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Innovative Training Network

TOMOCON

Deliverable Title

Detailed Plan for Virtual Demonstrations

Description

This deliverable outlines the plan and definitions for the virtual demonstrations, including used software tools, coupling of methods and tools including data protocols, definition of objectives, implementation and execution (e.g. web-based or local at one partner).

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Approved by Supervisory Board:		11.10.2018

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Revision Sheet

Revision Number	Purpose of Revision	Effective Date
0	Initial Issue	11.10.2018

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Detailed Plan for Virtual Demonstrations

This document provides a plan for the virtual demonstrations of the tomography-controlled Inline Fluid Separation, Continuous Casting, Microwave Drying and Batch Crystallization parts of the TOMOCON project.

Inline Fluid Separation

Background:

Gas-liquid separation is an important process for multiphase flow in the oil, natural gas and chemical industry. These industries have been developing separation systems and each time with a little improvement regards to split efficiency. The fundamental principal of working is dividing different phases in fluid mixture based on their density. One of these separation methods is performed inside of a pipe with a mounted swirl element. Instead of using gravity-driven separation of a multiphase flow the new method is using centrifugal force with much higher intensities than gravitational force. With sufficiently high fluid velocities, centrifugal force pushes the denser fluid against the walls of the pipe while creating a core of the less dense one in the middle. At the moment only stationary systems exist. Thus, the systems cannot cope with changes in the composition of the multiphase flows as changes in gas holdup and/or velocities. This leads to liquid carry over and/or gas carry under and decreases split efficiency. Especially the systems are not able to react in case of incoming slugs. Present and future industry standards demand a fully functional controlled system. To allow any automated control, fast tomographic sensor systems are required to derive control, parameters based on the current flow pattern and composition.

Experiments:

Initially experiments were made at the HZDR TOPFLOW test facility to identify major parameters influencing split efficiency for future development of a capable and efficient inline fluid separation system. Therefore, we were gathering information about the stability and dimensions of the gas core shape produced downstream of a swirl element by means of a high-speed camera. Wire mesh sensors have been utilized to characterize the upstream flow phenomena. With this setup several experiments with different superficial liquid and gas velocities have been performed. From those experiments first empirical relations between upstream flow and downstream vortex shape have been derived. As a result it has been found that the variation of gas holdup and mixture velocity mainly influence the stability and diameter of the gas core downstream the swirl element.
In the demonstration experiment we will utilize a set of two conductivity wire-mesh sensors (ESR 1) to quantify the cross sectional gas fraction and the structural velocity. Downstream of the swirl element the shape, stability and mean diameter of the gas core will be measured and characterized with TUL's ERT sensor. As a non-intrusive device, the sensor is capable of measuring volumetric void fraction without disrupting the shape of the vortex. The calculated parameters of data post-processing are sent to control devices and used to derive control values for the adjustment of the backpressure in the gas line and thus allow to narrow or widen the core to fit best to the inner diameter of the pick-up tube. However, total separation cannot be achieved and some of the water will be redirected to the air outlet and also minor gas portions can be carried over to the liquid outlet. The amount of this liquid carry over and gas carry under defines the split efficiency.

Based on initial thoughts with deduced conclusions our future setup will be a more complicated one and therefore a Supervisory control and data acquisition (SCADA) system will be

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needed with a complementary peripheral device such as programmable logic controllers (PLC) which will be connected to our main computer.

<u>CFD Simulation:</u>

Despite the widespread use of theories in fluid dynamics and sensor technology, empirical relations between measured and calculated variables are of high importance for future industry development. Most facilities or experimental rigs have a limited range of operation in which they can perform desired experiments. Sometimes cost and time are preventing further studies based on experimental results. To increase efficiency, we have resorted to computational fluid dynamics (CFD). Development has been made in making a processing model to predict certain results for automatic control using an original computational fluid dynamics (CFD) coupling a Eulerian model Volume-of-Fluid (VoF) method with a Lagrangian solver for bubbles/droplets resolution and an Immersed Boundary Method (IBM) for the separator description as a solid. The first part of using IBM for a complex geometry such as the one of the separator is already validated for single phase flow.

<u>Virtual demonstration:</u>

The virtual demonstration will be used to visualize and demonstrate the fundamental principles of a closed loop control system inline fluid separators. A schematic sketch of the planned demo is shown in Figure 1. All elements of the real flow loop, which have direct impact to the split efficiency, have to be modelled. However, pumps, mixers and large tanks are only needed in the real demonstration experiment. Instead, for the virtual demo the two phase mixture composition and velocity will be taken from prior experiments and three dimensional WMS data. The same data will be used to calculate cross sectional void fraction and mixture velocity directly. The behavior of the gas core downstream of the swirl element will be modeled from the high-speed camera data. The influence of the backpressure in the gas line towards the gas core diameter will be taken from CFD simulations. Since a complete FEM simulation of the EIT/ECT sensor would be required to simulate the electrical field distribution, a direct simulation of the measurement and image reconstruction is impossible for the virtual demo. Instead, the gas core diameter measured with high-speed camera will be used as the result from the tomographic unit. However, the measurement uncertainty, the latency and the frame rate of the tomographic unit should be included in the demo. A virtual PID-controller will be implemented to calculate control parameters for the valve to regulate the backpressure. This value is feed back to a look up table derived by CFD calculations to vary the gas core behavior. The complete virtual demo will be implemented in MATLAB® SIMULINK®. The demo should include gauges to show the current liquid carry over and gas carry under and enable interaction by modifying the current flow rates and flow pattern (bubbly, slug etc.). If possible an online version of the virtual demo should be implemented for public relations. For the data extraction and processing of wire-mesh sensor data the WMS framework will be used for obtaining upstream data sets and the WMS data processing unit. Dedicated framework will do the same for the ERT/ECT data processing unit. Downstream data sets will be extracted from digital image processing techniques in MATLAB® performed on images gained from high speed camera on regions of interest. Numerical simulations in 3D will be done using the CFD code JADIM developed at IMFT.

Project milestones are needed to mark specific points along a project timeline for successfully reaching the desired goals to achieve completion of the virtual demonstration for Inline fluid separation. The defined milestones are written below.

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Virtual demonstration milestones and dates:

M1. Define sensor and system geometry	15.10.2018
M2. Design a control loop in MATLAB/SIMULINK	10.02.2019
M3. Control loop is tested with virtual sensor and flow data	31.03.2019

In Table 1 the elements of the virtual demo with the responsible ESRs as well as realization dates are listed.

Table 1. Components of the virtual demo and realization time

Pos	*Components of the demo*	*Responsible ESR*	*Realization date*
1	Upstream data sets	HZDR	20.11.2018
2	Downstream data sets	HZDR	20.11.2018
3	WMS data processing unit	HZDR	05.01.2019
4	ERT/ECT data processing unit	TUL	05.01.2019
5	CFD simulation of back pressure effect	INPT	20.01.2019
6	Gas valve (including reaction time)	TUD	05.02.2019
7	Control unit	TUD	01.03.2019
8	Visualization of the demo including data sets	INPT	10.03.2019

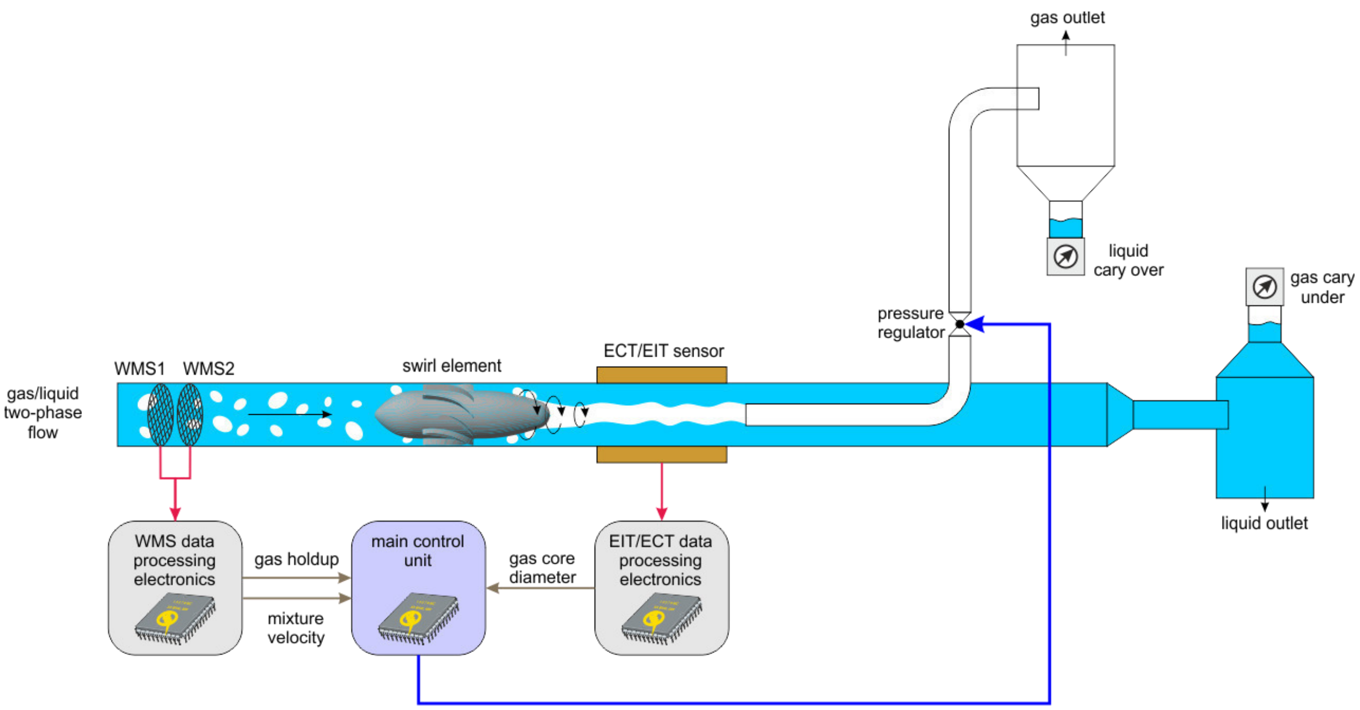

Figure 1. Simple schematics of two-phase flow loop for inline-fluid separation

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Continuous Casting

Background:

Continuous metal casting is an industrial process which produces 95 % of the entire world's production of crude steel and amounts in more than 1.5 billion metric tons per year [1]. In the production process numerous situations can occur that result in low or insufficient quality of the end product. Low quality means that the metal has to be melted and/or a new product has to be produced, resulting in additional energy and fuel consumption. From that, and the amount of steel produced yearly, it is obvious to conclude that any increase in process efficiency is beneficial. As stated in previously conducted research, the flow in the mold of the continuous caster influences the end quality [2]. Controlling and monitoring the flow in the mold and the submerged entry nozzle (SEN) of the continuous caster by using the CIFT sensor and combined ECT/MIT sensor is being developed. So far there are no systems and algorithms for this application, mostly it is because of the harsh environment, extreme temperatures and the opacity of the liquid steel. Tomographic data from CIFT and ECT/MIT sensors can be used to detect the actual state of the flow conditions, which may cause a defect. The measured flow condition can be used as an input for the control that could predict and prevent the production of the low-quality product and thus increasing process efficiency and reducing energy consumption.

Virtual demonstrator:

In the case of the continuous casting group, a virtual demonstrator is a reference to a joint simulation environment. The virtual demonstrator is divided in three major parts: (i) computational fluid dynamics (CFD), which will be done by ESR 5 at TU Delft, (ii) the two tomographic sensors contactless inductive flow tomography (CIFT), for which ESR 2 from HZDR is responsible, and a combined ECT/MIT sensor, which will be developed by ESR 12 at University of Bath, and the last part (iii) is the control system developed by ESR 9 at Technical University of Liberec. Due to the level of complexity and time required for the development of the numerical MHD solver for computational fluid dynamics, the virtual demonstration will be developed in two major phases. It was agreed within the group that the first phase includes only single-phase flow simulations, and the second phase will concentrate on multi-phase flow.

Figure 2. Data flow structure; Left - Phase 1; Right - Phase 2

[1] World Steel Association, "Steel Statistical Yearbook 2017," 2017.

[2] Pierre H. Dauby, "Continuous Casting: Make Better Steel and More of It!," *Revue de Métallurgie* 109, no. 2 (2012): 113–36.

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Data flow is shown in Figure 2. CIFT/MIT takes the data and the values from the CFD simulation, and uses it for a forward problem solver that calculates the values of the magnetic field that the sensors would detect (i.e. the magnetic field detected by the CIFT coils). The solution of the inverse problem is then calculated with the simulated sensor data, and the solution is further fed as an input to the control system. The control system uses the raw data as well as the reconstructions from CIFT and MIT/ECT to change the parameters and boundary conditions of the CFD simulation. The CFD simulation uses the new boundary conditions to create a new timestep of the simulation, and thus, the full circle of information is closed.

Data exchange will be done in the following way. After the CFD simulation for the specific timestamp is finished, it will be uploaded to the shared cloud storage. The CIFT simulation environment detects the new data present, extracts the needed information of the velocity field U, then runs the forward problem solution simulation, and from that the inverse problem is solved. Then, it will save data as a set of points described by the x, y, and z coordinates with appended reconstructed flow velocity information, and a separate data file with the raw sensor data. The control system will take the information from the data files created by CIFT, extract the useful information and feed it to the input of the control system, and it will save the output to a separate data file that will be used for the next CFD simulations.

Due to the lack of information for the identification of model parameters for the control loop, the first step of the first phase is to create a set of simulations for different boundary conditions representing different strengths of field of the electromagnetic brake for a single-phase fluid flow. The simulations will also cover the transitions between these steady states. It will be initially implemented as a five-state machine, in other words we will simulate all possible transitions between five different levels of the magnetic field of the electromagnetic brake. Furthermore, there is the possibility to run the simulations with three different flow rates in the SEN. This results in a total set of sixty simulations for system identification, and they will be also used as cases for the simple control loop that will verify the information exchange in a way that the controller selects optimal set of simulations based on the targeted angle of the jet.

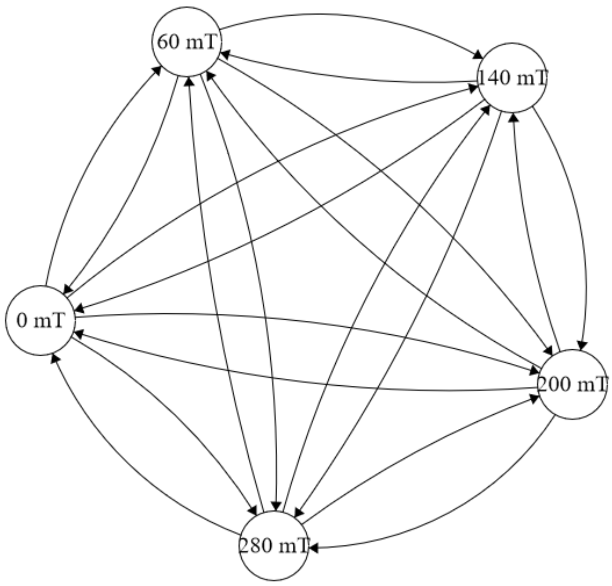

Figure 3. State machine with different levels of the magnetic field of the EMBr

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CFD simulations are performed with OpenFOAM which is a C++ toolbox for the development of customized numerical solvers, and pre-/post-processing utilities for the solution of continuum mechanics problems. The purpose of the numerical simulation, not only in the scope of the virtual demonstrator, is to develop a robust numerical model for two phase liquid metal flows under the presence of the actuating magnetic field of the electromagnetic brake. Upon successful implementation of the model within the virtual demonstrator, the extensive model validation will be done with experimental data from the Mini-LIMMCAST, and used for numerical support of the actuator design and control. To reduce the model complexity, it is divided in dedicated sub-models shown in Figure 3.

The CIFT part of the virtual demonstrator is written in C++ and Python programming language. The tools are handling the extraction of the velocity field from the CFD simulations to

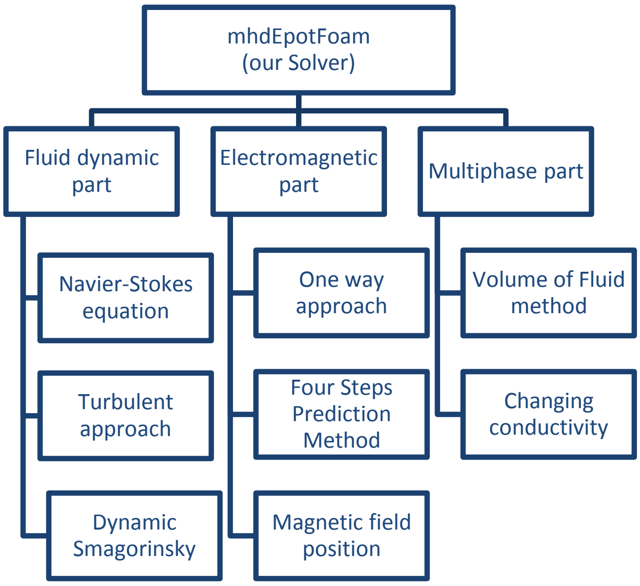

Figure 4. mhdEpotFoam solver

a coarser mesh, excitation field calculation and simulation, forward problem solver, inverse problem solver and various subsets of algorithms for data extraction and formatting.

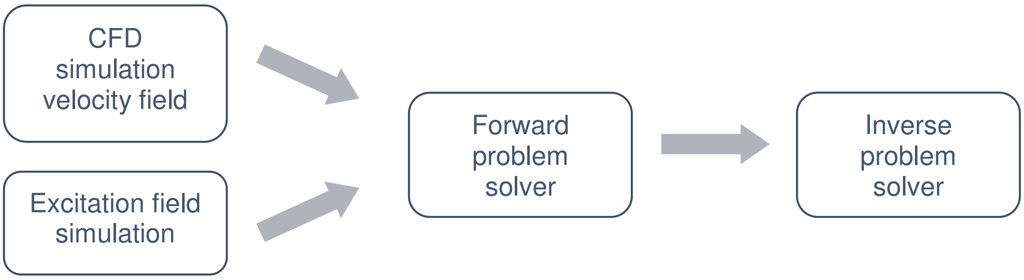

Figure 5. Data flow through CIFT simulation

As we approach to the second phase of the virtual demonstrator and extend the virtual demonstrator to multiphase flows, which will include the simulations from the MIT/ECT, the data transfer will be done in a similar way as for CIFT (Figure 5). Outputs from the MIT/ECT simulations will be also fed to the control loop in order to achieve optimal gas-liquid flow. Using the new information about the gas-liquid ratio, the CFD simulation will be started again.

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Control Loop

Controlled Variables:
Jet angle:

If the flow is single phase, an appropriate value of jet angle guarantees a desirable flow pattern. This angle will be kept controlled. However, the particular set point value of this angle will probably not be a unique specific value but some range of acceptable values. The jet angle can be determined both from CIFT and CFD simulations. Hence, it is possible to use system identification methods both on CIFT and CFD data and to check the correlation between the two.

In the case of multiphase flow, the value of the jet angle might not be the only objective to guarantee the desired flow pattern. The bubbles in the liquid might influence the path of the currents, which were induced under the influence of the EMBr, in such a way that the resulting Lorentz forces might change too. Under these circumstances, the behavior of the brake could be quite different to the case of single phase flow. The selection of the appropriate characteristics for control has to be determined once the CFD solver is completed and validated.

Manipulated Variables:
Magnetic field of the EMBr:

In the first phase, the magnetic field of the EMBr will be changed in discrete steps, because it is believed that the expected effect on CIFT could be easier compensated. Up to now, CIFT measurements were only performed, when the strength of the EMBr was not changed.

In the second phase of the demonstrator, the flow rate of argon gas will be a manipulated variable. The gas will be injected at the tip of the stopper rod. In this case, the complex two-phase flow in the SEN and the mold have to be calculated.

Conclusions

The virtual demonstrator is an essential tool to develop the control system and to optimize the process. The change of parameters of the simulations are much simpler, faster and cheaper to do than in the actual experimental setup, thus it offers a convenient way to test the new approaches for control. That makes the virtual demonstrator a tool that will be used during the entire duration of the project.

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Microwave drying of porous materials

Background:

Microwave technology faces growing interest in industry due to the possibility of volumetric and selective heating of dielectric materials. With that significant energy and time saving as compared to conventional, convective and radiative heating can be achieved. In the latter case heating of process materials is limited to the surface only and heat transfer into the volume is defined by the material's heat conductivity. Microwave heating has already proven its efficacy in many applications e.g. deindexing or sintering of ceramics, microwave assisted curing of carbon fiber reinforced composites in the automotive and avionic industries and others. Reduction of energy consumption of up to 70 % and processing time by about 50 % has been demonstrated. Due to the strong microwave absorption of water microwave assisted drying is a wide spread application of microwave in e.g. ceramics, food, chemical and pharmaceutical industries. Selective heating of residual moisture, within e.g. agricultural products or impregnated foams for heat insulations, is easily achieved by microwave heating. Non-uniform moisture distribution could be more efficiently addressed by intelligent control of distributed microwave sources. However, applying such a precise microwave control requires in-situ and non-invasive measurement of the unknown distribution of moisture inside the bulk.

Experiments:

TOMOCON targeted innovation: Within TOMOCON a concept of tomography-controlled microwave drying of porous materials, such as impregnated foams, shall be developed. An ECT sensor (UEF) as well as an MWT sensor (UEF) will be qualified for moisture distribution measurement and will be used to control microwave antennae power drivers (KIT). Technological challenges are the coupling of ECT electrodes either via contact using robotic actuators or contactless. For MWT a grand challenge is the derivation and solution of the inverse problem for limited data of a non-regular arrangement of only few microwave emitters. Here, extensive field modelling and development of appropriate iterative reconstruction schemes is necessary (UEF). Targeted are 10 Hz acquisition speed for bimodal measurement and < 1 s latency. The controller concept shall incorporate knowledge-based control and self-learning by making use of a data repository for different goods, geometries and process parameters (UEF, CTH). Multi-physics process modelling is used to predict microwave propagation, heat transfer and moisture removal by combining electromagnetic field simulations with CFD simulations for convective moisture removal using a porous body approach (KIT, UEF). The developed ECT and MWT sensor concepts will be implemented and validated on an industrial conveyor belt microwave system at KIT and tested for drying of polymer foams with real relevance to industry. The Demonstration will be performed at KIT's microwave laboratory HEPHAISTOS with a number of microwave processing and heating systems. The microwave lines there have an excellent technological level and highest flexibility for microwave system development, characterization and application. Different microwave systems up to walk-in ovens are available as infrastructure.

Benchmark target: The demonstration shall show a reduction of drying time of up to 25 % and quantify the associated energy savings.

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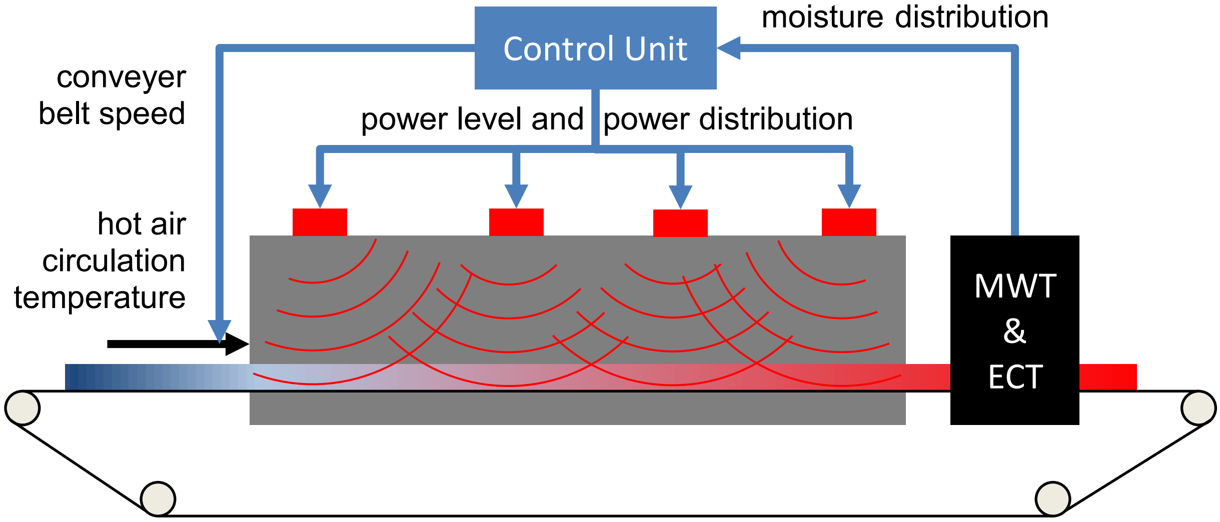

Figure 6. Simple schematics of the microwave assisted drying process

Virtual demonstration:

The virtual demonstration will be used to visualize and demonstrate the fundamental principles of a closed loop control system for microwave assisted drying of porous foams. A schematic sketch of the planned demo is shown in Figure 6. All elements of the real microwave assisted drying process, have to be modelled. However, since FEM models of the real HEPHAISTOS conveyer belt system including input and output tunnels as well as 18 slotted waveguide antennas and belt support structures, is too big, for the virtual demonstration a simplified design will be used (for example a closed segment of 1 m length with hexagonal cross section of 1 m circumferential diameter including 6 standard WR340 waveguide ports). Furthermore, for the MWT as well as ECT detector design to be used in the virtual demonstration, again a simplified model might be used although implementation of existing boundary conditions similar to the real experiments might be preferable. Simulation data will be used to estimate the moisture distribution of the foam using ECT sensor. This information can then be exerted to the controller as its input. In the following we will have a close look into each different task. The entire task can be separated to 7 subsections as follows: **1)** Microwave drying model **2)** MWT forward problem **3)** MWT inverse problem **4)** ECT forward and inverse problem **5)** MIMO Control algorithms **6)** Visualization of the demo including data sets. In the rest of the report it is tried to shortly explain each task. The complete virtual demo will be implemented in MATLAB® SIMULINK®. The demo should include (2D or maybe even 3D pictures of moisture distribution during time a Controller set-values for the microwave antennas resulting in a variation of field distribution within the microwave applicator). If possible an online version of the virtual demo should be implemented for public relations. In Table 2 the single elements of the virtual demo and the responsible ESRs are listed.

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Table 2. Components of the virtual demo and responsibilities

Pos.	Components of the demo	Responsible ESR	Realization date
1	Microwave drying model	ESR 7, 14, 15	30.09.2019
2	MWT forward problem	ESR 7	30.11.2019
3	MWT inverse problem	ESR 15	31.12.2019
4	ECT forward and inverse problem	ESR 14	30.04.2019
5	MIMO Control algorithms	ESR 14, 3, 7, 15	30.09.2020
6	Visualization of the demo including data sets	ESR 3	D5.3 - 31.03.2019

1 Microwave drying model

First of all, the foam (as a material under investigation) should be modeled as a function of moisture and temperature, this is because dielectric properties of a foam will change in each possible combination, see Figure 7. In this regard, we need to know the different method of dielectric characterization which some of these methods are already performed in the KIT. For instance, the following methods are employed for this purpose: 1) Transmission-Reflection method 2) Cavity perturbation method to name a few. Simulation of the drying process requires electromagnetic, heat transfer as well as CFD model by use of CST and COMSOL. Also, derivation of a fuzzy model for the system can be investigated in case non-model based control approaches are chosen to be employed.

2 MWT forward problem

The virtual model for measuring the scattered field as entitled “the forward problem” will be carried out by the CST software. In Fig. 8 a basic model of an oven can be seen. The dielectric property is $\varepsilon_r = 9.6 - j1.27$ $(tan\delta = 0.1333)$ which is obtained from Fig. 7. The dimension of the foam is $0.5m \times 1m \times 0.75m,$ its center is located right in the middle of the oven as shown in Fig. 8. Another model is created in the COMSOL software as shown in Figure 9.

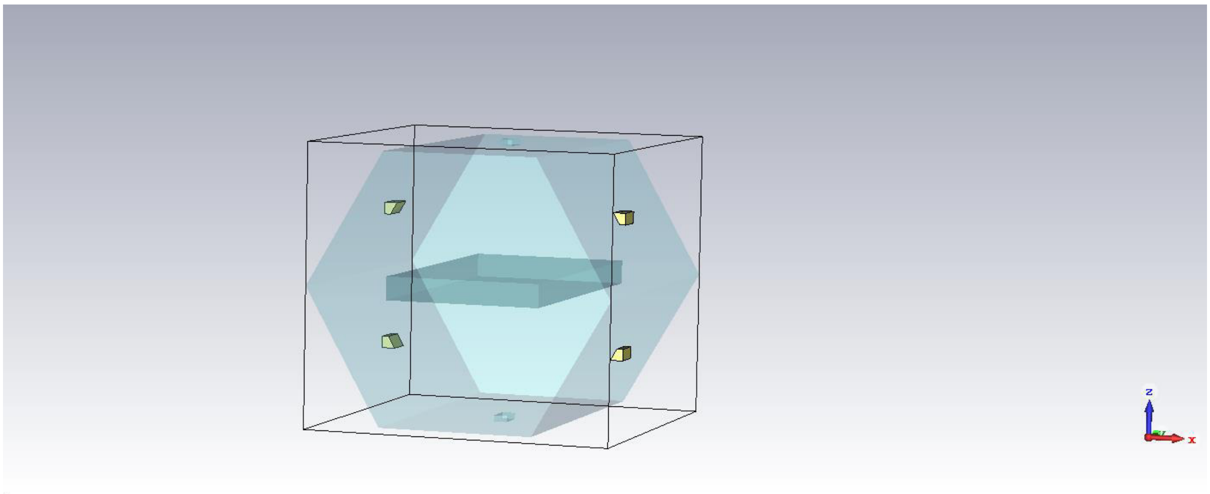

Figure 8. The Location and dimension of the foam in the oven, the model is created with CST software.

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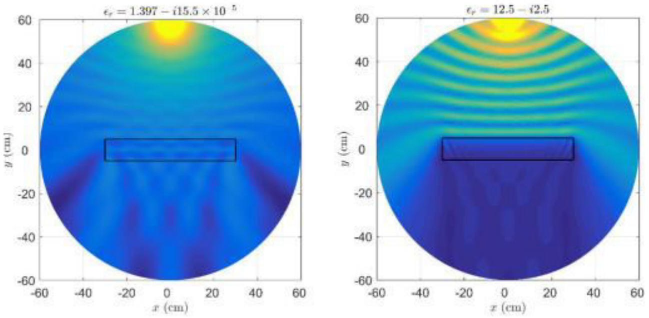

Figure 9. The medium, i.e. foam in the free-space is interrogated using dipole moment, after that a MDM matrix will be constructed for use in the imaging algorithm.

3 MWT inverse problem

Then, this data (S parameters) will be used in the MWT to obtain the moisture distribution (entitled “the inverse problem”), not only moisture distribution but also the effective relative permittivity of the foam (the nature of the quantitative methods). It should be noted that the MWT problem will be carried out using MATLAB to recognize the moisture level.

4 ECT forward and inverse problem

The forward and inverse problem of the ECT sensor are solved using MATLAB. Using numerical simulations, different configurations of this sensor can be tested to obtain the best and optimal configuration in reconstructing moisture distribution of the foam. Primary simulations are carried out using one of these configurations as illustrated in Figure 10. The reference permittivity distribution of the dried foam is equal to $\epsilon_r = 1.4$. The target positions are also shown in this Figure in which foam has a different permittivity ($\epsilon_r = 2.5$).

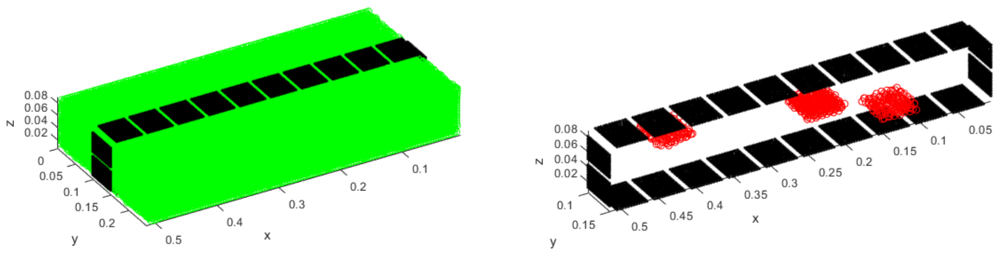

Figure 10. Sensor configurations. Black patches are the electrodes. Green patch is the imaging area and the red patches are the target positions with different permittivity with respect to the reference permittivity.

Employing the difference imaging method, 3D reconstruction of permittivity distribution can be done which is depicted in Figure 11. For the target positions we expect $\Delta\epsilon_r = 1.1$ and zero for other area.

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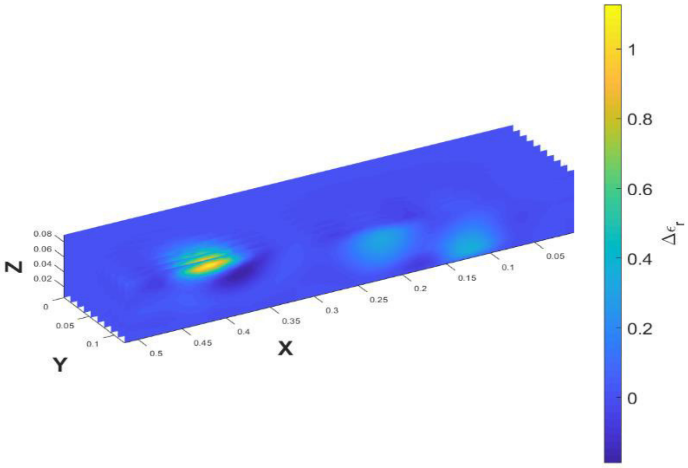

Figure 11. 3D reconstruction of permittivity

Considering a homogenous permittivity in y-direction, we can reduce the imaging area to a plane under the electrodes and do the 2D reconstruction. Figure 12 shows the result of 2D imaging with the same target positions as 3D.

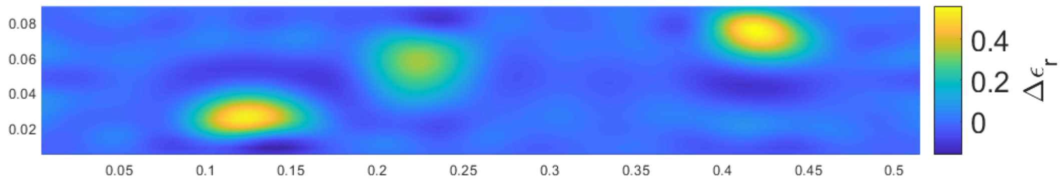

Figure 12. 2D reconstruction of permittivity

5 MIMO Control algorithms

The obtained data from MWT or ECT will directly be transformed to the control section to analyze the drying process and rearrange the excitation of antennas (i.e. power level) in a way to generate a non-uniform field for the drying process for the next round if required.

6 Visualization of the demo including data sets

The visualization part should be implemented among the whole process as simulated as a closed loop control system for microwave assisted drying of porous foams. For datasets visualization, (1) firstly, the moisture distribution obtained from MWT or ECT should be visualized for the decision making of the control section. (2) As a core part of the whole close loop control system, the datasets of control units should have various types visualizations corresponding to different types of data. For instance, the datasets of power level, conveyer belt speed and hot air circulate temperature should be collected and be transformed into intuitive forms. (3) The results of microwave drying of porous foams must be visualized via a straight-

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forward approach. (4) Finally, to evaluate results, visualization should also be utilized to obtain some diagrams offering explanation of the performance of microwave drying model. For the demo, visualization will be implemented in MATLAB Simulink or other effective tools.

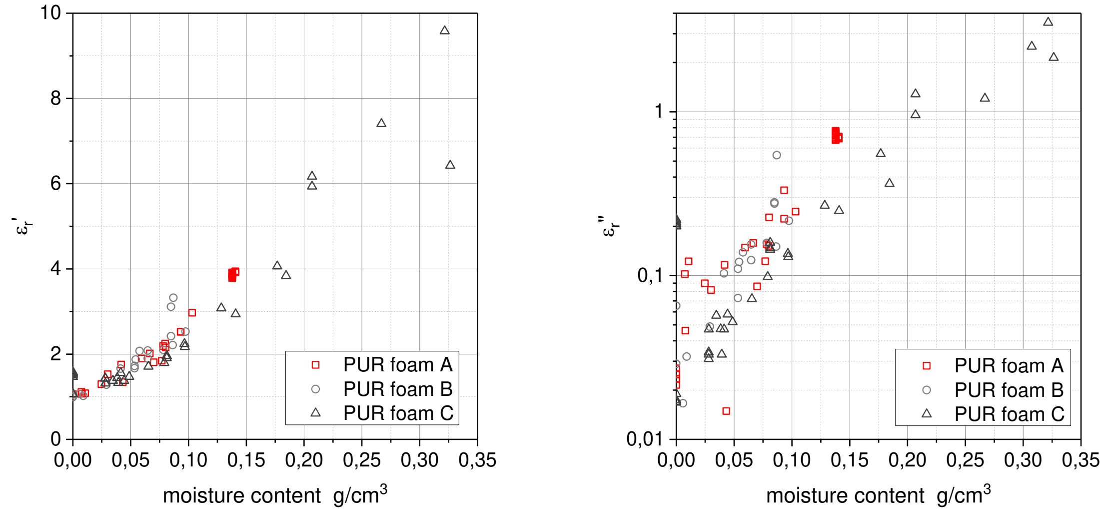

Figure 7. Dielectric properties of different grades of PUR foam as a function of moisture content at 2.45 GHz and room temperature

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Batch crystallization

Background:

Crystallization is a widely used technique in solid–liquid separation operation, purification technique and production processes. Crystalline products and crystallization touches every aspect of our lives. This single process combines both particle formation and purification and is vital to many processes like manufacturing of sucrose, artificial sweeteners, coffee extracts, pharmaceutical products, petrochemical industry, etc. Hence, control over the process of crystallization is required to obtain products with desired and reproducible properties and avoid expensive reprocessing steps. Process control has been in minor role in crystallization process design; an effective but under-explored way of crystallization process control is via robust tomography-based sensors and imaging instruments such as electrical resistance tomography (ERT) and ultrasound tomography (UST). However, simultaneous monitoring and implementation of feedback control of crystallization processes based on tomographic measurement (ERT/UST), hitherto, has not been reported and it has been limited to just monitoring of the processes. The combination of computational fluid dynamics (CFD) simulation, fast tomographic measurements sensors and feedback control systems offers a new approach and a perfect solution to ensure high product quality.

Experiments:

A batch wise precipitation process, as Initial experiment, was conducted in a lab-scale baffled stirred tank reactor (STR) with standard six-blade Rushton turbine. A peristaltic pump Masterflex L/S was used to maintain stable 2 mL/s flowrate of the reagent through a glass pipet (d = 1 mm) during 50 s of injection at several spatial points. At the end of each test, samples were taken for analyzing the crystals. Crystal size distribution (CSD) was measured by Laser Diffraction method in a particle size analyzer Mastersizer 3000 produced by Malvern company. ERT instrument were applied for monitoring local conductivities in the batch STR to reveal the spreading pattern of chemical reaction and precipitation at various operating conditions. As a result, the effects of feed location and mixing intensity have been found to influence the final crystal size distribution [1].

In the present work, lab-scale demonstrations will be carried out at the LUT crystallization lab. The specific target is to define the suitability of ERT/UST instruments for antisolvent crystallization of sucrose from ethanol-water mixture in a semi-batch mode. To the best of our knowledge, this is the first-ever tomographic real-time data acquisition of antisolvent crystallization, which will be further developed to test a new concept of control system.

Experiments will be conducted during 2018 with the commercially available measurement sensors such as, 1D Outotec LevelSense and 2D ROCSOLE system. Crystallization process will be conducted at various process conditions such as antisolvent addition rate, mixing speed and feed concentration. The lab-scale reactor size is from 0.2 L to 1 L. Standardized crystal suspension solution from smooth powdered sugar and coarse sugar crystals will be prepared at 4-6 suspension densities and 4 different impeller speed; these data will be used for validation and comparison of the acquired results from tomographic sensors. Data obtained from the tomographic measurement during the experiments will be analyzed by design of experiments (DOE) approach. DOE will be employed for screening the relevant factors that influence the process response. The initial process control parameters are fluid mixing intensity (ε) and antisolvent feed flow rate (F_e). These are the key factors to be considered in antisolvent crystallization processes, which have a profound effect on the supersaturation distribution and final product yield. Therefore, based on the experimental data from EIT/ERT sensors, a dynamic correlation will be made between these factors and crystal size distribution.

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As a result of these demonstration experiments, a connection between ERT/UST data points and initial process control parameters will be acquired. Dependence of crystal size and ERT/UST data on control parameters (F_e, ε) will be investigated. Crystal size distribution (CSD) measurement, sucrose mother liquid concentration, and crystallization yields will be measured as off-line and compared to the acquired results from tomographic measurements. This is due to the small volumes of the reactor at this stage, which limits the utilizing of on-line measuring instruments (e.g. FBRM). Additionally, crystal growth could be measured by instruments at DuPont. The data from experiments will be analyzed to get tomographic imaging correlation to crystal size.
Eventually, during the later stages of the demonstration, novel TOMOCON-targeted sensors (TUL, UOB) will be utilized and subsequent inline measurement and control of CSD will be conducted and compared.

<u>Tomographic sensors development:</u>

UoB

UST instrument will be developed jointly between NETRIX and UoB. We are aiming at developing multichannel UST device working in frequency range between 40 KHz to 1MHz with the possibility of multi-frequency UST reconstruction. The initial aim is to use the transmission mode UST considering imaging of sound velocity profiling and reconstruction. This is building on initial proof of principles developed by [1] that such an acoustic spectroscopy could provide valuable information for crystallization processes. To this end we are establishing steps in developing a multi-dimensional version of the work in [1] by using the UST method. Early developments are presented in WCIPT9 in [2]. Figure 13 shows our initial lab based tests, with several experiments in Figure 14 and reconstruction results in Figure 15 with a range of image reconstruction algorithm.

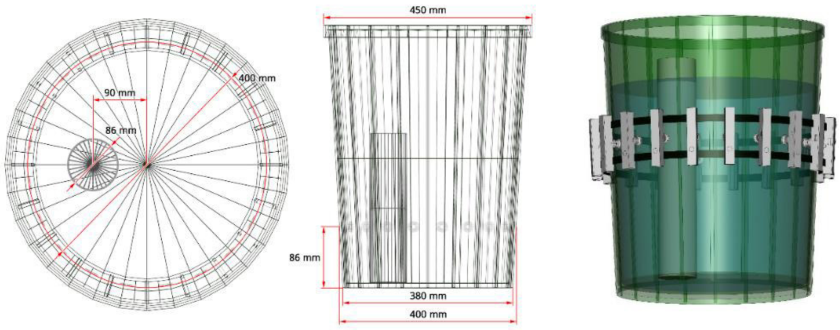

Figure 13. Experimental model

Dissemination Level: **Public**

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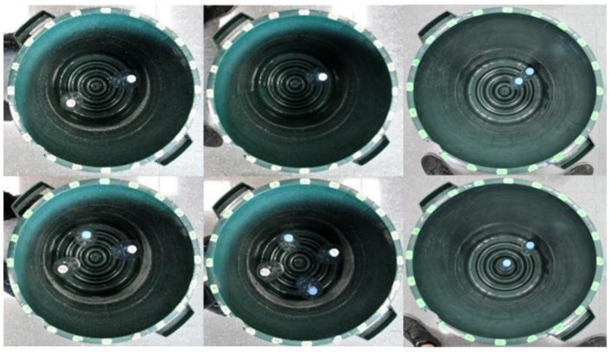

Figure 14. Experimental process

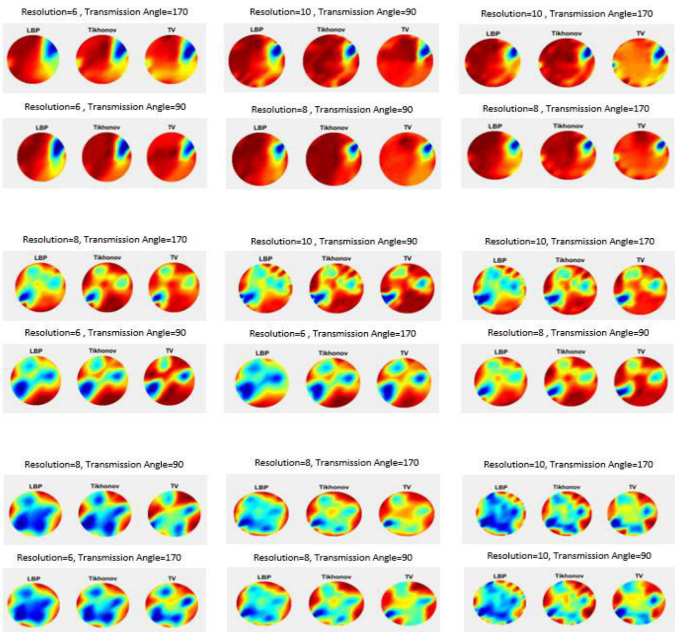

Figure 15. Reconstructed images from an experiment using a big tank and a transducers' ring system of 20 sensors with three different reconstructed algorithms (LBP, Tikhonov, TV). From up to down we simulate one, three and four bottles inside the liquid tank, respectively

The UST system is now being developed both in NETRIX and at the UoB, where further experimental works will be conducted in the near future before the industrial demonstrations. There has been extensive research in combining ERT and UST since the pioneering work of [3] in 2006. UoB will lead the data fusion and UST/ERT combination in close collaboration with TUL and subsequently in closed links with LUT to demonstrate such a multimodality imaging in lab based demonstration with modelling work in CFD.

TUL

Targeted sensor architecture (TOMOCON) would be developed at the Lodz University of Technology (TUL). The early experiments would be conducted on a 15 cm diameter reactor in 2018 followed by experiments on a 30 cm diameter reactor in 2019. The design would contain transparent/semi-transparent reactor vessel, copper pipes for hot water flow to con-

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trol temperature inside the reactor, servo-controlled stirrer, an inlet for anti-solvent addition and 3D-printed internal frame and baffle elements for sensor mounting and data acquisition. The sensors would be connected to an ERT computerized system. Two types of sensor architectures would be used to evaluate the progress of crystallization in the reactor. The ring architecture based on equidistant 6 sensors and 8 sensors circumferentially in 2-4 planes would be evaluated. The combination of the circular reconstruction and a novel vertical columnar reconstruction would be developed. Planar and 3D reconstructions would be performed using the centrally mounted sensors. Linear back projection (LBP), Landweber, and other image reconstruction methods developed at TUL [4] would be assessed and compared.

The initial observation would focus on the crystal distribution analyses based on the conductivity changes in the solution. Using solubility curve of sucrose at various temperatures the resistance values would be acquired and tabulated. Observation of real time changes post addition of the anti-solvent would be attempted thereafter. These observations would be compared to the images and results obtained using ultrasound transducers developed at the University of Bath. The effect of the high frequency ultrasound on the nucleation rates would be assessed using ERT modality.

Eventually at the later stages, a controlled crystallization would be targeted using feedback information from the reactor by controlling the temperature, stirrer speed and anti-solvent addition rate inside the reactor. For feedback control system purpose the fuzzy logic will be applied. It is due to their ability to perform various tasks in the similar manner as the humans think. The fuzzy controllers belong to the group of predictive controllers.

A comparative study of experimental data with respect to the CFD simulations would also be performed.

The status of a 15 cm diameter reactor with 6-sensor frame in August 2018 is shown in Figure 16.

Figure 16: Laboratory based crystallization reactor with 3-D printed frame to mount ERT sensors.

<u>CFD Simulation:</u>

Computational fluid dynamics (CFD) offers a new approach to understand the complex phenomena that occurs during crystallization processes inside stirred tanks. Coupling of governing conservation equations of a turbulent flow with integral equations of population balance and nonlinear algebraic equations of reaction kinetics makes the numerical simulation of a crystallization process a challenging task.

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For the present research, CFD simulations of antisolvent crystallization of sucrose from ethanol-water mixture in a semi-batch stirred vessel are in progress. The goal is to visualize the mixing of liquids with various viscosities and their coupling to local supersaturation. The Reynolds-averaged Navier-Stokes (RANS) equations in turbulent motion, along with the realizable k–ε turbulence closure model within the FLUENT 18.2 CFD software are employed. In the crystallization process, a population balance equation (PBE) can be considered to describe the changes in the crystal population, in addition to mass, energy, and momentum balance. Thus, it is possible to couple the transient Eulerian Multiphase Volume-of-Fluid (VOF) model and species transport equation with the PBE for studying the effect of solid particle distribution. Eventually, a numerical solution approach for the PBE can be quadrature method of moments (QMOM), which makes the crystal size distribution (CSD) evaluation (d_{32}) possible (Figure 17).

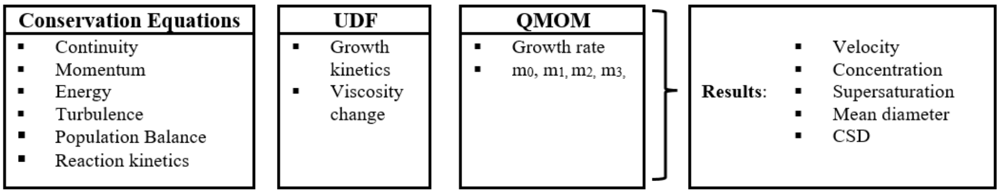

Figure 17. Solution strategy for CFD Simulation of antisolvent crystallization process

Coupling the PBE with the Eulerian multiphase model could remarkably increase the computing time of the simulation. Additionally, a large amount of parameters tuning are required to run a complete model for accurate results, which makes the assumptions during the process inevitable. Thus, for circumventing these issues, a series of user-defined functions (UDF) could be applied to the CFD solver. These user-defined functions, which contain the crystal size data from experiments, could be used for the calculation of crystallization kinetics. However, this approach to crystallization simulation needs further evaluation.

Demonstration:

The real demonstration experiment of the antisolvent crystallization process will combine novel TOMOCON-targeted sensors (TUL, UOB) and large-scale industrial reactor. A schematic sketch of the planned installation set-up of the demo is shown in Figure 18. During the initial experiments, we shall prove the feasibility and performance of controlling antisolvent crystallization process by using electrical tomography.

First-ever tomographic real-time data acquisition in lab-scale demonstrations will be carried out in the LUT crystallization lab together with TUL, UOB and support from DUPO with respect to later upscaling. At this stage, TOMOCON-based sensors will be tested and a crystal size dependent control parameter will be extracted from tomographic data analysis. Eventually, this parameter will be further used to develop a real-time tomography-based process control system for the antisolvent crystallization in order to run the process into desired product quality.

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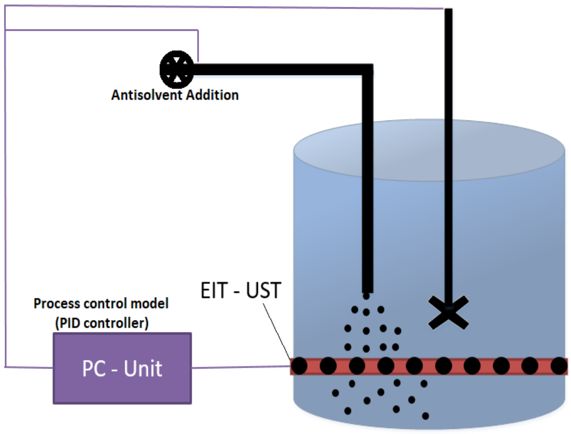

Figure 18. Antisolvent sucrose crystallization process unit; equipped with EIT/UST sensors and controller

Table 3 tabulates the responsible partners of the current demonstration.

Table 3. Components of the virtual demo and responsibilities

No.	***Components of the demo***	***Responsible ESR***	***Realization date***
1	CFD simulation antisolvent crystallization process	LUT	02-04/2019
2	ERT data processing unit and sensor design	TUL	01-02/2021
3	UST data processing unit and sensor design	UOB	03-05/2021
4	Control unit	TUL	03-05/2021
5	Visualization of the demo set-up and testing	LUT/UOB/TUL/DUPO	09-10/2021

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