

# PREPARATION AND CHARACTERIZATION OF NANOCELLULOSE

## Motivation

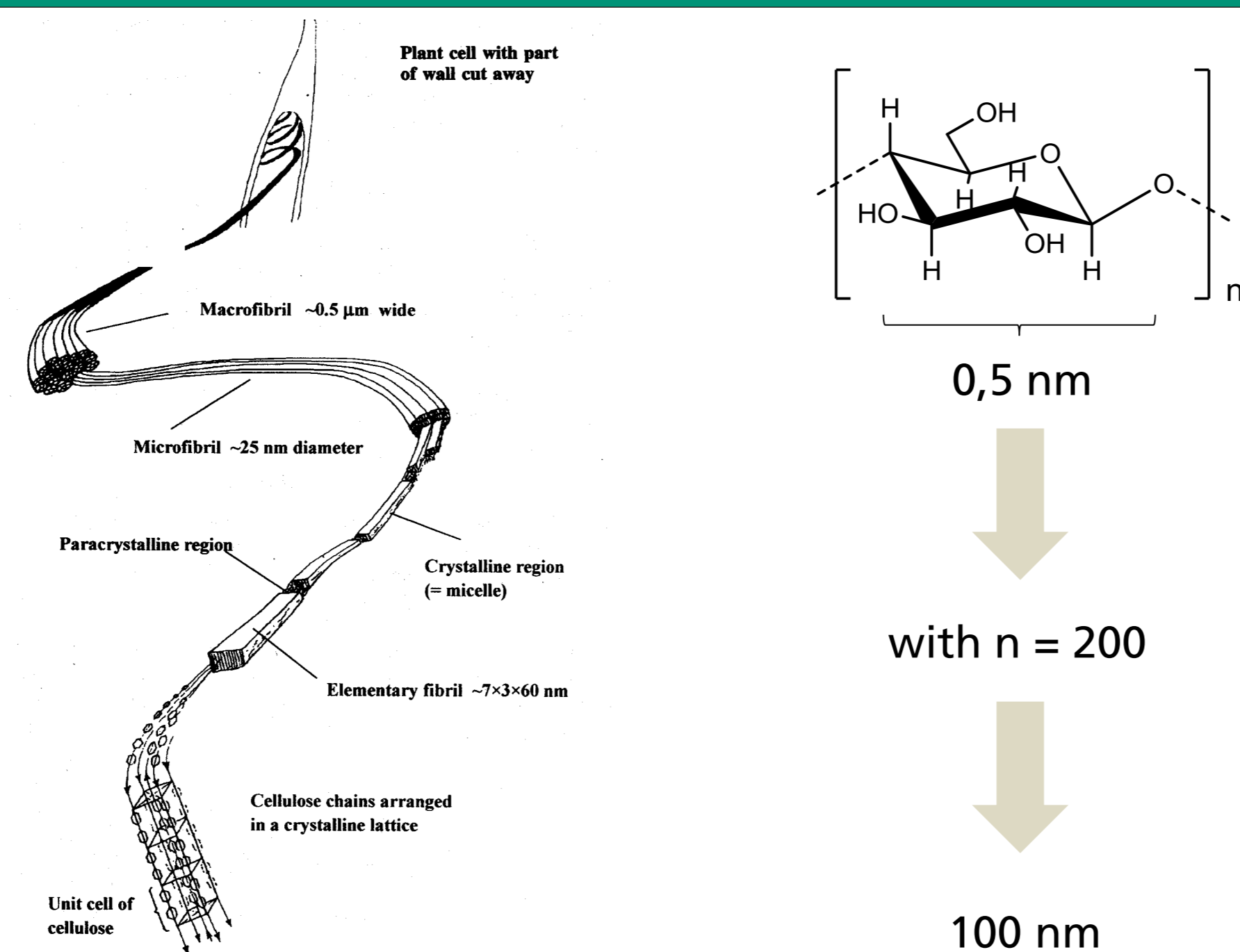


Fig. 1: Build-up of the plant cell from from macrofibril to microfibril to the elementary fibril down to the cellulose units (adapted from Roberts 1996)

As the most abundant organic and renewable polymer, cellulose is used in a wide variety of industrial products and applications (Kennedy et al. 1985; Klemm et al. 2005). In the recent past novel and interesting cellulosic materials were developed which were obtained by disintegrating cellulosic or lignocellulosic fibres using mechanical (Iwamoto et al. 2008; Abe et al. 2007), chemical (Moran et al. 2008) and enzymatic approaches (Rambo et al. 2008; Xing et al. 2007) or combinations thereof (Henriksson et al. 2007; Paeakkoe et al. 2007; Siró and Plackett 2010; Eichhorn 2011).

The aim of the project was the preparation of spherical cellulose particles with diameters in the nanometer range. We combine cellulose pre-treatment by grinding, dissolution and precipitation, hydrolysis or chemical derivatization of different pulps with subsequent mechanical treatment of aqueous suspensions of pre-treated or derivatized cellulose with a Microfluidizer® processor.

## Project

| Sample         |                             | DP (Cuen) |
|----------------|-----------------------------|-----------|
| Spruce pulp    | starting material           | 803       |
| WAM 1/04       | amorphization in NMMNO      | 725       |
| R05/99         | MF treatment                | 650       |
| Cotton linters | starting material           | 1316      |
| WAM 2/04       | amorphization in NMMNO      | 1146      |
| R05/98         | MF treatment                | 864       |
| R05/103        | Hydrolysis and MF treatment | 60        |

The preparation of the nanoparticles was carried out by different pre-treatments as grinding, amorphization (decrystallisation), hydrolysis or chemical derivatization of different pulps with subsequent mechanical treatment of aqueous suspensions of pre-treated or derivatized cellulose with a Microfluidizer® processor. As an example the results of amorphization from cellulose in combination with the mechanic treatment are shown following.

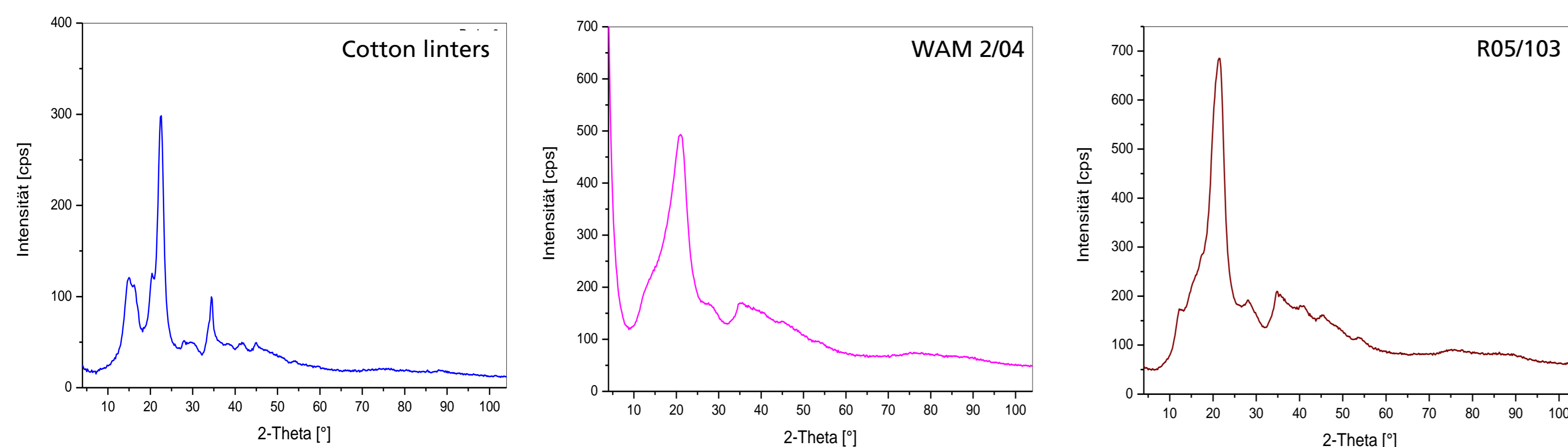


Fig. 2: Investigation of the decrystallized samples by WAXS

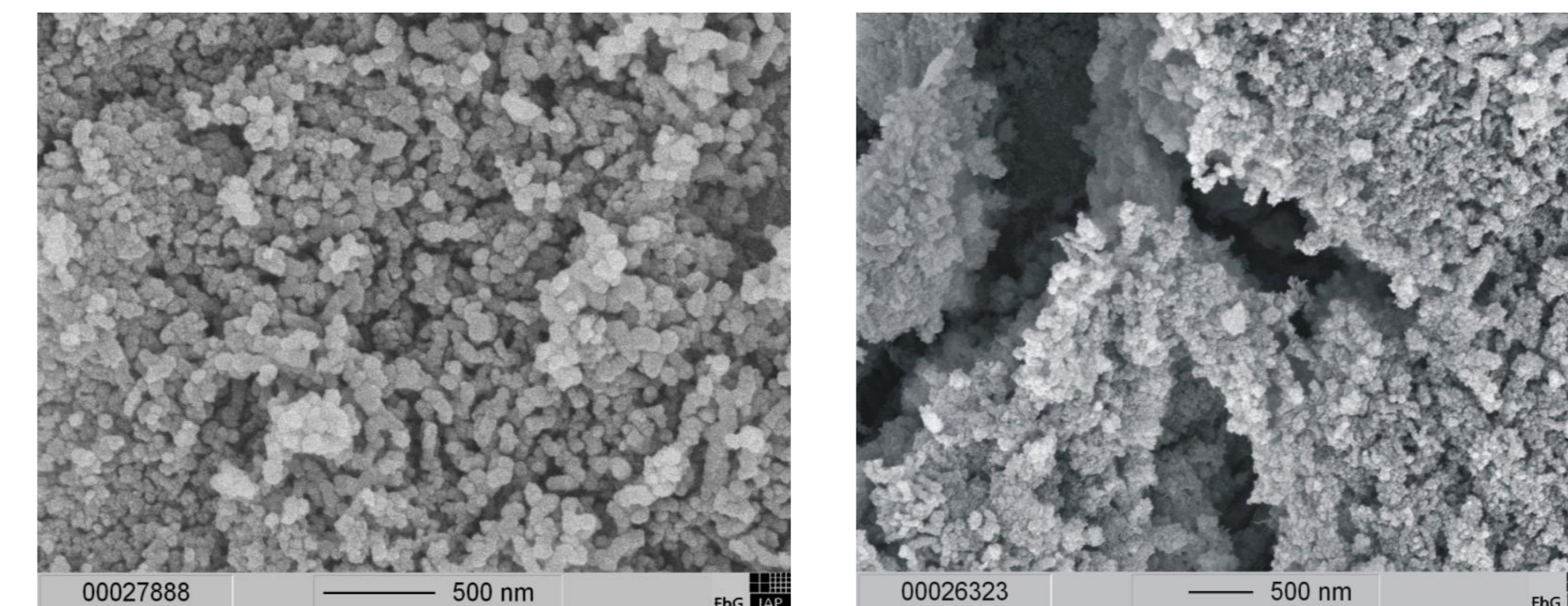


Fig. 3: SEM images of R05/98 and R05/103

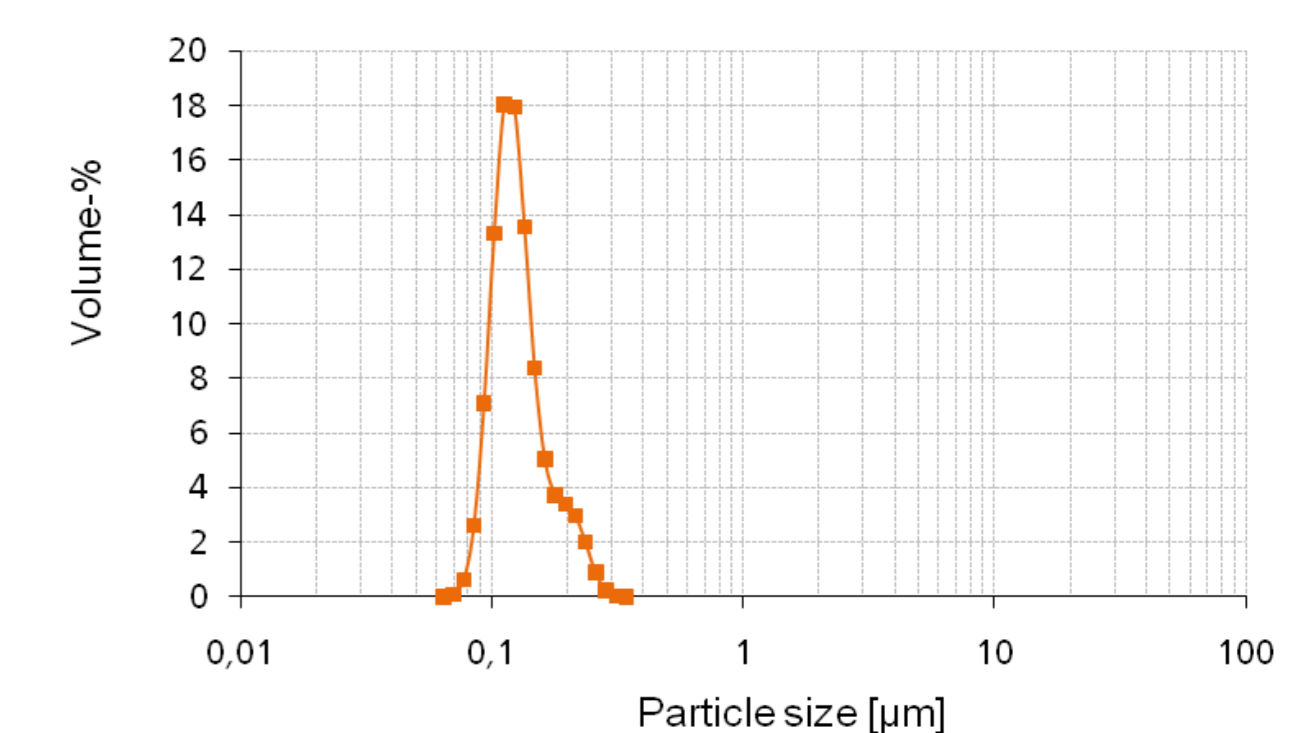


Fig. 4: Particle size distribution by dynamic light scattering ( $D_{90} = 193 \text{ nm}$ ;  $D_{50} = 128 \text{ nm}$ )

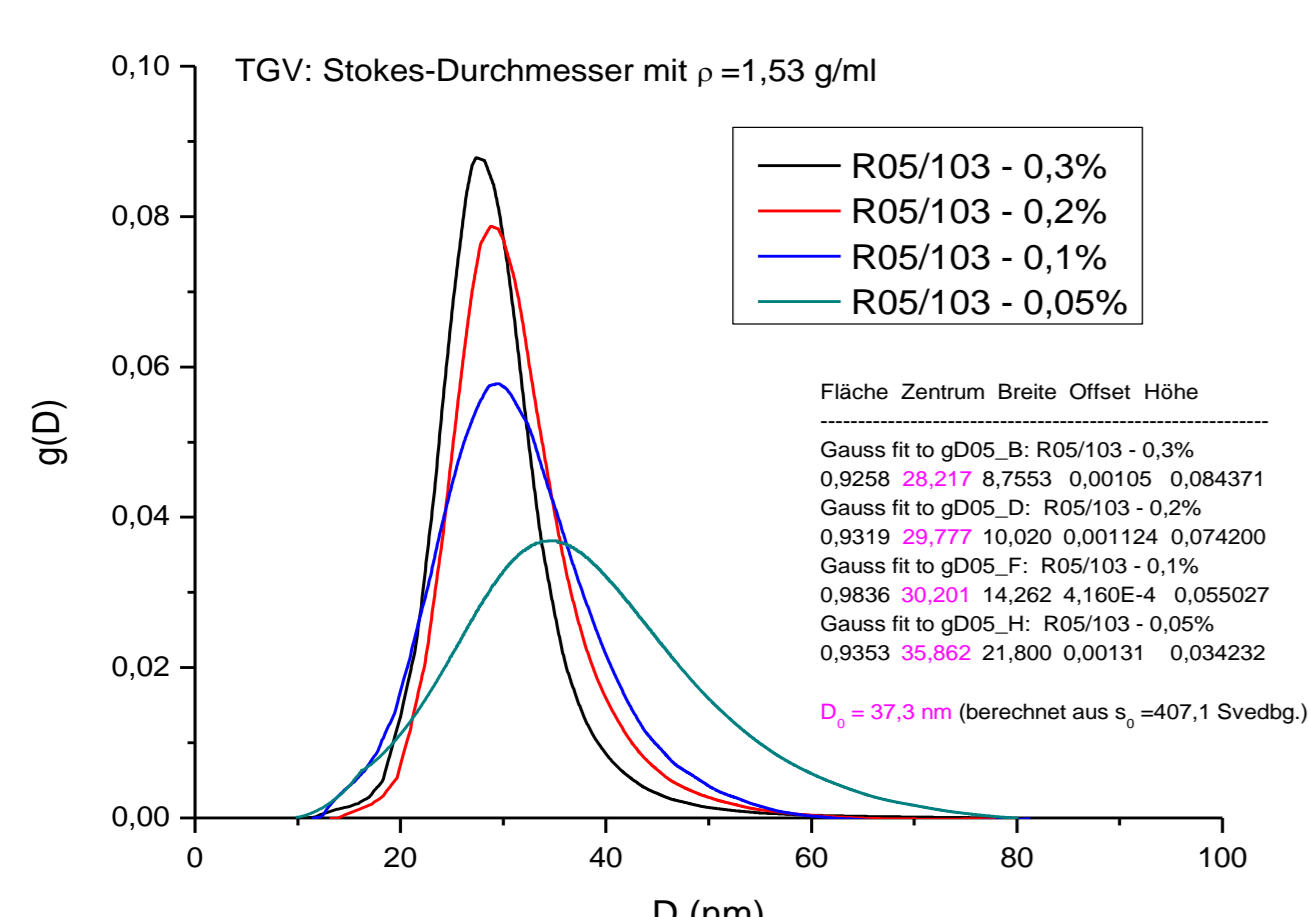


Fig. 5: Particles size distribution with ultracentrifugation

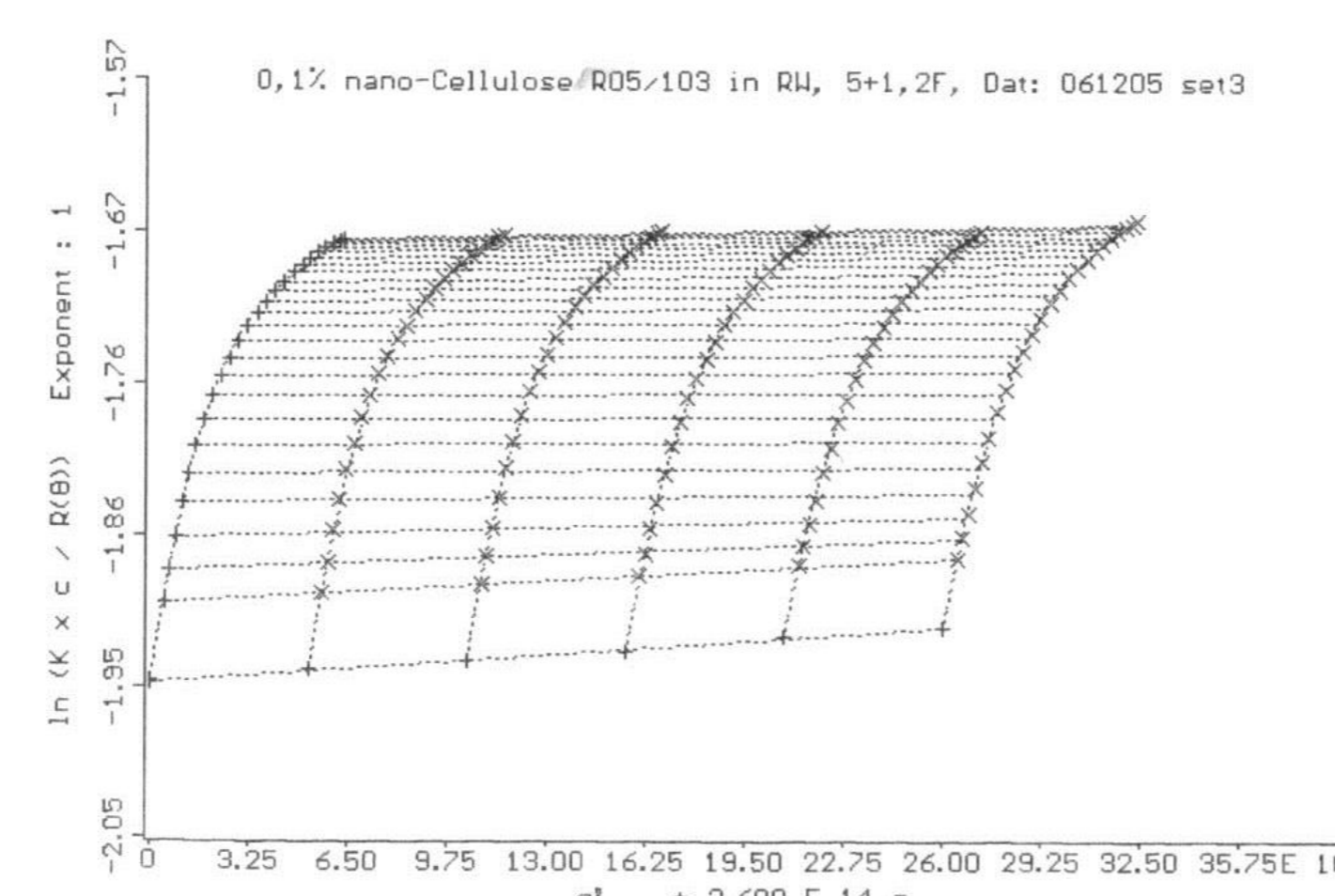


Fig. 6: Particles size distribution with static light scattering

Comparison of the particle sizes in dependence of the methods

| Method                   | Particle size [nm] | Advantages              | Disadvantages                             |
|--------------------------|--------------------|-------------------------|---|
| SEM                      | << 500             | optical analysis        | only small regions                        |
| Static light scattering  | 195                | direct and exact method | time-consuming, only monomodal systems    |
| Dynamic light scattering | 193                | direct and fast method  | not applicable for shear thinning samples |
| Ultracentrifugation      | 120-190            | direct and exact method | limited applicable for charged samples    |

## Summary

- Stable cellulose nanodispersion can be formed by an adequate pretreatment followed by a mechanical treatment with a Microfluidizer® processor
- Amorphization of cellulose as pretreatment is suited for preparation of nanocellulose
- Characterization of nanocellulose dispersion with different analytical tools result to similar particle size distributions

### References

- Abe K, Iwamoto S, Yano H (2007) Biomacromolecules 8: 3276  
Eichhorn SJ (2011) Soft Mater. 7: 303  
Henriksson M, Henriksson G, Berglund LA, Lindstrom T (2007) Europ. Polym. J. 43: 3434  
Iwamoto S, Abe K, Yano H (2008) Biomacromolecules 9: 1022  
Kennedy JF, Phillips GO, Wedlock DJ, Williams PA (Eds.) (1985) Cellulose and its derivatives: chemistry, biochemistry and applications. Horwood: Chichester  
Klemm D, Heublein B, Fink H-P, Bohn A (2005) Angew. Chem. 44: 3358  
Moran JI, Alvarez VA, Cyrus VP, Vazquez A (2008) Cellulose 15: 149  
Paeakkoe M et al. (2007), Biomacromolecules 8: 1934  
Rambo CR, Recouvreux DOS, Carminatti CA, Pitlovanciv AK, Antonio RV, Porto LM (2008) Mat. Sci. Eng. 28: 549  
Siró I, Plackett D (2010) Cellulose 17: 459  
Xing O, Eadula SR, Lvov YM (2007) Biomacromolecules 8: 1987

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