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News from the lab'

DTI

At DTI, a system for measuring sorption isotherms has been established.
The system is comprised of a saturator, a sample chamber, and a dew point hygrometer.
Air is inserted into the system and runs through heat exchanger, submerged in a calibration bath. From here, the air is passed through a saturator system, likewise submerged into the same calibration bath. The temperature of the bath, T1, will equal the dew point temperature of the sample air, Td1.
From the saturator, the air is passed, through a heated tube, to the sample chamber, located in an oven, at a known temperature, T2. The sample chamber has a design, so the gas will pass through the entire sample, before leaving the chamber. This makes for a more efficient water transfer from the gas to the sample, or the other way around, compared to if the water was passing over the sample.
The pressure in the sample chamber, P, is measured, and the air is passed through a filter, f, ensuring that no sample contaminants are brought towards the dew point hygrometer and capacitive sensor.
At the dew point hygrometer and capacitive sensor, the dew point of the output air is measured, Td2. If the dew point of the analyzed air equals the temperature of the saturator, Td1, no net-absorption/desorption of water in the sample takes place, and the sample is in equilibrium with the surroundings. Once this is the case, the sample chamber is taken out of the oven and weighted on a precision scale.
This procedure is performed at different temperatures (T1 and T2), giving a sorption isotherm of the material

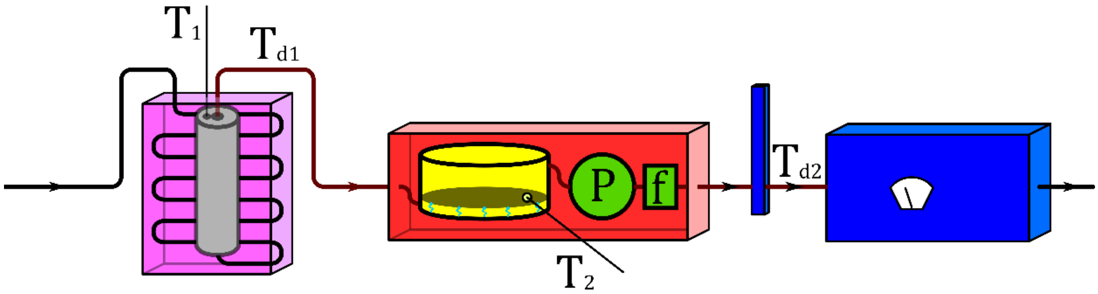

Figure 1 : Air is passed in to the system on the left. The air is saturated with water at a temperature T1 and passed through the sample. After the sample, the humidity of the air is measured using a dew point hygrometer as well as a capacitive sensor.

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Figure 2 : Sample chamber. The sample itself is placed on the filter in the bottom half of the chamber. Air, with a known humidity is introduced below the sample and retrieved from the top of the chamber. An entry for a thermometer is present in the design.

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INRIM

A new generation of high-temperature, high-pressure, humidity standards

A new kind of humidity generator has been developed at Istituto Nazionale di Ricerca Metrologica (INRIM, Italy) whose principle relies on a mass fraction vapour generator able to operate at dew-point temperatures above 100°C. The generator is based on a gravimetric ratio approach and a microwave resonator is integrated to that as a reference. This would provide SI-traceability for humidity measurements at temperatures up to 180 °C, dew point temperatures up to 140 °C and pressures up to 0.6 MPa (abs). The generator is based on controlled and SI traceable mixing of a water mass flow evaporated into a dry air mass flow. The generator operates over a range of absolute humidity from 25 g/m3 at atmospheric pressure to approximately 1000 g/m3 at 0.6 MPa.

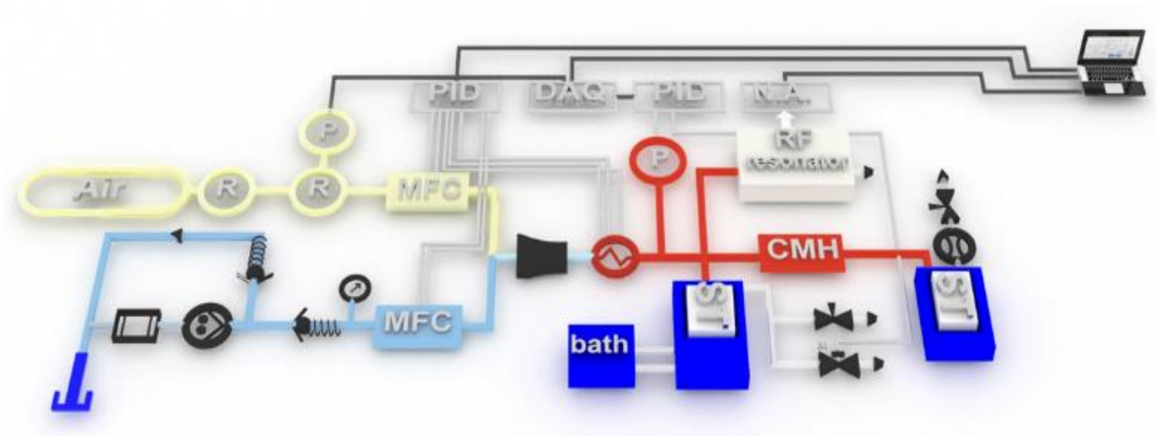

Figure 3 :

The vapour generator has been validated through absolute mole fraction measurements in the gas phase, by exploiting microwave-based hygrometry and by comparison against a primary humidity standard in the relevant dew-point sub-range. Microwave hygrometry is a well-known method able to detect the vapour mole fraction of binary mixtures with a high precision using the cavity microwave resonance frequencies. It exhibits a very high sensitivity due to the high polarizability of water and is well suited to cover the vapour amount fraction from 0.04 to 0.6 which matches the generator range. The water vapour mole fraction, as measured by the microwave hygrometer, and the specific humidity, as generated by the mass fraction vapour generator, were compared over a wide range of pressure and temperature.

Figure 4 :

The water vapour mole fraction, as measured by the microwave hygrometer (in blue), and the equivalent specific humidity, as generated by the mass fraction vapour generator (in orange), compared favourably over a wide range of pressure and temperature. It is worth mentioning that the relative uncertainty of the microwave hygrometer is about one percent in this range.

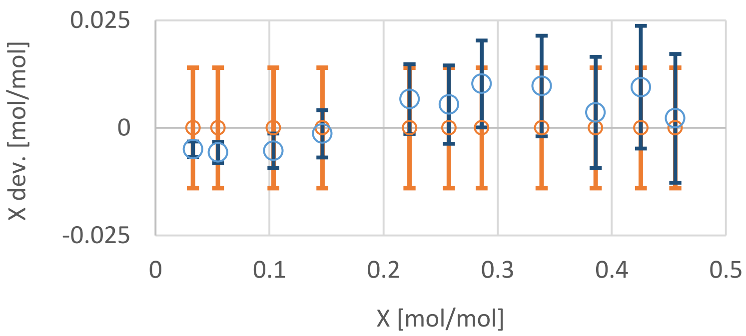

Figure 5 :

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UL
UL has performed the field measurements at the Novartis (formerly Sandoz) pharmaceutical company. The purpose of the measurements was to analyse the unwanted high humidity occurrences in transient condition within certain sterile Petri dish samples stacked in a large climatic sterile room. A new measurement approach was developed in order to enable a detection of the influence of microbiological processes to the transient humidity conditions within the samples. The new method is meeting the requirements on the high spatial resolution and accuracy within the sterile environment.

Figure 6 : Measurements of high humidity and temperature with sterile Petri dishes

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UNICLAM

The need for this research

Drying is estimated to cost European industry some 50 000 M€ per year (energy cost). Every 0.1 % improvement in drying efficiency due to better process control could save around 50 M€/year. Monitoring humidity in transient conditions and at temperatures up to over 100 °C is a key factor in controlling drying processes. Thus, by improving the reliability of these humidity measurements we can achieve annual savings of millions of euros in Europe.

Food industry is the second largest sector in the manufacturing industry in EU with total manufacturing turnover of over 900 billion euros. Drying and baking are key processes in this sector, and water activity – i.e. equilibrium relative humidity – is a key parameter in controlling quality of food and feed products. For determining water activity, material samples are taken from process and measured with laboratory analyzers. Significant amount of material is wasted because of slow feedback, and the optimization of energy consumption is limited by larger safety margins due to uncertainties increased by sampling errors and transient conditions.

The physics of food industrial processes combines heat and mass transfer with phase change in wet porous media and can impact the final cost and product quality attributes (such as microbial load, nutritional value, texture and organoleptic quality). The temperature and the moisture concentration can’t be considered as homogeneous variables. For example, during thermal sterilization of canned foods, the temperature close to the boundary of the can is higher than that on the center; as a consequence, the inactivation of pathogenic microorganisms will be less advanced in the center of the can. The design of the thermal process is, therefore, always based on the temperature course in that position which receives the least intense heat treatment. The difference between the bulk fluid temperature and the center temperature of food can be due to the low convective heat transfer coefficient between the fluid and the surface of the food or due to the low thermal diffusivity of the porous matrix (food). Therefore, the thermo-physical properties influence the design of any thermal process. The efficiency of the food manufacturing is strongly influenced by both boundary thermo-hygrometric conditions of the surrounding air (i.e. air temperature, relative humidity, air velocity, …) and thermo-mechanical food parameters including i) specific heat; ii) thermal conductivity, iii) density iv) geometry (i.e. thickness), v) permeability, vii) water percentage (moisture), viii) porosity. The knowledge of the moisture concentration and the moisture control techniques, could reduce deviations from the desired food quality, thus decreasing waste consequently guaranteeing high production rates. Many food properties depend on this parameter content, and large variations on its level can adversely affect post processing units. Then a reduction of moisture means a large economical saving for the mill.

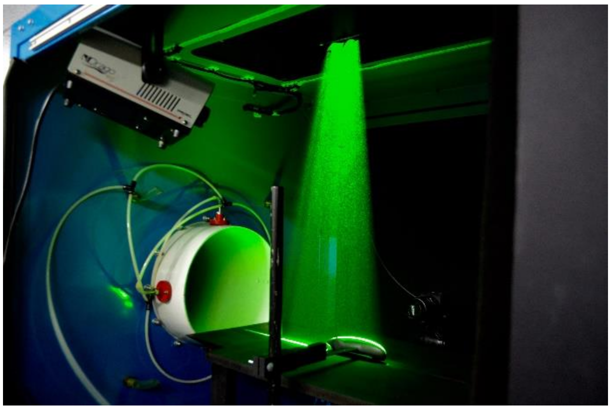
The impossibility of detailed moisture field determination inside a material via experiments makes numerical investigations very attractive for a better understanding of complex phenomena related to water and vapor transportation inside a material. The modelling of food processes allows analysis not only to understand such processes more clearly but also to control them more closely and make predictions.

UNICLAM experimental and numerical activities

Within the HIT project, UNICLAM will develop a tool for in-line water activity modeling and for a better understanding of the associated error sources.

Numerical modelling will allow: i) to understand the physics to explore new food processes, ii) to determine drying kinetics, time evolution of moisture content profiles, iii) to model the temperature dependence of sorption isotherms, iv) to model water activity and water mass fraction measurement processes.
The research unit activities are supported by the "Laboratorio di Misure Industriali" (LAMI), established in 1991, which proposes itself as a research centre of excellence in the field of industrial, scientific and legal metrology. In this area, the LAMI collaborates, at a national and international level, with primary metrology institutes (INRIM, PTB, LNE, NPL, NMIA) and with the Italian Ministry of Productive Activities in the field of legal metrology related issues. In the LAMI laboratory different research activity are conducted such as energetic, thermo-fluid-dynamic, heat transfer, thermal comfort, metrological, and environmental measurements. The metrological skills of LAMI involve mechanical (mass, length, force) and thermo-fluid-dynamic (temperature, pressure, flow rate, thermal flux, volume, humidity) measures. The metrological activity is focused on the design of complex measurement techniques and on the evaluation of their uncertainty budgets.
More in details, as concern the thermo-fluid-dynamic aspects, fluid flow in free, porous and partially porous domains are analyzed both experimentally and numerically. In particular, the flow visualization and velocity field measurement are possible thanks to laser-based Particle Image Velocimetry analysis .

Closed loop wind tunnel



Light diffusion from a laser beam

Figure 7 : Particle Image Velocimetry analysis (PIV system).

As regards the CFD approach, UNICLAM is able to perform numerical analysis by both non-commercial and commercial tools. A non-commercial algorithm, finite element method based, was developed and validated to study temperature, velocity, pressure and moisture profile in selected materials (such as non-woven fibers, polymers or pharmaceuticals) for single fluid phase, and dependence on material properties and state (such as thickness, vapor permeation and thermal/moisture exchange rate at the surface). Heat and fluid flow through free fluid, saturated porous media and across interfaces were simulated in forced, natural and mixed convection, and steady state conditions.

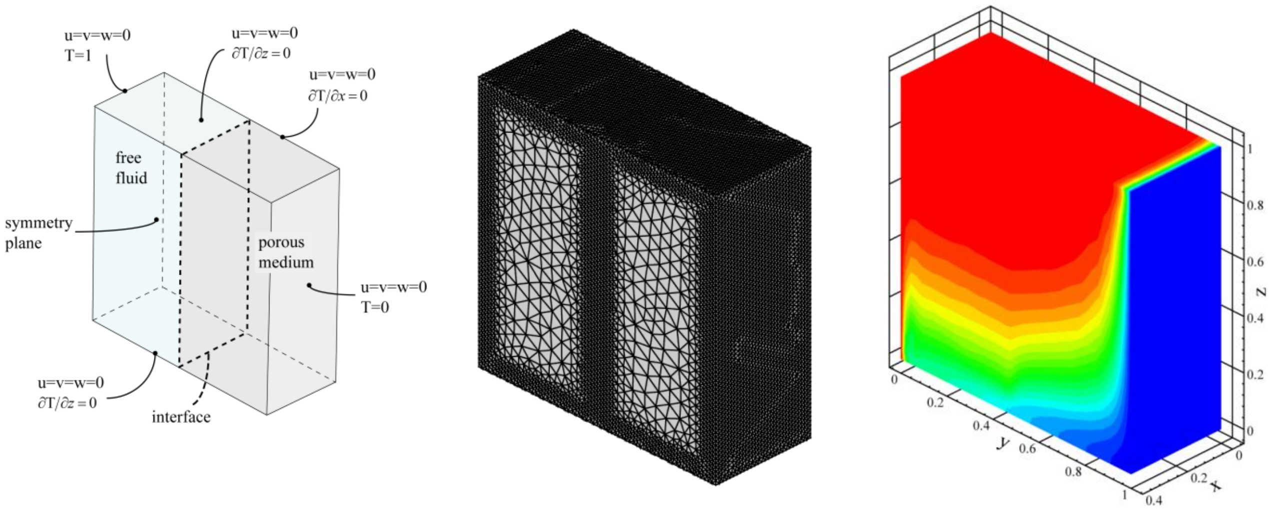


Figure 8 : The numerical results obtained for natural convection in a vertically divided cavity, with the vertical walls kept at different temperatures.

Thermal fluxes in the proximity of complex 3d systems like combustion chambers of waste to energy plants and 3d analysis of water removal from a cylindrical porous sample made of corrugated paper were performed by using commercial cod.

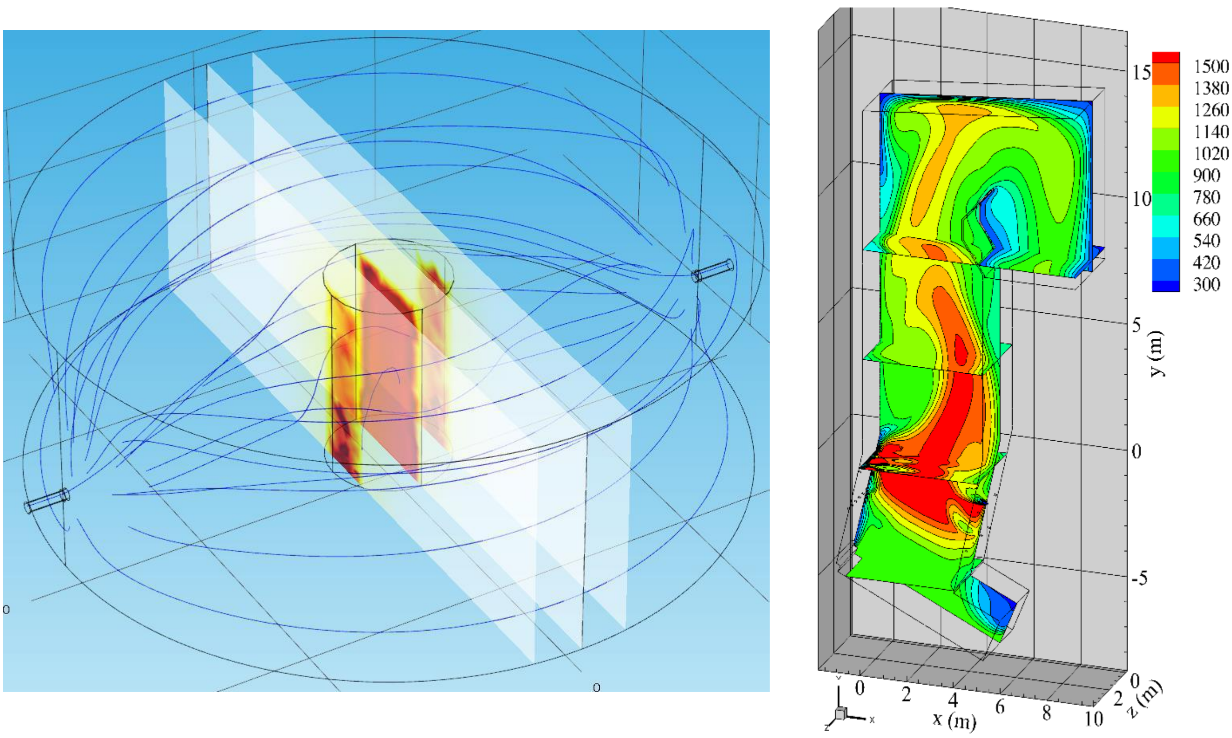
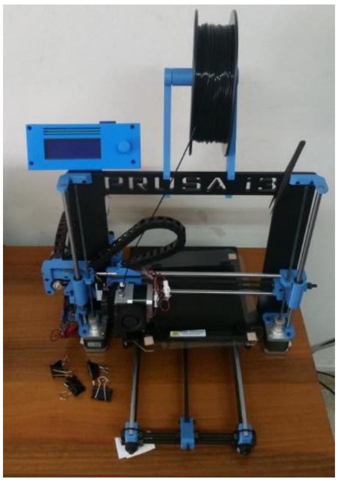
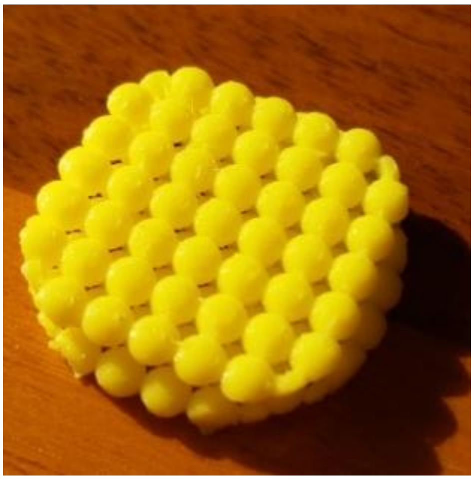


Figure 9 : Thermo-fluid-dynamic field obtained in a waste to energy plant (left), water removal from a cylindrical porous sample (right).

The validation of the numerical models can be performed by realizing prototypes supported using technologies such as 3D printing.



3D printer with a resolution of 50 µm



Plane of spheres (D=0.5 mm)

Figure 10 :

UNICLAM advances in the project

Numerical activity

The transient behavior of moisture-related phenomena requires a detailed knowledge of temperature, pressure, velocity and species concentration in the physical domain under investigation (e.g. at the surface, in the bulk, at the boundary layer). Such knowledge can be gained by developing a suitable detailed mathematical description of the porous regions in non-stationary conditions.
The main goal of the present WP is the development of a numerical model that predicts the temperature and moisture evolution in food industrial applications to take the necessary actions to improve and optimize performance to produce quality food at low operating costs. This approach implies the need to design a model from a compromise between simplicity and completeness. Numerical modelling will allow: i) to understand the physics to explore new food processes, ii) to determine drying kinetics, time evolution of moisture content profiles, iii) to model the temperature dependence of sorption isotherms, iv) to model water activity and water mass fraction measurement processes. In order to apply the model to real industrial processes, a 1D assumption was made and very simple equations were selected to predicts the temperature and moisture evolution in food by implementing the finite differences method. The proposed approach is based on Fick’s law with water vapor pressure and temperature as driving potentials. A model for hysteresis will be proposed and included in the calculations.

Experimental activity

In order to validate the numerical tool, UNICLAM realized an experimental setup for the determination of the absorption-desorption isotherms, in the temperature range of 20-80 °C, in selected porous materials. The equilibrium moisture content of the hazelnuts leaves at different temperatures in the range 0-80 °C is determined by a gravimetric technique, which is based on the use of saturated salt solutions to obtain constant relative humidity of surrounding air. Eight saturated solutions (LiBr, LiCl, KCH3CO2, K2CO3, KI, (NH4)2SO4, KNO3, K2SO4) were prepared by dissolving an appropriate quantity of salt in distilled water at a higher temperature then equilibration to ensure that they remain saturated when cooled. The experimental apparatus consists in the use of the different saturated aqueous salt solutions which, placed inside a glass jar, allow to obtain an environment with uniform and stable relative humidity. The glass jar is placed in an oven to control the temperature of the saline solution. The temperature control is achieved through a PID control system. The maximum temperature fluctuations in the oven are of about 1°C, while in the glass are of about 0.1°C.
An in situ chilled mirror dew-point hygrometer completely contained in the enclosure under test was installed. It covered a dew-point range from -40 to 60 °C, with an estimated reproducibility better than 0.15 °C, and an air temperature range of -10 to +60 °C, with an estimated reproducibility better than 0.05 °C, with a coverage factor equal to 2. Two PT100 four-terminal resistance thermometers, fixed with silicone, and an accurate data acquisition unit, with a 1-m°C resolution, were used. They covered a range of 0 to 50 °C, with an estimated reproducibility of better than 0.05 °C, and were calibrated with a PT25 transfer standard directly

traceable to national standards. Temperature measurements were carried out in such a way as to compare the solution and air temperatures and, consequently, to minimize the temperature gradient.

Figure 11 : Jar containing the saturated salt solutions and the analysed porous material (left), electronic thermos-balance containing the hazelnuts.

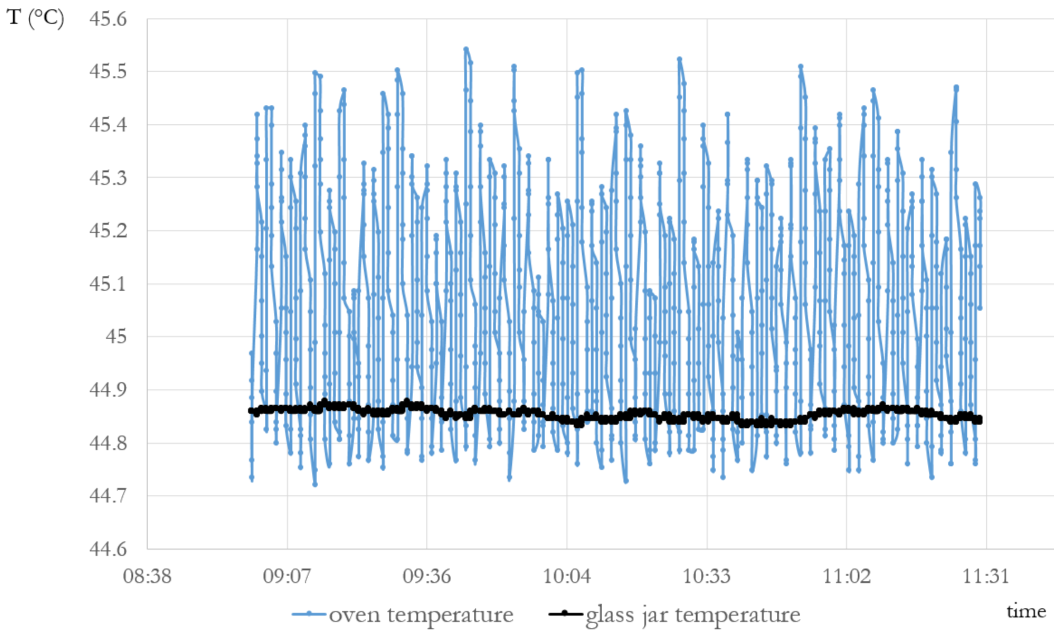


Figure 12 : Oven stability obtained by PID control. Temperature evaluated at the centre of the oven (blue line) and temperature evaluated within the glass jar (black line) as a function of time.

A layer of solid salts is maintained during the whole period of equilibration to confirm that the solution always remains saturated. A tripod was also put in the jar to place the hazelnuts samples. The saturated salt solutions allow obtaining a water activity ranging from 0.06% to 0.98% which is the characteristic range of relative hygrometers. The hazelnuts samples are weighted using an electronic thermo-balance (±0.001 g) (Figure 4) and placed in to the glass jar. Samples used for adsorption isotherms were dried 24 hours in the thermos balance until reaching maximum dehydration.

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VTT-MIKES

VTT MIKES is developing a new method for calibrating humidity sensors. The idea is to perform calibrations under non-static conditions and in this way reduce the calibration time by 50 % and still achieve an uncertainty of less than 2 %.

A humidity measurement chamber and humidification system based on mixing dry and humid air has been developed (figure 1). The mixing system allows fast and precise control of the chamber humidity in the range 10% to 90%. The chamber dimensions were carefully designed to achieve an optimum balance between response time and temperature stability. The whole system is placed inside a climate chamber in order to achieve a stable and adjustable temperature in the range from 5 °C to 80 °C. The humidity indications of the instruments under calibration are compared to a chilled mirror hygrometer which serves as a high precision reference.

Based on initial tests, the temperature uniformity and stability inside the chamber is better than 0.02 °C (figure 2). An example of a linear humidity ramp generated by adjusting the mixing ratio of dry and humid air is shown in figure 3. An optimum non-static calibration procedure, in terms of calibration time and accuracy, will be developed based on tests performed with different types of sensors.

Figure 13 : Humidity chamber with four relative humidity sensors and sampling tube (middle) connected to the reference hygrometer

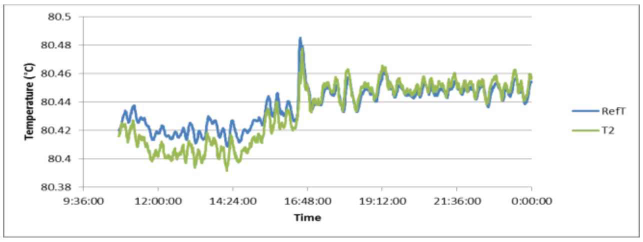

Figure 14 : Temperature inside the measurement chamber at a nominal temperature of 80 °C measured in the middle (blue) and in the corner (green) of the chamber.

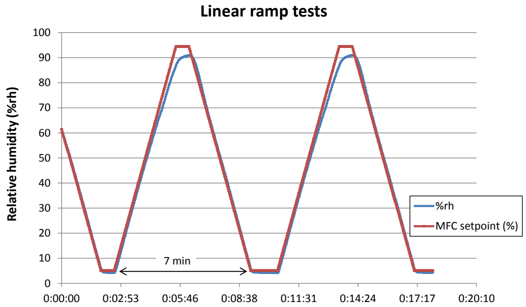

Figure 15 : Relative humidity inside the measurement chamber (blue) with different mixing ratios of dry and humid air.

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5 minutes with Tomi

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Short introduction of Tomi: he studied engineering physics at Helsinki University of Technology (now Aalto University). During his studies he worked in Laboratory of Biomedical engineering and built test equipment to characterize thin film high temperature super conductors. Year 2000 he joined Vaisala and worked couple of years as sensor test engineer and some time as Sensor development engineer in R&D. Currently he is working as Head of Production technology where his responsibility is development of calibration of Vaisala products (humidity, temperature, CO2, pressure, …), calibration equipment maintenance and production automation maintenance. He also lead accredited calibration laboratory in Vaisala. He has worked so far 16 years in Vaisala.
His team is involved practically all hardware related new product development in Vaisala developing production testing and Calibration equipment for environmental parameters. Vaisala's target is to produce high customer value with our products what comes to product performance, product features and overall quality. Short delivery times are also in our focus. In HIT project, we are interested on improving our humidity calibration cycle times to be able to serve customers faster.
Tomi has wife and two boys of age 10 and 7 years. His free time is filled with coaching kid's football team and driving the other one to trainings. The rest of the time he fills with sports like cycling around the year, basketball, downhill skiing and running.

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