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Introduction

Physics with Industry 2019: Creating original solutions for industrial problems

In November 2019 the tenth edition of Physics with Industry took place at the Lorentz Centre in Leiden. A group of 35 researchers worked together in small groups to solve real company problems. The five participating companies, selected by a scientific committee, that delivered an industrial case were NIZO Food Research, Somni Corporation, Feltest Equipment, ConSensor and Friesland Campina.

The cases had a broad range of topics. For instance, the researchers had to discuss biophysical problems with NIZO about speeding up the ripening process of cheese by breaking down the process into separate steps, showing possible solutions in increasing the enzyme efficiency and altering the nucleation sites and nucleation time of crystals. For Friesland Campina, the case involved investigating the physical phenomena in Lattiz membranes occurring during milk foaming by evaluating the Scanning Electron Microscope image of the membrane, looking at the bubble size distribution and making calculations for the surface tension of the bubbles.

The Somni case was about solving an optical engineering problem. The goal was to protect a sensitive fiber bragg grating accelerometer during transport and installation. Different options were examed, for example the possibilitie of using a magnet to detach the mass during transport, using liquid to damp transportation shocks, or improving the sensitivity allowing a smaller mass which reduces the chance for damage.

For two cases, the development of new measuring technologies was the central task. Feltest was looking for options to improve paper production. The researchers designed a sensing technology for measuring felt thickness by exploring possibilities in measuring with ultrasound, electromagnetic radiation, magnetic resonance imaging and conductivity. With ConSensor, the researchers looked at improving chloride concentration measurements in concrete to better prevent corrosion of steel in reinforced concrete. Therefore they looked for solutions in the fields of pH-based sensors, absorptions based spectroscopy, electro-magnetic induction based detections and chemical sensors.

The scientific quality of the workshop was guaranteed because of the coaching of an academic and an industrial supervisor. This ensured companies of a valuable result and allowed the participants to enjoy the workshop because of the scientific challenges and the experience of working with a real industrial problem.
Developing a sensor for determining chloride content in reinforced concrete

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1. Description of company case

• **Problem description**: Reinforced concrete is one of the most widely used construction materials globally\(^1\). Concrete consists of aggregates and paste, a mixture of cement and water, which binds the aggregates. Reinforced concrete is a concrete in which steel is embedded in such a manner that the two materials act together in resisting forces. Concrete structures in coastal and in marine environments or infrastructure objects exposed to de-icing salts suffer from chloride ingress\(^2\). In particular, because concrete is a rather porous material, chloride ions can diffuse through concrete structures (Figure 1). Once the free chloride ions reach the steel rebar in concrete, the steel corrosion starts. The corrosion leads to the formation of the rust products on the steel surface. The rust products expand causing the concrete to crack and thus compromising the structural integrity, provided that conditions for corrosion are met (presence of oxygen, water and increasing amount of chlorides). Chloride-induced corrosion of steel is among the main causes of damage of concrete structures and premature damage of new constructions\(^3\). Thus, a successful determination of chloride concentration in concrete is of a paramount significance, allowing preventative measures to be taken promptly. Such an approach would prolong the lifetime of concrete structures making them more durable as well as address safety concerns.

**Figure 1**: (left) The effect of chloride ingress on bridge columns. (right) Schematic depiction of chloride ingress through the concrete cover. The gradient represents the chloride concentration along the depth profile.

• **Scientific challenge**: The existing techniques for determining the chloride content in concrete suffer from many disadvantages. Most of the techniques are destructive, requiring the extraction of a 10 cm diameter concrete sample from an existing construction\(^4\). Such a sample is then analyzed in a lab for the chloride concentration. The extraction of such samples can be done for a limited number of times before causing damage to the existing building. In addition, it tends to be laborious and time consuming.
Other non-destructive techniques mainly rely on embedding a sensor within the concrete. However, chloride sensors, such as the silver/silver-chloride sensor\(^5\), have much shorter working lifetimes compared to the onset of chloride-induced steel corrosion. Furthermore, such sensors allow local measurements (spot measurements), without details on concentration profile within concrete cover (distance from concrete surface to steel reinforcement). Chloride concentration profile would be essential in detecting and predicting chloride diffusion across the concrete cover, allowing accurate estimation of concrete construction service life as well as serving as an indicator for preventative intervention.

Accordingly, it would be of large importance if we develop a measurement approach and device that would be chloride specific, have a lifetime that is longer than the construction lifetime and provide information within 1% error. Furthermore, the developed solution should be cost effective, relatively compact, portable and easy to operate. Ideally the sensor should be able to detect chloride in concrete in both existing constructions as well as future constructions. The critical concentration of chloride in concrete is 0.4% by cement weight. The technique should be therefore rather sensitive to be able to detect relatively low concentrations of chloride in concrete. Additionally, concrete pore water is highly alkaline (pH = 13-14)\(^6\). This means that the device should have a long term chemical stability.

- **The main scientific question**: To develop a method for determining the chloride concentration in concrete along the concrete cover depth in order to prevent corrosion of the steel rebars in concrete.

### 2. Solution(s)

Taking into account all the limitations and shortcomings of current techniques, we came up with 5 different solutions to address the scientific question. The proposed solutions, their working principles and their evaluation as viable solutions are shown in Table 1:

**Table 1: Possible solutions**

<table>
<thead>
<tr>
<th>Sensor</th>
<th>Working principle</th>
<th>Detection principle</th>
<th>Measurement type</th>
<th>Advantages</th>
</tr>
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<tbody>
<tr>
<td>pH-based sensor</td>
<td>a solid pH probe which changes absorption based on pH environment</td>
<td>Detect the pH close to the reinforcement which is an indication of corrosion initiation</td>
<td>local</td>
<td>on site measurement non-destructive</td>
</tr>
<tr>
<td>Absorption based spectroscopy</td>
<td>Fibre optic based sensor sensitive to chloride ion absorption</td>
<td>Detect change in intensity of light that bounces back from concrete proportional to amount of chloride ions</td>
<td>local</td>
<td>on-site measurement non-destructive direct measurement of chloride</td>
</tr>
<tr>
<td>Corrosion detection based on electro-magnetic induction</td>
<td>induced current around a magnetic core is detected. Depending on current measurement corrosion of magnetic core is quantified</td>
<td>Detect corrosion and thereby anticipate corrosion in the reinforcement before it begins</td>
<td>local</td>
<td>on-site non-destructive</td>
</tr>
<tr>
<td>Chemical sensor</td>
<td>Chemical detection of chloride ions by reaction with silver nanoparticles</td>
<td>Change in the optical properties of Ag nanoparticles after binding to chloride ions</td>
<td>local</td>
<td>On site Less destructive compared to current extraction methods Chloride specific Detector is re-usable Not expensive</td>
</tr>
</tbody>
</table>
Chloride concentration along depth profile possible

| Disadvantages                  | indirect measurement of chloride lifetime of sensor unknown | complex precise sensor required further research needed | indirect measurement of chloride | requires sample preparation requires multiple sampling and validation |

All of the above cases were judged on the basis of the complexity of the solution, affordability, how selective it is (detecting chloride ions) and portability. Based on the number of advantages of the chemical sensor and taking into account the scientific question, of requiring a chloride specific sensor, we choose to focus on the chemical sensor and develop it further.

The working principle of this method is based on the oxidative etching of silver nanoparticles (AgNPs) to form AgCl in the presence of Cl\(^-\) and H\(_2\)O\(_2\)\(^2\) (reaction (i)). This chemical reaction will take place in a device consisting of a channel embedded within a Polydimethylsioxane (PDMS) mold. The dimensions of the channel are approximately 0.2 cm in width and 5 cm in length. The channel contains an aqueous solution of AgNPs of about 10 nm diameter.

\[
H_2O_2 + Ag^0 + 2Cl^- + 2H^+ \leftrightarrow 2AgCl_{(s)} + 2H_2O \tag{i}
\]

This device is schematically shown in Figure 2. When a sample containing Cl\(^-\) ions is injected into the channel, together with a solution of H\(_2\)O\(_2\), AgCl particles are formed. This chemical change results in a corresponding change in the optical properties of the solution. In particular, the change in optical properties can be measured as a reduction in the transmission of light through the medium.

Figure 2 also shows the complete schematic of our proposed measuring assembly. It consists of a handheld detector containing a light emitting diode (LED) operating on visible spectrum. The light passes through the PDMS device containing the reacted solution. The change in transmittance of light will be registered via a silicon photo diode sensitive in the similar range of wavelength of the emitting diode. This change corresponds to a predefined chloride ion concentration which shall be displayed in the display of the hand-held device.

**Figure 2:** (left) Schematic depiction of the concrete channel from which the Cl ions are extracted. A channel is embedded within PDMS where Ag nanoparticles (blue) are dispersed. As the Cl ions (red) diffuse towards the injected water, the water infused with Cl is sucked towards the Ag decorated channel. (right) Schematic of the device in which the channel (with Ag and extracted Cl ions) is inserted to quantify the Cl concentration. The intensity of the detected light by the photodiode will change depending on the concentration of Cl which reacted with the Ag nanoparticles.

The measurement proceeds as follows:
1) A prefabricated or drilled channel (of a small diameter) in the concrete is used to access the concrete at different depths.

2) A small volume of water (~100 µL) is injected into the channel and is left to interact with the surface at the bottom to allow the Cl- ions to diffuse to it.

3) The volume is collected with a pipette and injected into the device. The device contains a channel embedded within a PDMS (see Figure 2) in which silver nanoparticles are dispersed.

4) As the Cl- ions interact with the silver nanoparticles in the device’s microchannel, the optical properties of the channel change. In particular, the channel turns from transparent to opaque. This causes intensity of light detected by the diode to drop down. A correlation between the detected light intensity and the concentration of Cl- gives the concentration of ions based on a pre-defined calibration.

Is our method sensitive enough to the small concentrations of Cl- in the concrete? We need to make sure that the changes in optical transmission upon Cl- interaction with Ag NPs are significant to be detected.

Considering a hole in the concrete of 1 cm diameter, we have calculated the amount of Cl- ions on the surface of the bottom of the hole to be about 2x10⁻⁵ µg when the concentration of Cl- in the concrete is critical (onset of rusting), see equation 1.

\[
\text{mass of } Cl^-/cm^2 = \text{wt}\% (Cl^-) \times r(Cl^-) \times \delta \tag{1}
\]

In this equation wt\% (Cl-) is the weight percent of the Cl- in concrete at a critical concentration (0.4% mass of cement), \( r \) is the ionic radius of Cl- ions and \( \delta \) is the density of concrete (~2500 kg/m³).

We considered the volume of liquid in the microfluidic channel to be 100µL, so the concentration of Cl- ions would be in the order of 10⁻⁷ M according to equation 2.

\[
[Cl^-] = \frac{m}{mw \times V} \tag{2}
\]

In this equation \( m \) is the mass of Cl- ions on the surface, \( mw \) is the molar mass of Cl- and \( V \) is the volume in the microfluidic channel.

Setting the initial concentration of AgNPS in the microfluidic channel similar to the concentration of Cl- as calculated in (2), we can calculate the optical absorbance, according to Beer-Lambert’s law, equation 3:

\[
A = elc \tag{3}
\]

Here \( e \) is the molar absorption coefficient of 10 nm AgNPs, which is in the order of 10⁸ M⁻¹cm⁻¹ (@ ~400 nm), \( l \) is the light path length, given by the dimensions of the microfluidic channel (0.2 cm), and \( c \) is the concentration of AgNPs. The resulting absorbance is in the order of unity. In terms of transmission of light, that attenuation of light is 90%, which will be easily detected by any type of photodiode detector. The advantage of this method is that the initial concentration of NPs can be modified to adjust the initial transmission of light. Moreover, with similar concentrations of Cl- reacting with the AgNPs, the changes in transmission will be considerable and measurable.

Scientific and Industrial relevance of the proposed solution:

Scientific
- A novel approach for detecting chloride concentration in concrete based on chemical reaction with Ag nanoparticles
- Our approach provides new possibilities for concrete research

Industrial
- Simple detection method and cost-effective construction method
- Individual components are well established in science and industry
3. Suggestions for next steps

The process for efficiently collecting the Cl- ions on the surface of the concrete needs to be optimized. A validation method needs to be designed. The change in concentration of the Cl- ions needs to be mapped to the measured change in intensity which shall enable a calibration of the hand-held device. The degradation of NP needs to be estimated with time so as to put a limit on the life-span of the PDMS device. Eventually we envision the whole setup to be portable and light-weight in nature. Also, the package should be robust to withstand the harsh environment of a construction site. For this, the packaging needs to be designed efficiently.

4. Approach followed during the workshop week

- Literature study and getting acquainted with the existing challenges, solutions and their limitations
- Preliminary experiments to better understand underlying behavior of concrete (measuring pH and concrete porosity)
- Derivation of the chemical equations combined with development of method for capturing chloride ions
- Physics of the setup (finding the operation spectrum and diodes with suitable sensitivity that is compatible with the PDMS channel)
- Defining the limitations of this approach and suggesting further improvements of the proposed solution

5. References

Caliper Sensor for Moving Wet Fabrics

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1. Description of company case

· Situation / background

Feltest provides measurement tools for the paper making industry with a focus on measuring properties of the press fabric, also called the felt. The function of the felt is to absorb water from paper fabric during the pressing of paper between the press rolls. Wearing of the felt leads to changing felt density and water uptake. As a result, the paper wetness changes, resulting in inhomogeneous paper roll thickness and ink absorption by the paper. These consequences are severe for the paper producer and require interventions that can be costly if not detected on time. Therefore, a felt thickness measurement would provide vital and timely information for paper makers so that actions can be taken to ensure constant paper quality. The measurement tools of Feltest must withstand harsh environmental conditions, such as high humidity, splashing water, temperatures varying from 10-50 °C, wideband sound and vibrations and varying degrees of dirt.

· Problem description / scientific challenge

The goal for this case is to propose viable sensing technology for measuring the absolute or relative felt thickness ranging between 1 and 3 mm thickness.

The requirements for such a sensing technology demand that it is: 1) handheld, 2) battery operated, 3) capable of measuring via the topside only, 4) able to measure 1 to 3 mm thick felt, 5) capable of handling an unknown quantity of (polluted) process water ranging from 200 to 500 g/m² water per 1200 g/m² fabric, 6) capable of measuring the full width of the felt (up to 11 meters), 7) capable of handling temperatures ranging from 10-50°C, 8) capable of measuring the felt line moving up to 40 m/s, and 9) capable of measuring with an accuracy of ± 0.01 mm.

· Research question

Can we develop a concept for a sensor to measure the (relative) thickness of in-use press fabric (felt) in paper mills?
2. Solution(s)

We came up with the following solutions to obtain an absolute or relative measure for the felt thickness:

2.1 A contact ultrasound device for measuring the relative felt thickness

Ultrasound, i.e. sound waves with frequencies higher than the audible limit of human hearing, is an established technology used to image or detect material surfaces by reflecting on them [1]. The time required for the reflected ultrasound to be detected indicates the distance between the probe and the reflective surface using a known value for the speed of sound, shown in Figure 1. The speed of sound depends on the density and bulk modulus of the media that is traversed. In our case, the unknown mixture of water and felt material makes a quantitative felt measurement difficult. However, qualitative measurements of felt thickness as a function of wetness and wearing may still be very well possible as shown by preliminary experimental results shown in Figure 2. We therefore suggest to perform ultrasound reflection measurements using a device in contact with the felt, to minimize environmental ultrasound effects. The wetness of the felt while in use, guarantees proper contact between the transducer and the felt. Given thickness variations of up to 200 µm, the ultrasound probe should work at a frequency of at least 26 MHz. By measuring the absolute thickness with a contact(-less) caliper at the edges of the felt, the absolute felt thickness can be determined. However, changes in the density of the felt introduce errors in the result. Accuracy significantly improves by increasing the number of measurements, i.e. by increasing the device sampling rate or by walking along the felt line several times. Figure 3 shows the result of applying these techniques to improve measurement error.

2.2 A non-contact optical measurement device of the felt thickness

Electromagnetic radiation, similarly to acoustic ultrasound waves, reflects at interfaces between different materials and thus is used to measure material thicknesses. These methods are based on differences in the refractive index of the material and the incident and exiting angle of the material as shown schematically in Figure 4. Depending on the position of the exciting light ray, an estimate can be made of the film thickness using a known or estimated refractive index of the felt. Considering the complex structure of felt fabric of which we show microscope images in Figure 5 and the chemical composition which is predominantly water and nylon-6 or -10 [2,3], i.e. organic polymers, we need to carefully consider the used type of radiation. Optical absorbance by the matrix and the presence of optical cavities, created by the cross woven yarns, with diameters of 100 µm, narrow the range in the probing wavelength to 20-100 µm. We took into account that felt thickness variations up to 200 µm demands probing frequencies in that range to ensure optimal refraction. Even with a suitable optical probing frequency, it remains cumbersome to provide a refractive index for the entire felt layer.

2.3 A (non-)contact magnetic resonance measurement of the felt thickness

An alternative promising measurement method that we did not explore in depth is magnetic resonance imaging (MRI). MRI is an expensive but established technology that is used to measure the water content of the felt, which is correlated with the felt thickness. Handheld MRI sensors are known on the consumer market that allow one-sided measurements [4].

2.4 A contact conductivity measurement of the felt thickness

Conductivity measurements of wet felt have been successfully performed by Feltest and have proven to give water content profiles similar to that of commercial microwave cavity water sensors. Measurements of the water content might be used to provide a profile for the felt thickness under the assumption that the entire felt absorbs similar amounts of water. Because the height of the water column is directly related to the felt thickness, using a conductivity meter calibrated with a layer of processed water of known thickness might provide enough information to determine the thickness. Furthermore, if the conductivity of the process water is known, quantitative felt thicknesses may be obtained.
A non-contact optical profilometry method on the press roll surface profile

We briefly considered a possible technology for assessing the surface profile of the press roll itself, which plays an important role in dewatering the paper and imprinting the felt. The theory that this method is based on has been investigated and has been shown to be achievable on rough surfaces of different composition [5]. However, this theory has to be evaluated for its applicability on the printing press roll and the practical implementation, in particular the accessibility restrictions of the machine with the measurement equipment.

3. Suggestions for next steps

We propose Feltest to further investigate the following issues:

1. **Ultrasound:** Performing accurate measurements requires a good enough signal-to-noise ratio. Therefore, understanding the background noise generated by the paper mill is a crucial step in proving the feasibility of the method.

2. **Ultrasound:** Better understanding of felt wear (density changes) and effects on the measurements is equally important in the product design.

3. **Ultrasound:** Once the concept has been proven extensively, the viability of market solutions for the ultrasound probe (transducer) should be explored [7,8].

4. **Optical:** The assumption that radiation will not scatter within the felt is optimistic, and scattering may still occur due to the felt’s highly irregular microstructure. Simple tests may show whether the assumption holds or whether an optical technique based on reflection is not viable.

5. **MRI:** MRI measurements of the water content need to be correlated with the thickness of the felt. Note that the conductivity measurement may potentially provide the same information, although the MRI measurement might be performed without contact.

6. **Conductivity:** The assumption of uniform water absorption over the entire felt layer may not be realistic based on different (mixed) sublayers of the felt. Furthermore, changing felt densities, resulting from wearing may also complicate this assumption. We do not see a solution to this problem.
4. **Approach followed during the workshop week**

Initially we inventorized all system parameters and subsequently brainstormed about viable sensor techniques to measure the felt thickness single-sidedly. Then, we focused our efforts on one technology, namely ultrasound detection, in order to better understand the underlying physical principles and to quantify the error by varying system parameters within realistic bounds. After focusing on the fundamentals of the technology we performed thickness measurements using a commercial ultrasound setup *Defelsko PosiTector 200 Adv* [6] on a wet piece of used felt. The aim was to determine if such a technique could measure anything on the felt at all, which it could to a certain but promising extent. However, the measurements could not be interpreted unambiguously and we decided to investigate the physical principles behind an optical measurement technique based on light reflection.

5. **References**


Appendix

- Figures

Figure 1: Ultrasound concept

Figure 2: (Left) Schematic with measurement points on a partially wetted piece of used felt for the thickness measurement using ultrasound. (Right) Preliminary ultrasound measurements showing a difference in behavior.
Figure 3: Simulation of a profile measurement including a random error of 10%. After applying a moving mean, the final measurement closely resembles the actual thickness.

Figure 4: Schematic of the reflection and refraction effects used by an optical measuring device.
Formulas for analysing the ultrasound signal

Consider the speed of sound in the material in terms of the travel distance and time,

\[ c_s = \frac{d}{\Delta t} \Rightarrow d = c_s \Delta t , \]

where \( d \) is the thickness of the felt and \( \Delta t \) is the travel time of the ultrasound through it. Because the speed of sound is unknown and depends on the lateral position, i.e. \( c_s(x) \), we measure at two neighbouring positions \( x_1 \) and \( x_2 \) that are located at a small distance from each other. The ratio of the felt thickness at the two positions therefore does to first order not depend on \( c_s(x) \):

\[
\frac{\delta(x_2)}{\delta(x_1)} = \frac{c_s(x_2) \Delta t(x_2)}{c_s(x_1) \Delta t(x_1)} = \frac{\Delta t(x_2)}{\Delta t(x_1)} \left( 1 + \frac{\delta c_s(x_2)}{c_s(x_1)} \right).
\]

This method allows us to calculate a relative thickness profile, assuming that variations of the speed of sound are small at neighbouring positions, i.e. \( \Delta x \frac{\delta c_s(x)}{c_s(x)} \ll 1 \), in which case the ratio is

\[
\frac{\delta(x_2)}{\delta(x_1)} \approx \frac{\Delta t(x_2)}{\Delta t(x_1)} .
\]
Other possible solutions that failed

We briefly considered thickness measurement methods of which we soon realized they won’t work, namely:

- Window-cleaner/car, due to safety problems regarding the device located on the opposite side of the accessible felt.
- Optical measurements with wavelength > 100 µm (as discussed in 2.2).
- Thermal measurements are ruled out due to the intrinsic low speed.
- Mechanical measurements that involve “hitting” or puncturing the felt as they can weaken and even destroy the felt at the high moving speeds.
- Optical diffraction measurements as no periodic structure exists in the felt as demonstrated by the felt structure shown in Figure 5.
Making Bubbles, Breaking Boundaries

A quantitative model for Cross Flow Membrane Foaming process

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1. Description of company case

1.1. Situation / background

Lattiz is a milk foam making machine commercialized by FrieslandCampina. The machine works based on membrane foaming process, which includes a cross flow of (hot) milk and pressurized gas. Milk is flown along the outside of a conical membrane and air is flown through the membrane from the center. The dead-end of the membrane pushes the gas to the side of the membrane, resulting in a cross flow between milk and gas. Currently, all required knowledge about the membrane foaming process is obtained by (lab scale) experimental methods.

1.2. Problem description / scientific challenge

The aim of this project is to formulate a quantitative physical model to describe the cross flow membrane foaming process, including the polydisperse interconnected membrane, in order to identify the steering parameters to obtain a good quality milk foam.

1.3. Research question

What physical phenomena occur at the membrane during foaming process, and what parameters determine the quality (size, stability, uniformity, etc) of milk foam in a cross flow membrane foaming process?

2. Solution(s)

Membrane analysis. The bubble formation occurs at the outer surface of the membrane. Therefore, it is important to analyse the membrane properties. We analysed the SEM image of the membrane outer surface using image analysis to study the pore size distribution (see Figure 2). A large variation in pore sizes exists on the membrane surface with a significant amount of pores with diameters above 10 µm (see Figure 3a) These large pores could possible dominate in the bubble formation process.

Bubble size measurement. Bubble size distribution at varied gas and liquid flow rate was plotted (see Figure 5 and Figure 6). It was observed that by increasing liquid flow, the size distribution broadened and shifted to smaller size. On the other hand, the gas flow rate did not affect the bubble size distribution at this regime.

Force balance on bubble formation. The bubble formation is modelled by single pore bubble formation and illustrated in Figure 1. As a first approximation, we assume that the bubble stays attached at the membrane until the fluid flow shear overcomes the pinning force arising from the interfacial surface tension. We find the surface tension contact force by taking a horizontal membrane with no shear, and balancing interfacial tension to the buoyant force detaching the bubble in this case (see Figure 1a). The buoyancy body force is given by $F_B = \Delta \rho g V$, and the interfacial contact force by $F_c(l) = f(y) l \sin \theta \hat{e}_t$, where $f(y)$ is a yet undetermined factor, $\hat{e}_t$ is tangent vector to the interface at the contact points by the pore, $l$ is the length of the interface at the contact with the pore aperture, and $\theta$ is the water contact angle. Taking the limiting case $\theta = \pi/2$, we obtain

$$f_{int}(y) = \Delta \rho g \frac{R_{eff}^2}{d_p}$$
With this result, we can then study the system of interest: vertical membrane with vertical milk flow (Figure 1b). Buoyancy is negligible in this case. Bubble detachment is then given by the shear force from the fluid flow overcome interfacial pinning force found above. We assume shear is given by a stokes drag type of force, given by [1]:

\[ F = k_x(6\pi \eta \mu v_f) \]

where \( k_x \) is the wall correction factor which is assumed to be close to one, \( H_B \) is the dimension or height of the bubble in the direction perpendicular to the flow, \( \mu \) is the water/milk viscosity, and \( v_f \) is the velocity of the fluid. From the force balance, we find \( H_B \).

![Figure 1: Schematics of (a) bubble formation from a single pore; (b) an assumed bubble geometry in the case of a horizontal membrane with pores of size \( d_p \); (c) an assumed bubble geometry for a single pore bubble detachment in a shear flow.](image)

Assuming an ellipsoidal bubble at detachment, we equate this to the one of the final spherical bubble. This way we determine the scaling of observed bubble size \( R_B \) with milk flow velocity \( v_f \) and with the pore size \( d_p \). Moreover, our model can also be used to predict membrane polydispersity, based on the resulting bubbles observed in the foam.

\[ R_B \propto v_f^{-2/3} \]
\[ R_B \propto d_p^{-1/3} \]
\[ d_p(R_{obs}) \propto R_{obs}^3 \frac{3c}{4\sqrt{3}\pi\Delta p g} \left( \frac{3c}{4\sqrt{3}\pi\Delta p g} \right)^2 \]

To test our model, during this week we conducted an experiment with the horizontal membrane system to determine surface tension. Using a 960 fps video, we measured a bubble of size \( R_{eff} = 675 \mu m \) at buoyant detachment. Using other parameter values provided by the company, we found a pore size of \( d_p = 10.3 \mu m \), which is in very good agreement with our analysis of the SEM image (see Figure 2).

3. Suggestions for next steps

3.1. Improvements of the existing experimental setup.

**Improving imaging.** High speed imaging would significantly alleviate the issue of unwanted bubble coalescence at the downstream part of the experimental setup. High speed imaging can be done either by replacing the existing camera on the microscope with for example an high speed USB camera (XIMEA) or using a mobile phone as we have demonstrated in this workshop. An alternative to high speed imaging is flashed illumination. Using an LED the illumination can be flashed to make a snapshot of the process.

**Automating the bubble counting.** The bubble size can be determined using OpenCV circle detection. However, developing the algorithms for processing the existing image stack can be problematic. Therefore, small tricks can be included while performing the experiments such as: 1) reducing the lens magnification; 2) changing the lighting to amplify the contrast between the edges and the centers; 3) taking a picture in focus and then taking a photo just out of focus followed by the image subtraction.
3.2. Experiments proposal

Reducing the gas flow rate. It will be interesting to investigate how the bubble diameter changes if we reduce the gas flow rate. This would also elucidate the minimum flow rate essential to achieve the same “quality” foam.

Varying the membrane length. We hypothesize that there are different mechanisms involved in the droplet depinning from the membrane surface depending on the relative position of the pore compared to the upstream and downstream extremes of the membrane (see Figure 7). Hence, it would be interesting to try experiments with varying the membrane length and observing if it alters the bubble size.

Exploring different membranes. Different membranes with different porosities and pore sizes with more controlled structure (e.g. trach- etched membrane, see Figure 8 should be explored.

Shear-cell experiment. Shear-cell experiment [2] can be performed to study various hydrodynamic forces acting on the bubble during growth and detachment (see Figure 9).

4. Approach followed during the workshop week

We started with a kick-off brainstorming session in smaller groups to generate more visualization of what phenomena may occur at the membrane during bubble formation. Afterwards, we came together to discuss and converge the ideas into one direction for the model and to analyse the data given by FrieslandCampina. We also did a small experiment to obtain more data to support our model. The details of our model and experimental data are given in a more detailed report which we handed to FrieslandCampina for their reference.

5. References


Appendix

A. Membrane image analysis

Figure 2: SEM picture of the membrane surface with detected contours.

Figure 3: Analysis of pore dimensions obtained from SEM image. (a) Pore size histogram with (blue) even bin size of 1 um and (yellow) varying bin size to emphasize presence of large pores, (b) Major and minor axis of the fitted pores.

Figure 4: Pore activity curve of dry and wet membrane.
B. Bubble size distribution plots

![Bubble size distribution plots](image1)

*Figure 5:* Normalized counts of bubble size distribution on: (a) varied liquid flow (gas flow = 20 NL/h); (b) varied gas flow (liquid flow = 0.7 L/min). WPI = 1%, milk density = 1002.3 kg/m³.

![Bubble size distribution plots](image2)

*Figure 6:* Mean bubble diameter on: (a) varied liquid flow (gas flow = 20 NL/h); (b) varied gas flow (liquid flow = 0.7 L/min). WPI = 1%, milk density = 1002.3 kg/m³.

C. Bubble formation mechanism

![Bubble formation mechanism](image3)

*Figure 7:* Overview of the process of bubble formation at the membrane and relevant forces acting on the bubble. Single bubble formation by shear force is depicted on the top. A possible situation in which more and more bubbles fill up the channel is depicted at the bottom. In this case the force exertion by bubble contact needs to be explored.
D. Proposed experiments

Figure 8: Scanning electron microscope image of a track-etched membrane.

Figure 9: Schematic of shear-cell setup used by Loubiere et al. 2004. Growth and initial rise of a bubble with diameter 1.5mm.
Speeding up the tyrosine crystallization in cheese

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1. Description of company case

• Situation / background: Cheese making consists of two main steps: first the milk is curdled by the addition of Rennet followed by introduction of bacteria which metabolize the milk lactose and proteins. Whereas lactose is metabolized fast, in a few hours, the degradation of proteins is much slower and takes place over months when the cheese is left for ripening. During the ripening period, the bacterial enzymes hydrolyze the protein chains into peptides and amino acids. Out of these, tyrosine is the most interesting as, over a period of time, it crystallizes in the cheese matrix giving an aged cheese a much desired crunchy texture. With currently used methods, the tyrosine crystals appear only after a minimum of six months of ripening. Therefore, speeding up the crystallization process will allow to reduce the storage time as well as the faster sale which is much more favorable from an economic point of view and is of a main interest in the cheese industry.

• Problem description / scientific challenge: The main scientific challenge here was to analyze all the successive steps in tyrosine crystallization and evaluate the influence of different factors on a particular step. The factors include the relation of crystal formation to the size and location of bacterial colonies, rate of protein/amino acid diffusion in the cheese matrix, the enzymatic activity of proteases and peptidases involved, the nucleation and growth of tyrosine crystals and the influence of confinement induced by the cheese matrix. Identification of the rate limiting steps will supply handles for accelerating the ripening process.

• Research question: How can we speed up tyrosine crystallization in cheese? And what is the rate-limiting step in tyrosine crystallization in a cheese matrix?

2. Solution(s)

• Improve the efficiency of the enzymes: By analysing the amount of tyrosine existing in crystallized form in a representative block of cheese, we found that only 12% of the total tyrosine present in the corresponding volume of cheese ends up crystallizing. One solution to speed up the formation of tyrosine crystals would be to increase the efficiency of tyrosine conversion from protein. The enzyme efficiency can be improved by increasing the temperature for a brief period within the temperature and time window where the other curtail properties of cheese is not affected.

• Decrease the size of the curd particles: Analysing the available confocal images and from literature, we found that the bacterial colonies and consequently tyrosine crystals appear preferentially at the interface/junction of two curd particles and the ‘eyes’ of the cheese. Hence, making the size of curd particles smaller would result in making more interfaces/junctions and hence provide more nucleation sites. In addition, more interfaces/junction would mean smaller distances between the bacterial colonies reducing the distances on which protein molecules need to diffuse.

• Addition of tyrosine crystals as seed particles: It appears that despite the availability of large amounts of Tyrosine, it still has a long induction time to nucleate. Hence, prior addition of tyrosine crystals in the milk as seed particles would likely curtail the induction time.
3. Suggestions for next steps

Based on the analysis done, we would recommend performing following experiments:

- To find the growth profile of tyrosine crystals by measuring the crystal sizes on monthly or fortnightly basis.
- To increase the number of proteolytic bacteria and check if the number density or size of crystal is increased.
- Increase the temperature, for a brief time, up to the extent that the other properties of cheese are not affected and see the influence.
- To add small Tyrosine rich peptides at the beginning to see how it affects the rate of crystal growth.
- Reduce the curd particle size for more inter-particle zones, which may increase the number of favourable sites for crystal growth.
- Study tyrosine crystallization in non-protein gels (e.g. alginate) to isolate the biochemical process of enzymatic action from the physical process of tyrosine crystallization.

4. Approach followed during the workshop week

We began by breaking down the crystal formation process into separate processes, according to the following scheme:

![Crystal Formation Diagram]

From this scheme, we initially looked at each of the steps separately. Using estimations from literature and back of the envelope calculations, we computed analytically the diffusion rates of protein and tyrosine in a cheese matrix and created a simulation for conversion of protein into tyrosine. Below is a table with some of the most important parameter values we found.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Estimated value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tyrosine crystal size</td>
<td>0.8 mm</td>
</tr>
<tr>
<td>Distance between tyrosine crystals</td>
<td>1.5 cm</td>
</tr>
<tr>
<td>Typical size of curd particle</td>
<td>0.9 cm</td>
</tr>
<tr>
<td>Percentage of total tyrosine in the crystals</td>
<td>12%</td>
</tr>
<tr>
<td>Diffusion constant tyrosine in cheese</td>
<td>3E-11 m2/s</td>
</tr>
<tr>
<td>Diffusion constant alpha-S1-casein in cheese</td>
<td>4E-12 or 3E-17 m2/s (depending on calculation method)</td>
</tr>
<tr>
<td>Initial concentration alpha-S1-casein</td>
<td>4.5 mM</td>
</tr>
<tr>
<td>Average radius bacterial colony</td>
<td>400 um</td>
</tr>
<tr>
<td>Protease activity outer wall bacteria</td>
<td>50 nmol/min/mg</td>
</tr>
</tbody>
</table>
For the first part of casein protein diffusion through cheese, we found two possible diffusion constants using two methods. One was to use the diffusion constant of dextran in model cheese. The other assumes that curd particles fuse after 5-7 days due to casein diffusion between the curd particles. With a curd boundary distance of 10 um this results in a much smaller diffusion constant (see table). The reality is most likely in between, as the diffusion constant will decrease when proteins are cut into smaller peptides and monomers. The very high initial diffusion constant may cause a lag in protein diffusion through the cheese and reaching bacterial colonies where enzymes can cut them into eventually tyrosine.

Estimating the number of proteolytic enzymes on the bacterial cell wall, combined with the number of bacteria on the outer surface of a typical bacterial colony and the typical activity of an enzyme resulted in a number for the amount of proteins that can be cut per second (1E10 proteins/s). This value was higher than the maximum calculated protein flux arriving per second (1.5E9 proteins/s for the faster diffusion constant or 4.2E6 proteins/s for the lower diffusion constant) at the colony surface, suggesting that the enzyme activity is not the rate limiting step.

Simulation of the cutting process of proteins into peptides and eventually tyrosine amino acids results in a plot with the time evolution of tyrosine molecules produced. In a situation where more proteins are available per enzyme, the shape of this graph will not change.

Considering the growth of the tyrosine crystals, we looked into the resistance of the cheese matrix. Since the matrix is dense the growing crystal needs to overcome a certain force, which is determined by the elastic modulus of the cheese. The force exerted by a growing crystal roughly relates to the surface tension of the crystal. The force balance between the two gives us the maximum size of the crystal to be around 40 um. Which is realistic considering that the observed crystals are not single crystals but clusters of many smaller crystals.

5. References


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Appendix

Monomers vs time

From Protein Source to Enzyme location

\[ P(x = 0, t = 0) = 0 \]
\[ P(x > 0, t = 0) = P_0 \]

\[ \text{Flux} = \frac{\partial P}{\partial x} = \frac{DP_0}{\sqrt{\pi D t}} \]
Fiber Bragg Grating-based accelerometer: shock protection

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1. Description of the company case

Somni is a company that produces Fiber Bragg Grating (FBG)-based accelerometers of high sensitivity in different-frequency ranges. The working principle of the accelerometers, depicted in Fig. 1, is based on the following mechanism; in a short segment of fiber, one creates a grating with alternative refractive indices \( n_3 \) and \( n_2 \). This allows efficient reflection of a particular wavelength \( \lambda \) defined by the period of the grating period \( \Lambda \):

\[
\lambda = 2n_{eff} \Lambda. \tag{1.1}
\]

All other wavelengths are transmitted (see Fig. 1). Stretching (compressing) the fiber changes the grating period by \( \Delta \Lambda \) and, subsequently, shifts the reflected wavelength by \( \Delta \lambda \).

![Figure 1](image1.png)

**Figure 1.** FBG working principle. Top: A fiber Bragg grating structure, with refractive index profile and spectral response. Middle: Refractive index modulation in the core of the fiber. Bottom: schematic representation of unstrained fiber spectral response.
The FBG is used in a classic accelerometer setup. This contains a (big) mass and a (loose) spring where vibrations are translated into movement of the mass. Using a fiber with the Bragg grating as a spring to which a mass \( m \) is attached allows creating a simple harmonic oscillator (see Fig. 2). The equation of motion for this system is:

\[
ma = -kx, \tag{1.2}
\]

where \( k \) is characterized by the stiffness of the FBG. In the simplest case, the displacement \( x \) is given by

\[
x = A\cos(\omega t + \varphi), \tag{1.3}
\]

with angular frequency \( \omega \) and amplitude \( A \) defined as:

\[
\omega = \sqrt{k/m}, \tag{1.4}
\]

\[
A = \sqrt{x(0)^2 + v(0)^2 m/k}, \tag{1.5}
\]

where \( x(0) \) and \( v(0) \) are the initial position and the initial velocity, respectively.

Since the acceleration is directly proportional to the displacement \( a = -\omega^2x \) and displacement determines the change of the FBG periodicity \( x \sim \Delta \lambda \), the acceleration in such a sensor is quantified in terms of the shift of the reflected wavelength in the FBG \( \Delta \lambda \).

![Schematics of a simple FBG accelerometer.](image)

**Figure 2. Schematics of a simple FBG accelerometer.**

It is seen from Eq.(1.5) that the sensor’s sensitivity depends on the attached mass. Increasing the mass \( m \), one can increase the sensitivity. However, according to Eq.(1.4), higher mass lowers the operating frequency, which is often not desired. To create a highly sensitive sensor that is working at a higher frequency range, engineers of Somni designed a proprietary transducer mechanism, which significantly amplifies the strain on the fiber by movements of the mass.

Following this strategy, Somni was able to create a correctly working sensor with 800 pm/g sensitivity and a resonance frequency above 350 Hz. Unfortunately, the sensor is not able to withstand the transportation and installation shocks. Acceleration movements stronger than 5g (this corresponds to 1 % maximum stretch of the FBG) result in irreversible damage to the fiber and (or) the transducer mechanism. Thus, the company tries to find a solution that can protect the sensor from being broken during the transportation and installation process.

**Solution 1: Locking with a permanent magnet.**

Detaching the mass from the spring and fiber during the transportation and installation process would solve the problem. The suggested scheme (see Fig.3) employs the magnetic force between the ferromagnetic end of the arm and the mass. To lock the system, the manufacturer screws the “lock-in” screw, which should detach the mass from the arm. When the sensor is installed, the customer unscrews the “lock-in” screw, and the mass is attracted back to the magnetic arm.
Figure 3. The schematics of the magnetic lock-in its (left) unlocked and (right) locked positions.

The detachment force should not exceed the upper threshold value that strains the fiber above 1%. This force corresponds to the maximum acceleration of 5g, which is 24.5 N. On another hand, the mass should not detach from the arm in the normal measuring range of ±2g, which corresponds to the lower threshold value of 9.8 N.

Commercially available NdFeB (Neodymium Iron Borate) magnets are suitable candidates for the locking mechanism. Having residual induction of 0.28 T, such a magnet [1] can generate a vertical pull force of about 22 N, for an area of 1 cm² (see the characteristics of the magnet in Table 1). The magnet has a Curie temperature above 310 °C, which means that it’s remnant magnetization remains stable (its vertical pull characteristics change no more than 1-2%) within the required working temperature range: -20 °C – +70 °C.

Table 1. Technical specifications of the commercially available suitable NdFeB magnet.

<table>
<thead>
<tr>
<th>Material</th>
<th>NdFeB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimensions</td>
<td>10x10x3 mm</td>
</tr>
<tr>
<td>Performance</td>
<td>0.28 T</td>
</tr>
<tr>
<td>Vertical pull</td>
<td>2.2 kg</td>
</tr>
<tr>
<td>Stability below</td>
<td>+80 °C</td>
</tr>
</tbody>
</table>

Solution 2: Shock damping with liquid

Here we explore the possibility of filling the cavity around the moving mass with a liquid. The idea is that the viscous drag with the liquid would damp the motion of the mass so the transportation shocks will not break the accelerometer. We know that the device breaks if the mass moves more than 16 μm which happens when it is subjected to an acceleration of 5g. Let’s say we would like to reduce it by 1g with the presence of the liquid.

When the mass (in the current design, \( m = 0.5\ k.g \)) moves in the vertical direction by a distance \( l \), it displaces a volume of liquid \( V = A_b \times l \), where \( A_b \) is the transverse cross sectional area of the mass. This volume of displaced fluid has to move around the mass to fill the void space. To do so, it is forced to use the narrow channels around the block, which we assume have a cross sectional area of \( A \). Therefore, using mass conservation for the fluid (assuming no compression), we can estimate the velocity of the liquid \( v \) to be

\[
v = \frac{A_b v_b}{A}
\]

where \( v_b \) is the average velocity of the mass. For the current sensor design, \( v_b = 40\ mm/s \).
To estimate the pressure difference we may use the Darcy-Weisbach equation [2] for the pressure drop $\Delta p$ of a liquid in a channel with length $L$

$$\frac{\Delta p}{L} = \frac{f_d \rho v^2}{2D}$$

where $f_d$ is the Darcy coefficient, $D$ is the channel characteristic dimension ($D \approx \sqrt{A}$), $\rho$ and $v$ are the density and velocity of the liquid, respectively. For laminar flows, the Darcy coefficient is given by $f_d = \frac{64}{Re}$, where $Re = \frac{\rho}{\mu} vD$ is the Reynolds number [3]. Putting everything together, we get the force exerted by the fluid to the mass to be

$$F = 32 \left( \frac{A_b}{A} \right)^2 \mu v_b L.$$

This relation provides some insight into the design parameters for this solution. In order to increase the pressure, we need to use a high-viscosity fluid or reduce the cross section area of the channels where the fluid moves in the device. We use the current value for the channel length $L = 40 \text{mm}$ to plot the force as a function of the area ratio $r = A_b/A$ for different fluids, depicted in Fig. 4. The horizontal dashed line in Fig. 4 corresponds to the fluid $F_m = 150N$ (corresponding to 30g) which will be ensuring that he sensor is not damage during transportation. With the current mechanical design of the sensor, where the cross-sectional areas ratio is $r \approx 3.8$ we see that even for glycerol at 4 C the exerted force by the liquid will not be enough. However, by making this ratio bigger we can achieve our goal. With a ratio $r = 53$ we get over the threshold even for glycerol at room temperature. This translates, for a block with $A_b = 40 \text{mm} \times 10 \text{mm} = 4 \times 10^{-4} \text{m}^2$, to an average gap of $= 75 \mu m$, which is within the current fabrication possibilities.

Further, whichever liquid is put inside the sensor will also need to be removed once it has arrived at its destination. Glycerol seems to be the best choice out of the ones considered here. It could be removed from the sensor by rinsing with water several times. After this process, residual water could be removed by drying with air.
Figure 4: Viscosity damping. Force exerted by the fluid to the block when the block moves with a mean velocity of 40mm/s as a function of \( r \), the ratio of the cross-sectional areas of the mass and the channel filled with fluid. Different curves correspond to different possible fluids. The horizontal dashed line represents the force 150N (30g), needed to prevent damage. The inset at the bottom left depicts a vertical view of the mass with rectangular shape and area \( A_b \). The surrounding light region represents the channel with cross-sectional area \( A \), where the liquid flows to fill the void space as the mass moves in the direction coming out of the drawing.

Solution 3&4

Another two solutions have been proposed and detailed out during the workshop which involved changes to the optical part of the transducer. The ideas in this direction aim to increase the sensitivity of the sensing mechanism allowing the use of a smaller mass and keeping the sensitivity of the accelerometer. Smaller mass will make the whole system less fragile, thus reduce the risk of breaking during transport (like for other modules already in production that have smaller masses). Preliminary tests have been carried out with rather positive results. Since these solutions are being considered for a patent, we cannot disclose the details here.

References

[1] https://www.first4magnets.com/rectangular-c35/10-x-10-x-3mm-thick-n42-neodymium-magnets-2-2kg-pull-p2441#ps_1-667

