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# Nanocellulose based piezoelectric sensors

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13.09.2018

Kestävää kasvua ja työtä -ohjelma
TAMPEREEN TEKNILLINEN YLIOPISTO
Vipuvoimaa EU:lta 2014–2020
Euroopan unioni
Euroopan aluekehitysrahasto

# Outline

- Piezoelectric nanocellulose sensors
- MFC-film sensor demonstrators
- Applications and business potential

# Piezoelectricity of cellulose

- **Piezoelectric effect** = Electric charge separation by applied mechanical force

- The piezoelectric tensor $d_{mn}$ is determined by the **symmetry of a crystal lattice**

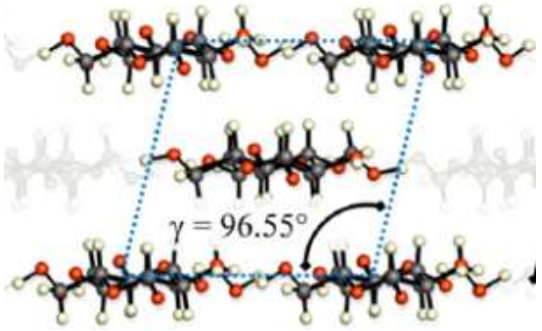
  [E. Fukada, *J. Phys. Soc. Japan* (1955)]

- The **monoclinic C2 symmetry** and the cancellation effects in wood cellulose crystal structure result into **piezoelectric coefficient matrix**:

$$d_{mn} = \begin{pmatrix} 0 & 0 & 0 & d_{14} & 0 & 0 \\ 0 & 0 & 0 & 0 & d_{25} & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 \end{pmatrix}$$

$$\text{where} \quad d_{14} = -d_{25}$$

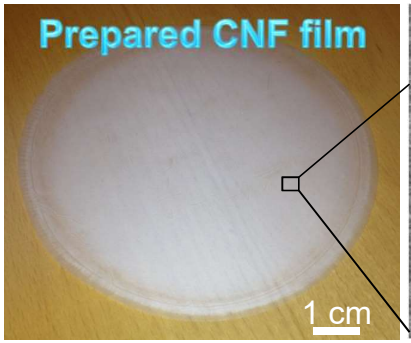
Cellulose crystal (CNC) $[[C_6H_{10}O_5]_n]$ unit cell:

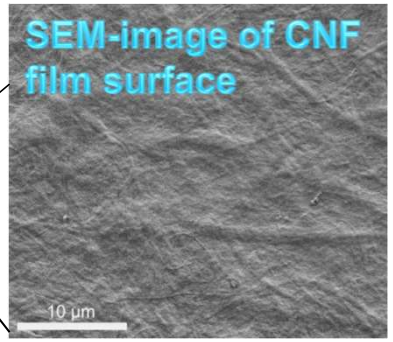



[Zuluaga et al. (2013)]

# CNF film fabrication

- Cellulose nanofibril (CNF) obtain by **mechanical homogenizing** process from a bleached birch cellulose mass in aqueous solvent
  - 3 passes through a Masuko grinder
  - 6 passes through a microfibrillator

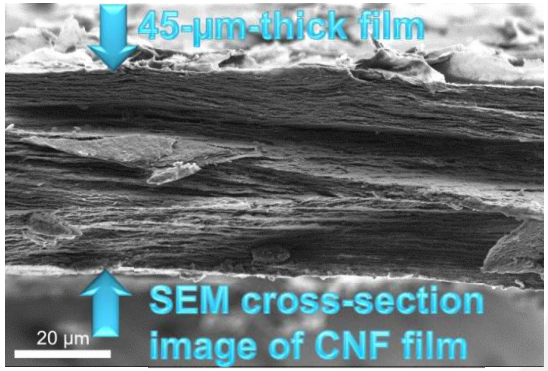



- CNF film by **pressure filtering**, followed by pressing and drying in **hot-press** (2 h @ 100 C)
  - A bendable 45 µm CNF film

Amorphous nanocellulose regions

Cellulose nanocrystals (CNC)

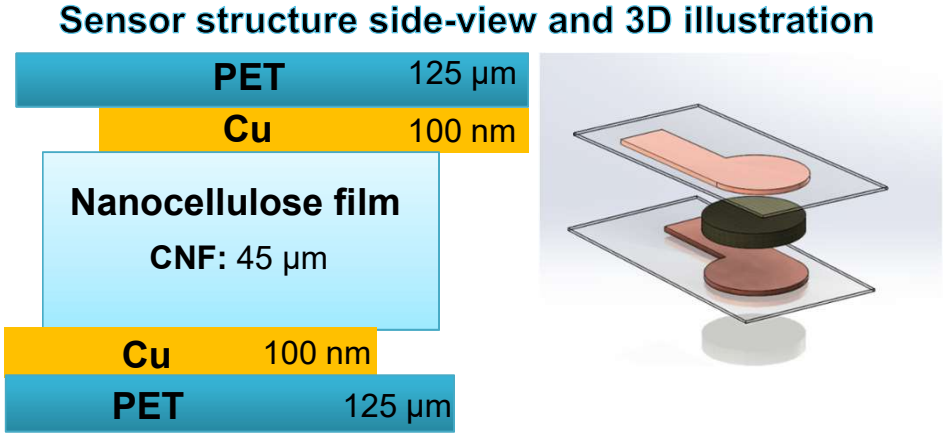


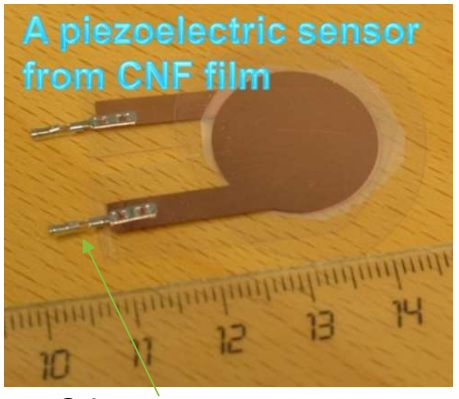

- CNF film contains both **amorphous cellulose and crystalline CNCs**, but CNCs are not optimally aligned inside the film.

For more information: Rajala *et al.*, "***Cellulose nanofibril film as a piezoelectric sensor material***", ACS Applied Materials & Interfaces **8**(24) (2016) 15607.

# Piezoelectric sensor assembly

- CNF-film pieces assembled **between two evaporated copper electrodes** on polyethylene terephthalate (PET) substrate using adhesive film
- **Crimp connectors** (Nicomatic Crimpflex) were used for getting a reliable contacts to the copper electrode on flexible PET substrate




For more information: Rajala *et al.*, "***Cellulose nanofibril film as a piezoelectric sensor material***", ACS Applied Materials & Interfaces **8**(24) (2016) 15607.

# Piezoeletric sensor measurements

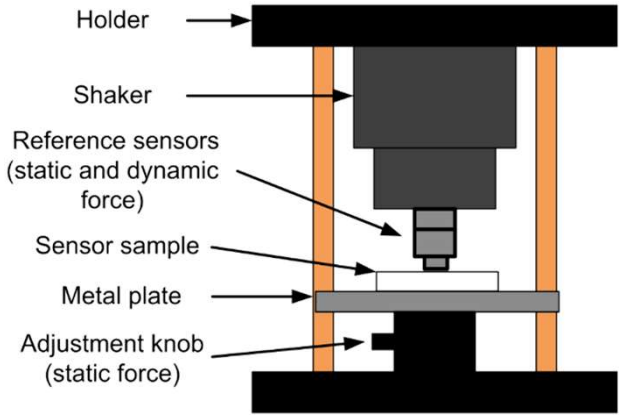
- Piezoelectric sensitivity measurement setup:
  - **Mini-Shaker** (Brüel & Kjaer type 4810)
  - **Dynamic and static force** sensors (normal direction)
  - **Charge amplifier** for sensor output measurement

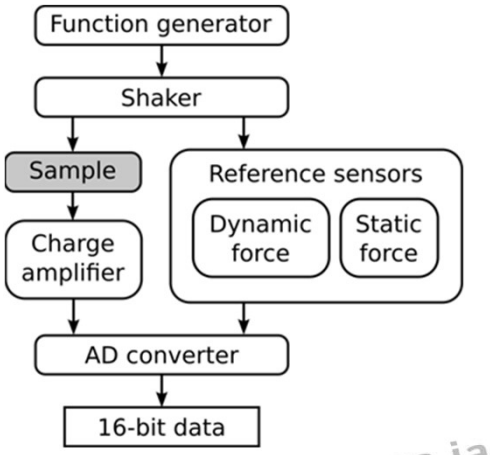
- Measurement parameters
  - **3 N static force** to keep sample steady and **1.3 N dynamic force** excitation with **2 Hz sinusoidal** input voltage

- **Sensor sensitivity** by dividing the generated charge by the dynamic force, giving a **unit pC/N**
  - The **sensor sensitivity** closely related to **perpendicular piezoelectric coefficient $d_{33}$** (from piezoelectric tensor)

$$Sensitivity = \frac{Q_{sensor}}{F_{dynamic}}$$

[For details see: S. Tuukkanen *et al.*, Synthetic Metals (2012) or IEEE Sensors (2015)]

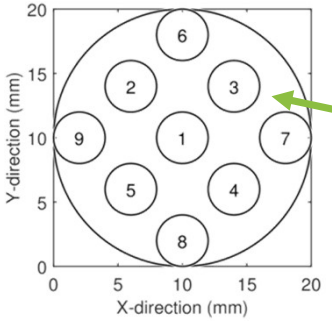


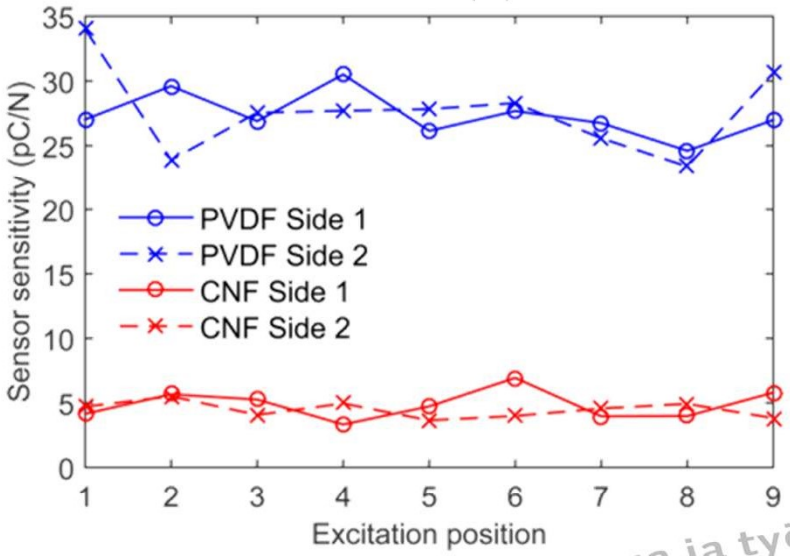
# Sensitivities of CNF sensors

- Average sensitivity:
  - **4.7 pC/N for CNF film**
  - 27.5 pC/N for PVDF reference sensor
  - For comparison: 2.3 pC/N for quartz

- Small variations in sensitivity between different positions on the sensor electrode
  - Good homogeneity of the CNF film




Sensor electrode positions

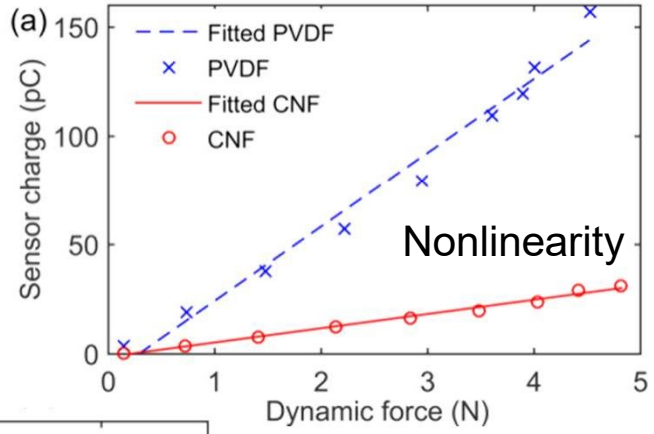


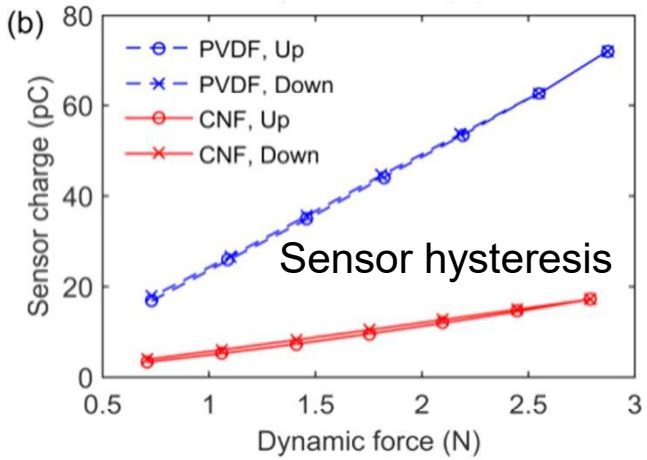

For more information: Rajala *et al.*, "***Cellulose nanofibril film as a piezoelectric sensor material***", ACS Applied Materials & Interfaces **8**(24) (2016) 15607.

# Linearity and hysteresis of CNF sensors

- **Nonlinearity** was found to be (0.86 ± 0.48) pC for CNF and (6.47 ± 3.76) pC for PVDF
  - **Charge vs. Force** curve
  - Fitted a first degree polynomial via least squares minimization

- **Sensor hysteresis** was **below 1 pC** in maximum for both sensors
  - Increasing force vs. Descreasing force

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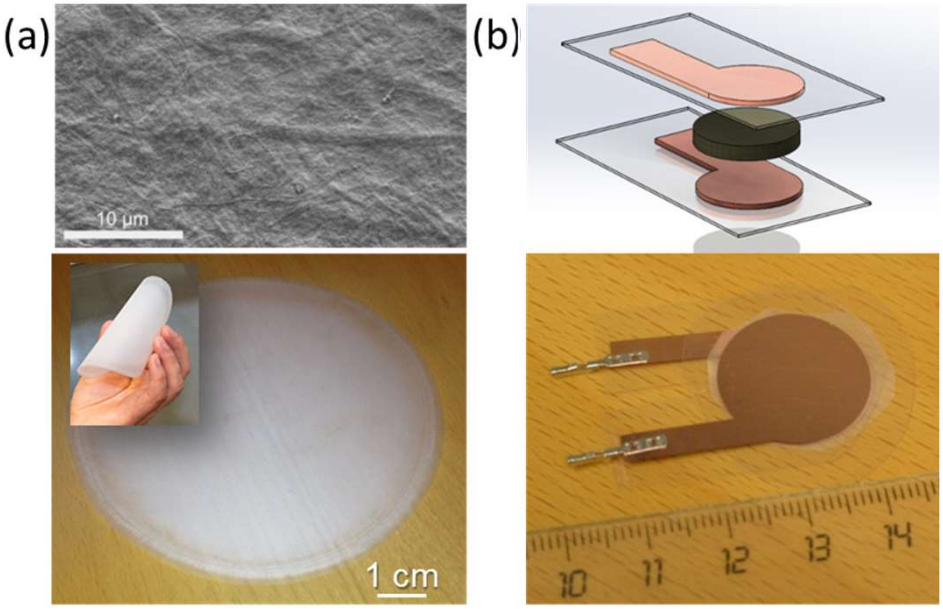
# Summary of previously published work

- **Nanocellulose is a promising solution-processable, renewable and disposable piezoelectric material!!**
  - ➢ Wide range of sensor applications
- **Future perspectives:**
  - ➢ CNC crystal orientation/polarization to enhance sensor performance
  - ➢ Improved mechanical properties by additives
  - ➢ Bio-based electrodes materials

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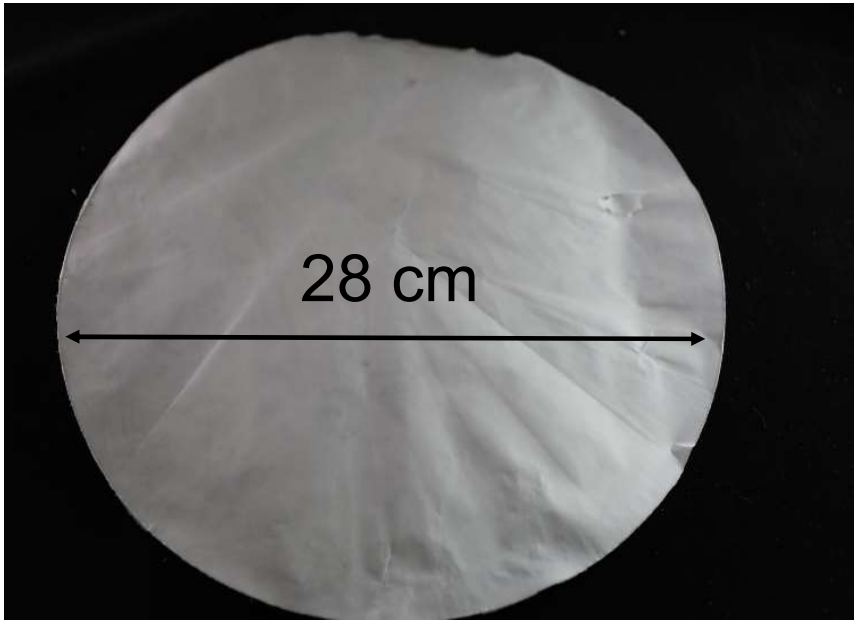
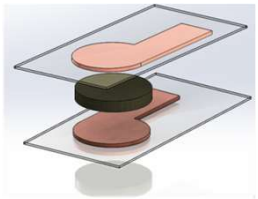
# MFC-film fabrication demonstrator

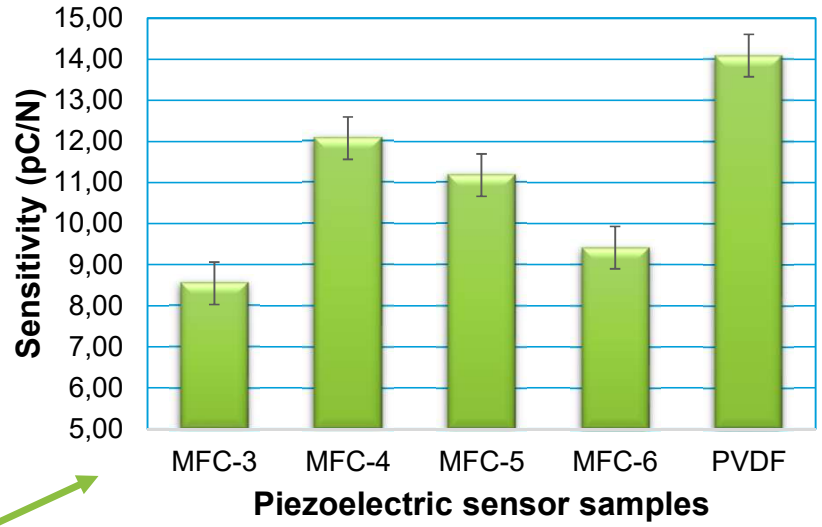
- MFC-film was prepared by **casting method** on hot-plate on a plastic substrate
- Dry MFC-film peeled of from plastic film manually
- Robust and flexible freestanding about 50-µm-thick MFC-film was obtained
- Can be used as a substrate for printed electronics and as a sensor material



# MFC-film sensor demonstrators

- For sensitivity measurements, MFC-films were sandwiched between evaporated Cu-electrodes on PET
- Commercial referefence sensor (Measurement Specialities Inc.)



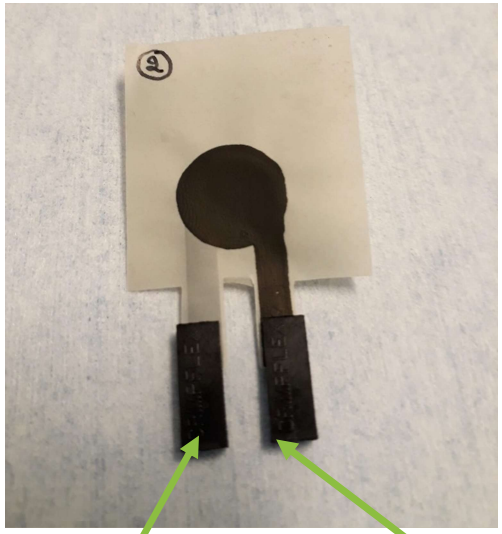




- Measured piezoelectric sensitivity 8-12 pC/N, which is slightly higher than in recently published CNF-film sensors.

# Screen printed electrodes on MFC-film sensor

- An example of screen printed graphite-ink electrodes on MFC-film
- Electrodes printed and cured on both sides of the film subsequently.

→ Measurements ongoing…

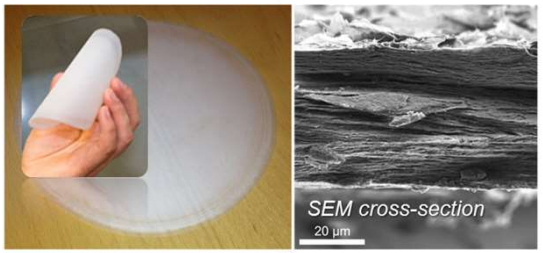


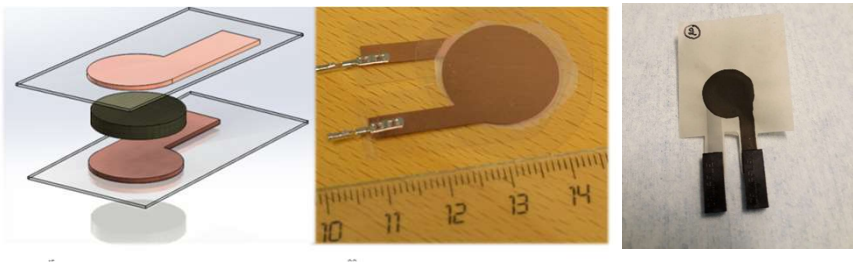
# Applications and business potential

**Business challenges:**

- Genuinely bio-based sensor elements
- One-concept recyclable or disposable sensors

Nanofibrillated cellulose (CNF) film characterization



**The solution:**

- Nanocellulose as piezoelectric sensor material!
- Potential applications, e.g.
  - Internet-of-things (IoT)
  - Ambient intelligence
  - Personal point-of-care diagnostics
  - Sensors for physiological measurements
  - Elements for energy harvesting applications

**Result for industry:**

- Pilots in BioÄly-project provide further information about the applicability and suitability in industrial applications!!

# *Thank You! Kiitos!*

**Project's Principal investigators:**

*Johanna Lahti,* ***Sampo Tuukkanen****, Tero Juuti,*

*Tomas Björkqvist, Matti Mäntysalo*

**Project staff**:

*Jari Keskinen, Jarkko Pakkanen, Sanna Siljander,*
*Juuso Toriseva, Hanna Christophliemk,* ***Arno Pammo***

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