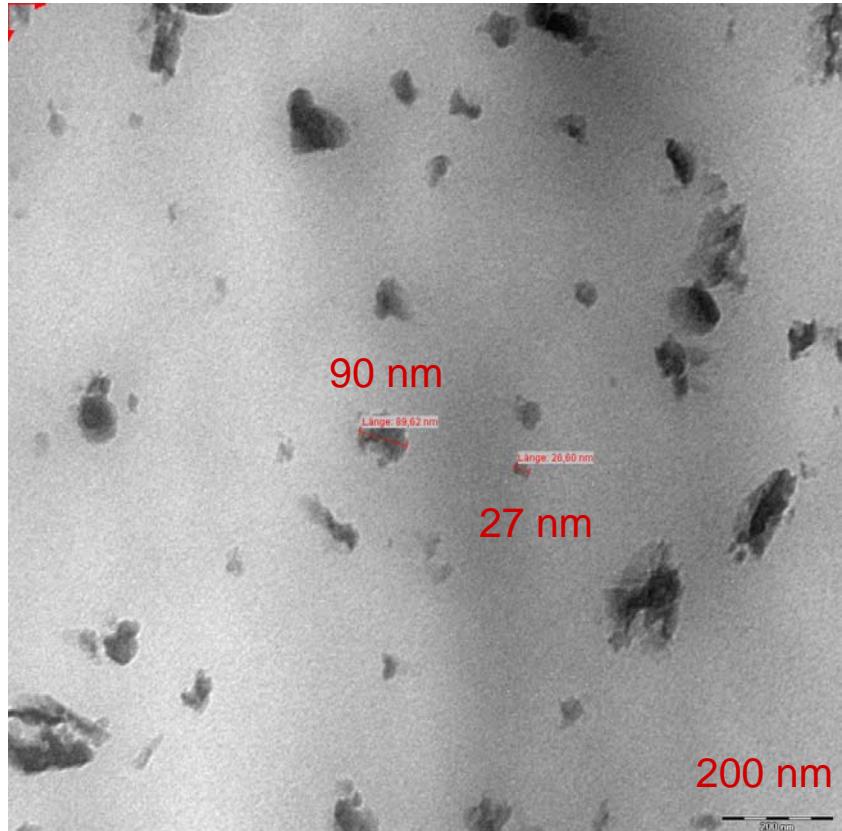


# Monitoring der Reaktiv-Extrusion zur in-situ Generierung von nanoskaligen TiO<sub>2</sub>-Partikeln in PP

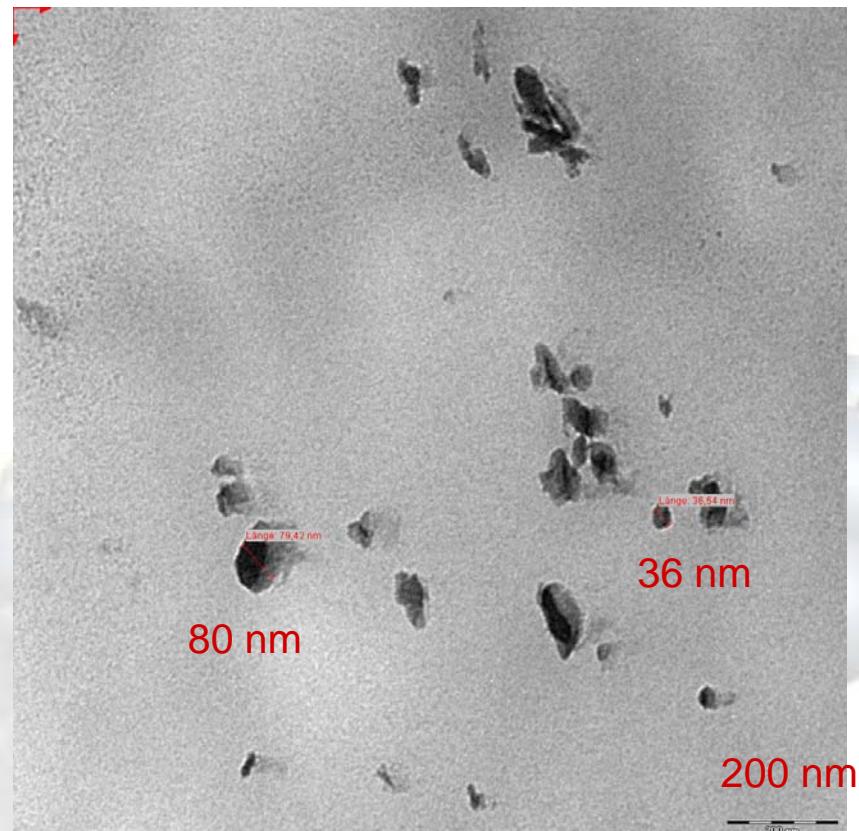


Determination of particle size of  $\text{TiO}_2$  with different water content during extrusion  
Scanning Electron Microscopy (SEM) images

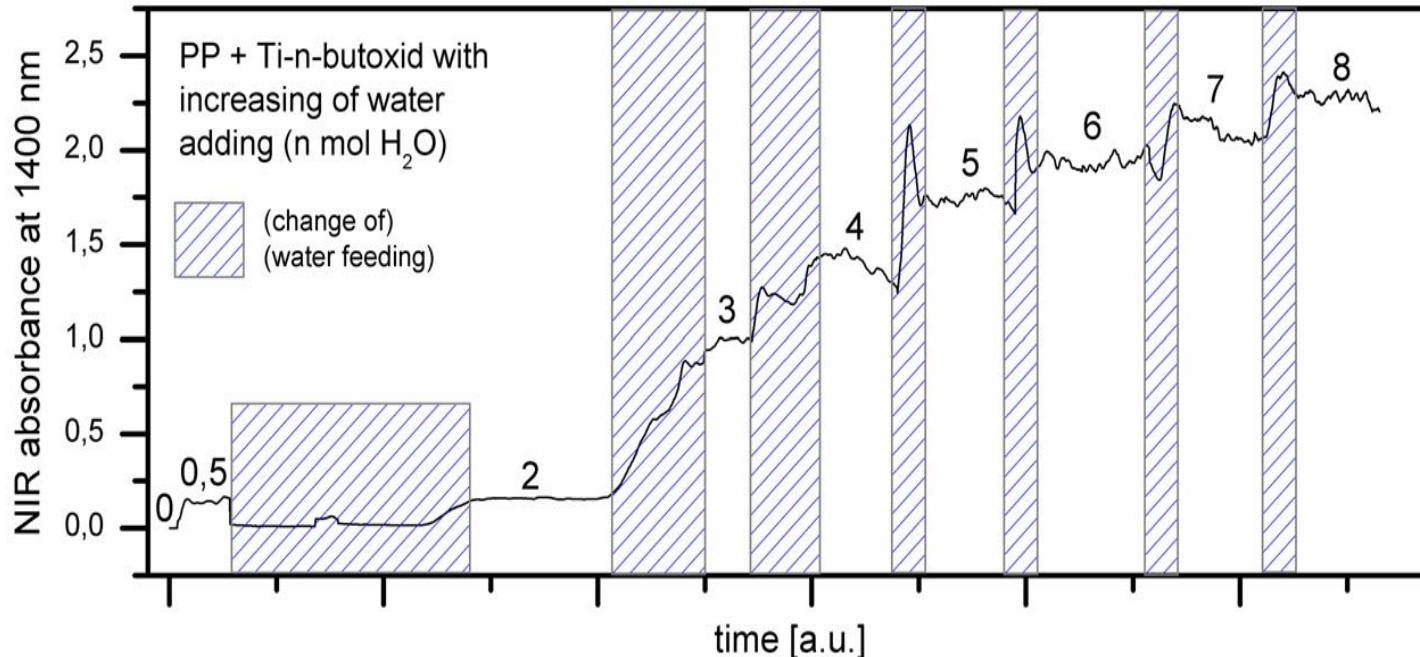
left: TEM image of PP/ $\text{TiO}_2$  (2 mol  $\text{H}_2\text{O}$ )



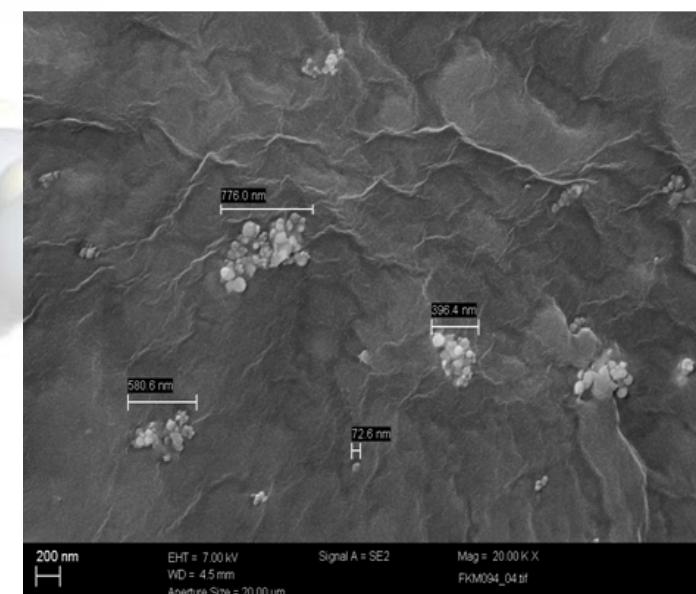
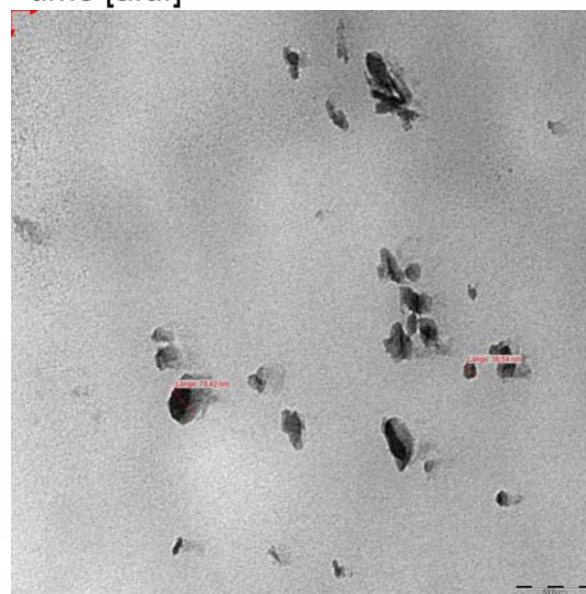
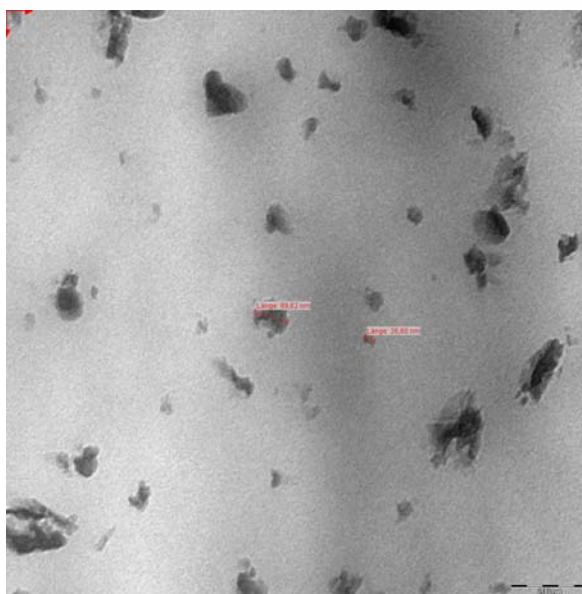
right: TEM image of PP/ $\text{TiO}_2$  (4 mol  $\text{H}_2\text{O}$ )



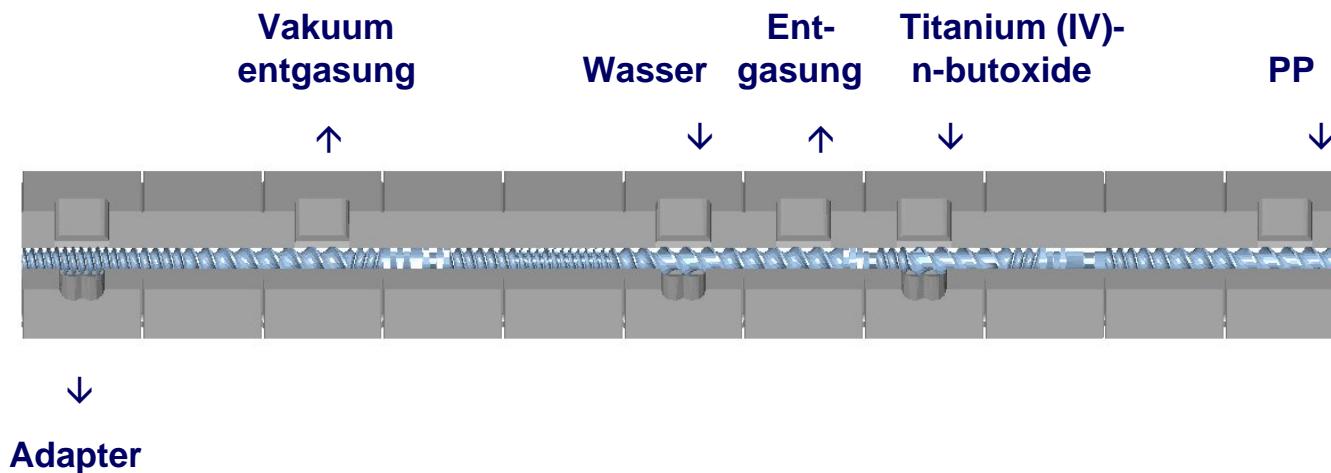
# Monitoring der Reaktiv-Extrusion zur in-situ Generierung von nanoskaligen $\text{TiO}_2$ -Partikeln in PP



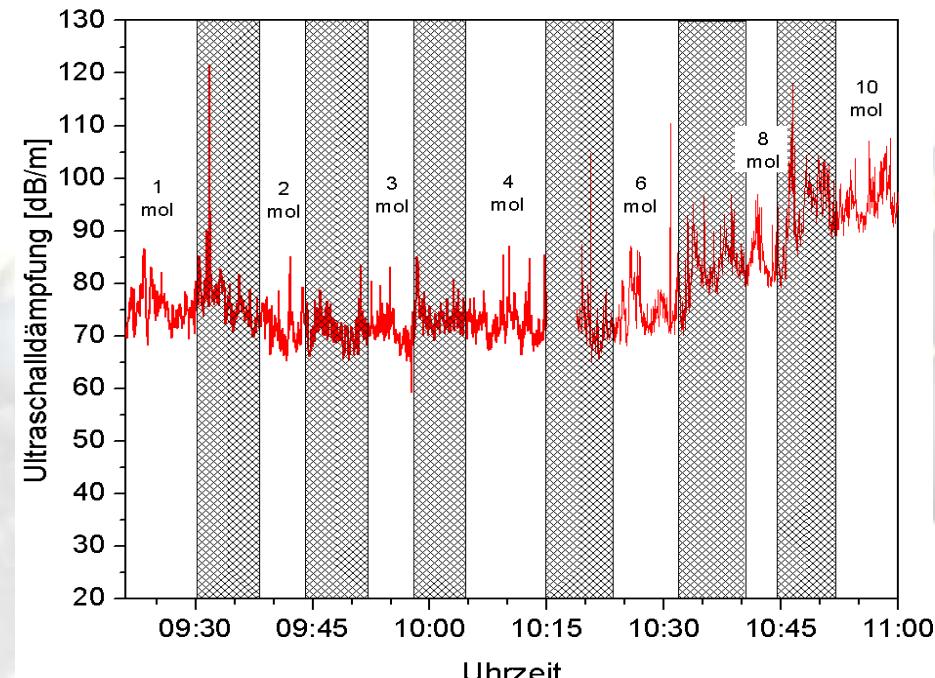
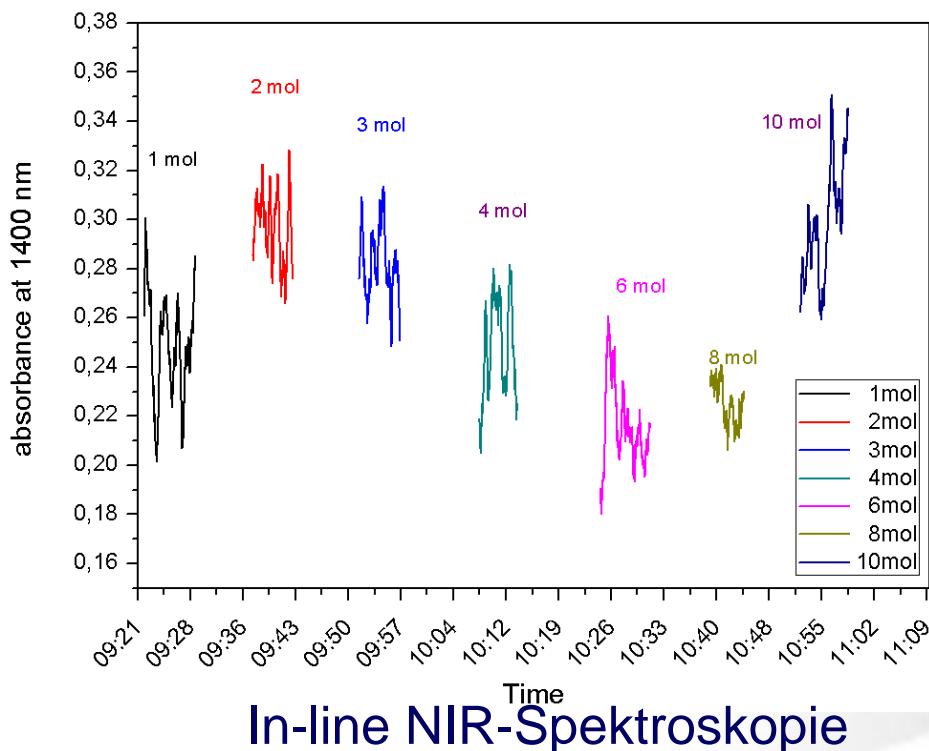
sample	average particle size SEM (nm)
PP+10%Ti-n-butoxide +	
0 mol $\text{H}_2\text{O}$	0
3 mol $\text{H}_2\text{O}$	152
4 mol $\text{H}_2\text{O}$	215
5 mol $\text{H}_2\text{O}$	268
6 mol $\text{H}_2\text{O}$	310



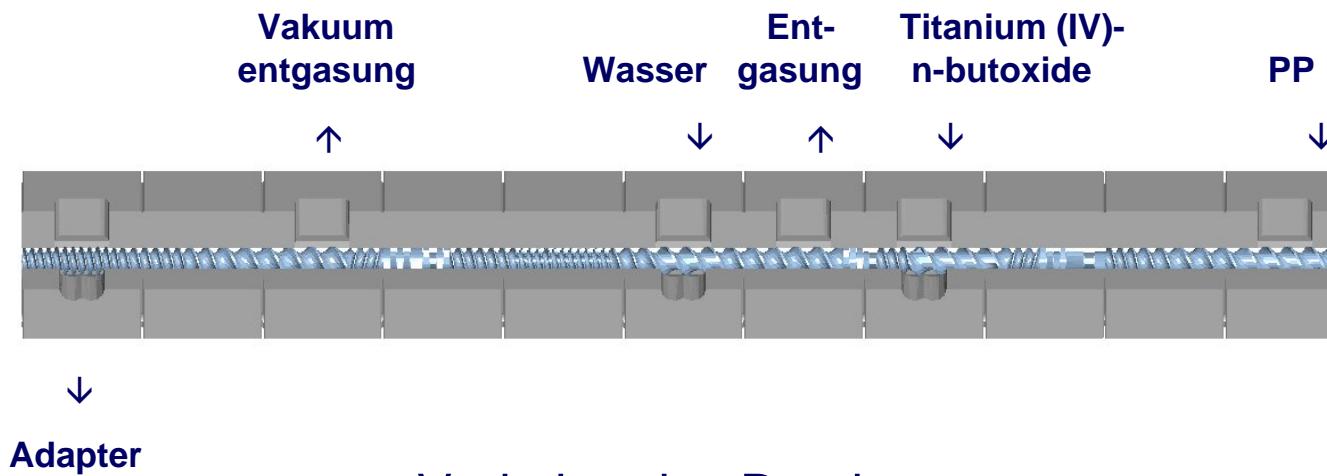
# Monitoring der Reaktiv-Extrusion zur in-situ Generierung von nanoskaligen TiO<sub>2</sub>-Partikeln in PP



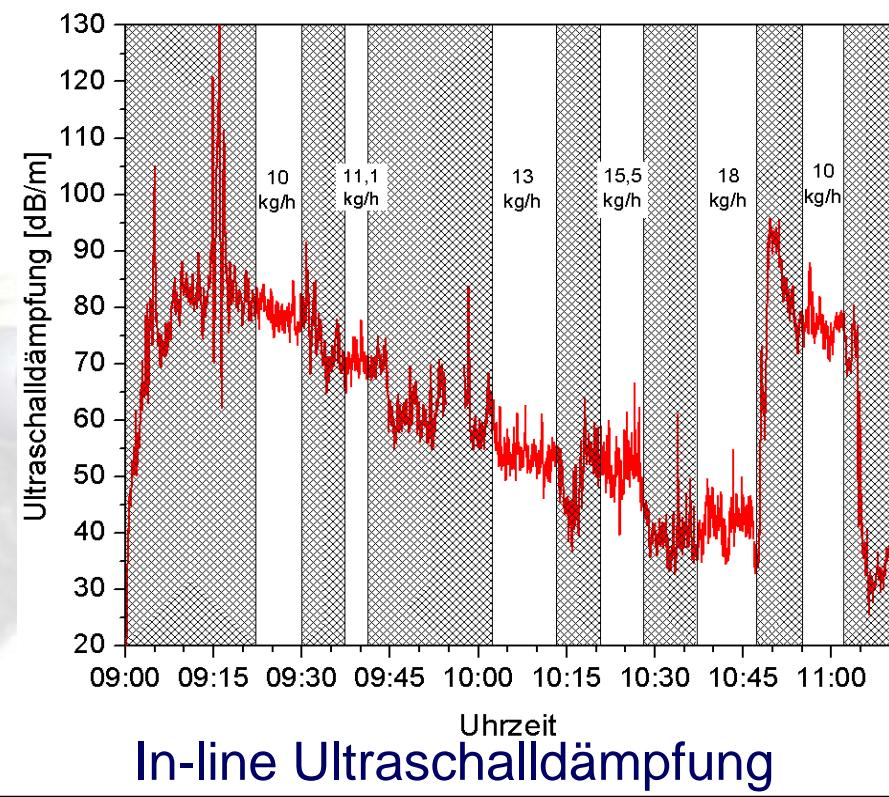
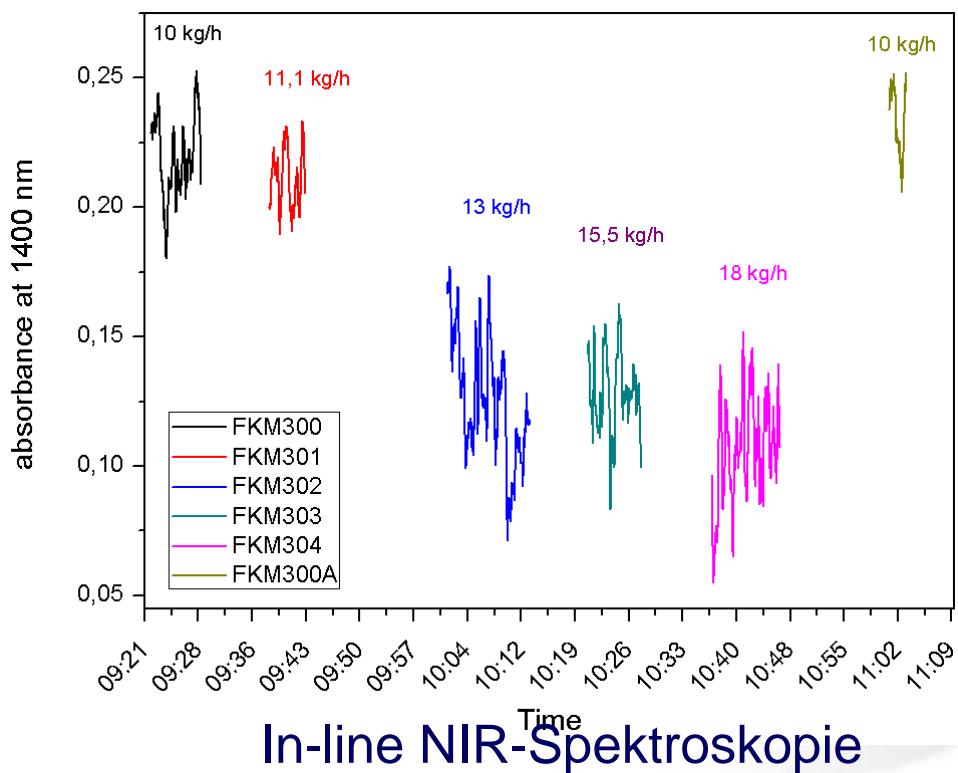
Variation der Wasserzugabe



# Monitoring der Reaktiv-Extrusion zur in-situ Generierung von nanoskaligen TiO<sub>2</sub>-Partikeln in PP

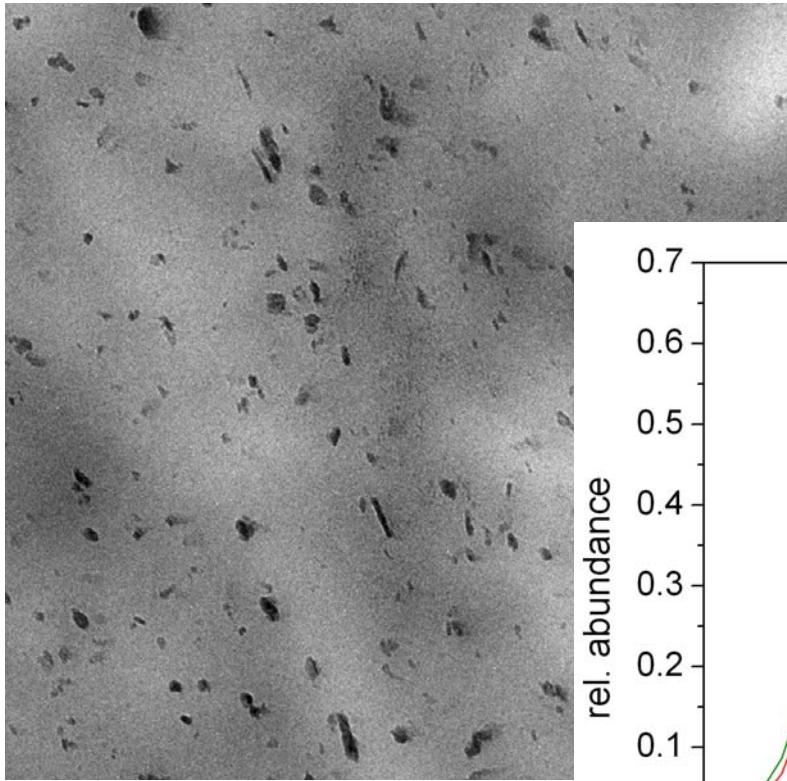


## Variation des Durchsatzes

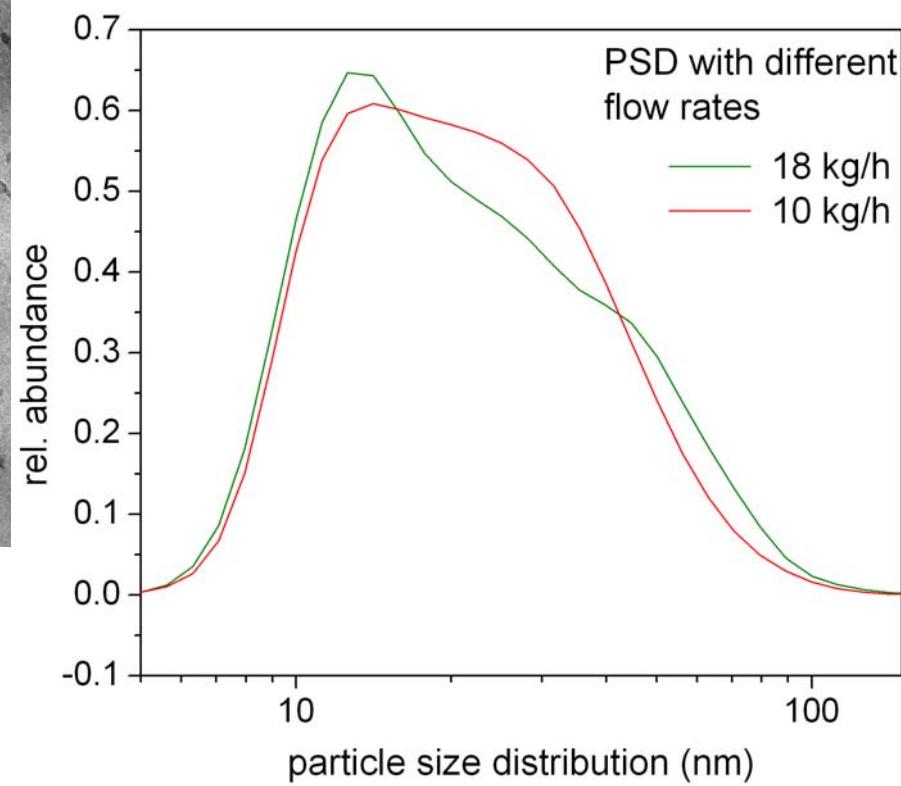


- Calculation of particle size distributions (PSD) using TEM measurements

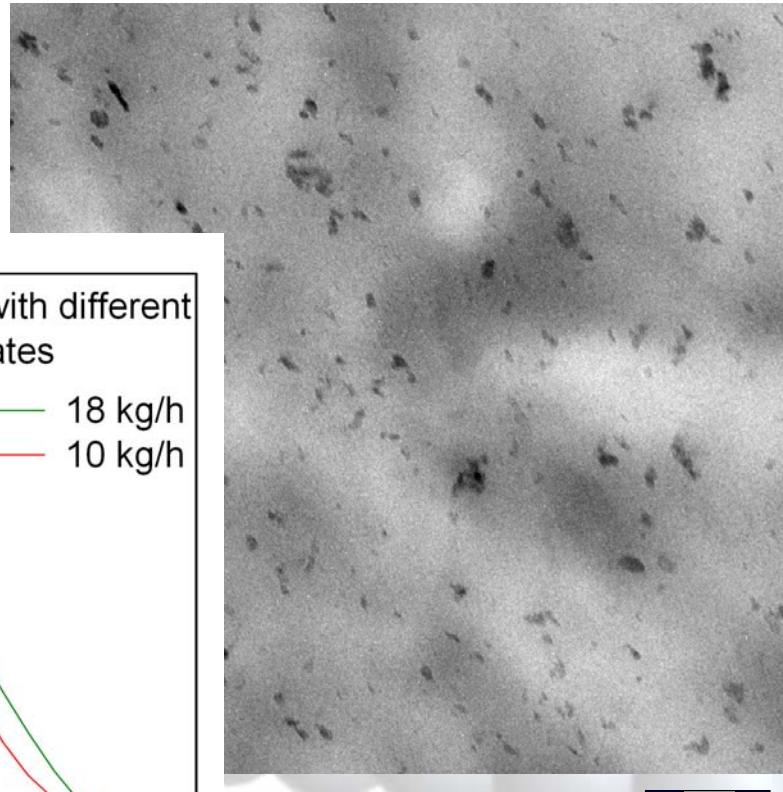
output: 10 kg/h



500 nm



output: 18 kg/h



500 nm

## Prozessüberwachung bei der Polymerverarbeitung und der Polymersynthese

Polymere mit Additiven, Polymer Blends, Polymercomposite,  
Nanocomposite und Hybridmaterialien

mit **in-line/on-line ATR-IR, NIR, RAMAN und Ultraschallmessungen**

Quantitative Bestimmung von:

- Zusammensetzung
- Partikelgröße
- Dispersion (Interkalierung/Exfolierung)
- Konzentration
- Umsatz/Kinetik
- Mechanische Eigenschaften

In Echtzeit in der Schmelze oder in der Lösung im Reaktor/Mixer/Extruder

Durch Variation der Prozessbedingungen (Drehzahl, Durchsatz, Temperatur) kann man in Echtzeit die optimalen Prozessbedingungen bestimmen

Messungen entlang des Extruders analysieren das Misch- und Dispergierverhalten

# Danke für ihre Aufmerksamkeit !

Dank an Mitarbeiter und Partner:

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DKI Darmstadt  
(Ultraschallmessungen)

