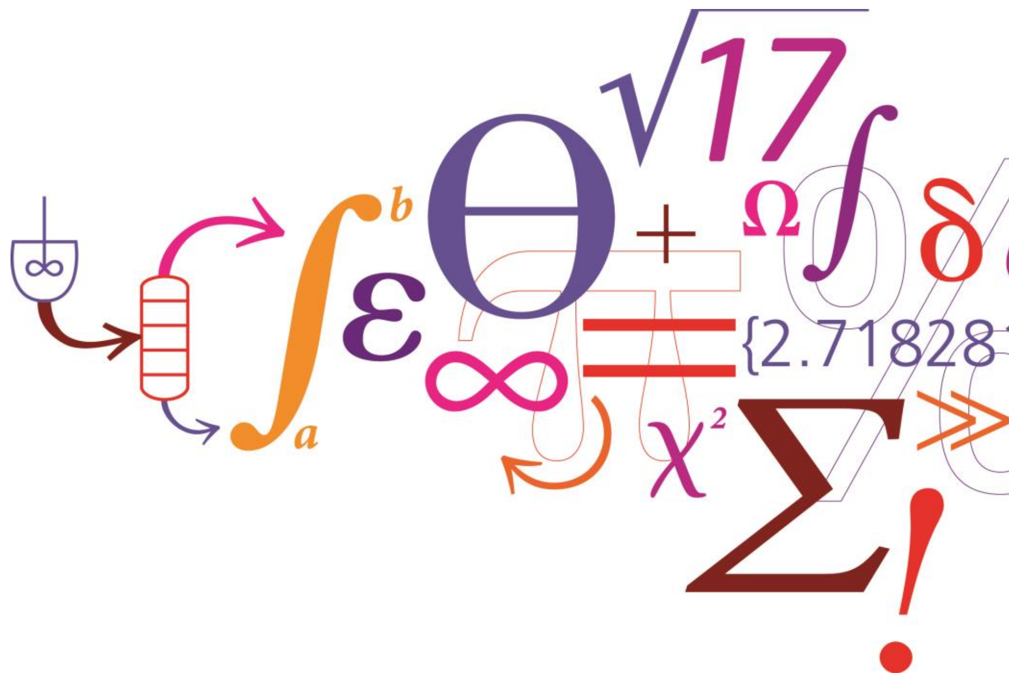
Chemical process technology equipment and lab courses

Pilot plant testwork combines theory and practise and simulate full scale behaviour.



DTU Chemical Engineering
Technical University of Denmark

Gross list of equipment/technologies

Distillation (batch and continuous)
Absorption, stripping and flow in columns
Drying (spray drying, fluid bed, tunnel drying)
Fluidbed coating
Filtration and membrane processes
Pump, liquid flow and gas flow systems
Liquid agitation (mixing) and aeration
Centrifugation, hydrocyclone
Solid and liquid extraction
Pilot plant fermentors
Organic synthesis
Fixed bed reactor (ion exchange, immobilized enzymes)
Evaporation and crystallization
Transport and separation of solids
High temperature processes (burner, catalyst etc.)
CO_2 absorber (simulated fluegas cleaning, CCS)
Process control experiments
Various mobile equipment, CIP equipment

Liquid flow in pipes

Any chemical engineer will experience the challenge of transporting a liquid through a pipe system having a number of different components. Ways of metering the flow, measuring and calculating friction pressure drops under various conditions can be experienced. In addition also non-Newtonian liquids and their flow behaviour can be tested.

Pump systems

This setup deals with the problem of matching pump behaviour with system demand. Changing pump speed and system restistance change the resulting operating point.
The concept of NPSH and cavitation, adapting pump performance to fluid properties, pump efficiency and determination the validity of the laws of affinity for centrifugal pumps can be experienced.

Agitation and aeration

Mixing of liquids is used in almost any process industry, but the theoretical understanding of the phenomena is rather poor. The setup allows for testing various kinds of mixing conditions and impellers – including rotating jet heads – using tracer injection to measure the blending time. PLC process control and touch panel is used for operating the plant.

The plant can also be used for determining mass transfer during aeration – a way to simulate an important aspect of fermentation processes. The hydrogenperoxide method is used for measuring $K_l a$-values.

Even CIP-nozzles can be tested in the plant.

Bubble column

In a bubble column a jet of air is introduced at the bottom of a liquid column as a mean to mix gas and liquid phases. The two phases can also be premixed in the nozzle in order to improve the K_la-value of the mass transfer, and also application of several nozzles in parallel is an option.

The bubble column reactor is used for study of two phase reactions and flow behaviour and to simulate for instance mass transfer in fermentation processes.

The hydrogenperoxide methode is used for K_la determinations.

Gas flow in pipes

Friction pressure drops across various components and different methods for measuring gas flow can be studied, and experimental loss coefficients determined for comparison with literature values. The validity of thermodynamic laws for gas compression and the Bernoulli flow equation can be justified.

Fan characteristic properties, flow profiles in the duct relative to varying Reynolds numbers and pressure drop across heat exchangers in various configurations may also be investigated.

Filtration in a filter press

Filtration is used in any industry to separate undissolved solids from the bulk liquid. Either the fluid or the solid may be the valuable product.

The plant includes a down-scaled industrial filter press with 6 cakes, a vessel with up to 250 liter suspension, circulation pump, instruments etc. The filter cake can be washed and air dried.

Results can be used to up-scale the plant to industrial level.

Rotary drum filtration

Rotary drum filtration is a continuous process – on contrary to many other filtration technologies. A disadvantage is the limited pressure difference which can be achieved across the filter cake – due to application of vacuum as the driving force.

The filter area is 0.5 m^2 and the cake can be removed either by a knife or using rollers.
The drum can be precoated and the cake washed to improved the separation.

Liquid/gas flow through packed columns.

In hydraulic design of counter current packed columns for mass transfer applications the loading points and flooding points are important figures, which can be determined by careful experiments. The flooding point and liquid hold up for beds with 3 different shapes of packings can be determined for various liquid-gas ratios.

Heat transfer

Next to liquid flow heat exchanging is the most important unit operation for the process industries. The test equipment comprises 2 long concentric pipe heat exchangers – each 4.5 m, which can be run counter current or co-current and with cold and hot water on either side. In addition the plant consists of a plate heat exchanger for steam.
Heat transfer coefficients and pressure drops can be measured and calculated. Also heat loss to the surroundings can be determined.

Distillation in bubble-cap tray tower.

This plant is used to separate 2-propanol and water in a continuous distillation at atmospheric pressure up to a concen-tration of appr. 70 mol % 2-propanol, where an azeotrope is formed at 80 ^{o}C. The column is about 2 m in height and has 9 physical stages. The steam heated boiler with 300 liter solution feeds the column from below. The condensate is partly refluxed and partly taken to the product vessel.
At equilibrium the composition of samples from every stage can be determined by a densitometer. Results allow calculation of he actual number of stages according to the McCabe-Thiele method.

Batch distillation

Many industrial distillations are performed batch-wise. This plant is designed to separate a batch of max. 100 liters using a column with a structured packing. The distillation capacity is 30-40 kg/h. Based on a simple alcohol-water separation it is possible to determine the number of stages, mass balance, minimum reflux ratio, heat efficiency, pressure drop etc. and compare with theory.

The transparent glass equipment provides a chance to visibly follow the process.

Absorption in packed columns.

Cleaning of polluted gases in an absorber - or scrubber - is an important technology for environmental protection, and chemicals are often used to enhance the absorption, as for instance in flue gas desulphurization on power plants.

This 4 m high column filled with Raschig rings is used for absorption of ammonia from air using water. The number of transfer units or absorption stages can be determined by means of samples taken from various levels in the absorber.

The plant also has a small column for humidifying air and another for post absorption.

Drying in a tunnel

Drying of chemical products is often necessary to ensure stability during handling, storage and further processing. In this simple tunnel dryer a batch of wet product is dried on trays using hot air with the possibility to vary the temperature as well as the air flow. The trays are directly connected to a balance, and the weight is together with the air humidity and temperature continuously recorded on a computer. From the drying curve and the process conditions the drying behaviour of the product can be calculated.

Spraydrying

This continuous drying method is applied in many different industries. The unit operation is a good example on the concept of particle technology and may encounter aspects like drying time, droplet size and product size distribution, application of Mollier H-X/psychrometic diagram, drying efficiencies, cyclone cut size and analytical techniques for characterization of powder material.

This Niro/Gea Production Minor pilot plant can be operated with different atomizers under various conditions.

Fluidization and fluid bed drying

This small batch setup is applicable for studying particle fluidizing behaviour such as minimum and terminal fluidization velocity, bed expansion and voidage, particle wear, slugging, elutriation etc.

Drying processes can be performed and the drying rate and critical humidity determined.

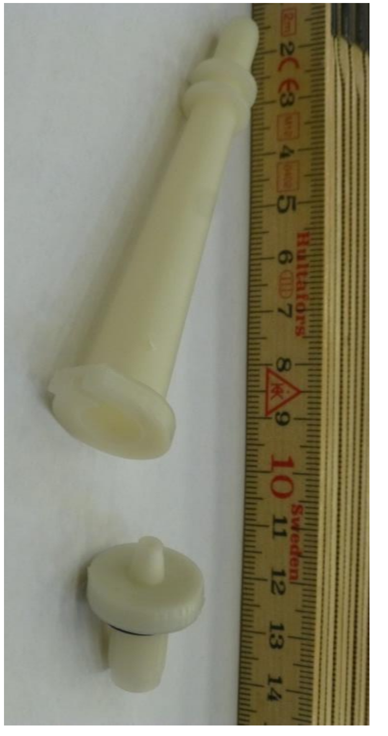
The plant is designed with expanded freeboard and exchangeable constriction plate.

Centrifugation

The disc-stack centrifuge has two separate liquid outlets and one outlet for ejected sludge, so it can be used as a separator as well as a clarifyer.

The plant is suitable for learning how to operate a ”separation-by-density” machine and determine the operation mode, work with Stokes law, calculating settling times and scale-up to industrial size.
The equipment can also be used to highlight the concept of hygienic design.

Hydrocyclone separation



Hydrocyclones are quite small compared with gas cyclones, but yet able to separate small particles from a liquid with high throughput.
As a teaching setup the plant demonstrates the separation of 20 μm potato starch particles from water in exactly the same way as in the industrial application.

Liquid-liquid extraction

This process concerns the transfer of a solute from one liquid phase to another. It can take place using a mixer-settler unit in which the liquids are emulsified into each other. The mass transfer is dependent on the fluid flows and the mixing speed, which also controls the kind of emulsion (water-in-oil or oil-in-water) being formed. If surface active agents are present the simple extraction can be very complicated due to difficulties in separating the emulsion into the two immiscible phases.
The plant comprises 4 glass vessels each 100 liter, pumps, stirrers and mixing unit.

Solid-liquid extraction

This process concern leaching of a solute from a solid. One well known example is coffee making. The plant is designed for batch extraction of pectin from citrus peels using hot acidified water. The capacity is 20 liter of water. The extract can be separated from the solid residues using a basket centrifuge or a filter.
When used for pectin the product is precipitated from a the clear extract by mixing with alcohol followed by filtration and drying.

Crystallization

The plant is a down-scaled version of a commercial sugar crystallizor, but the application is not limited to sugar. In the equipment a batch of appr. 300 kg solution of the product of concern is concentrated by evaporation under vacuum and further crystallized. The process is controlled using a camera-microscope connected to a monitor.

The equipment also includes a vessel with steam jacket for feed preheating, feed pump, vacuum pump with a condensor, temperature, pressure, level and conductivity indicators and 4 independant heat transfer surfaces in the boiler.

Evaporation

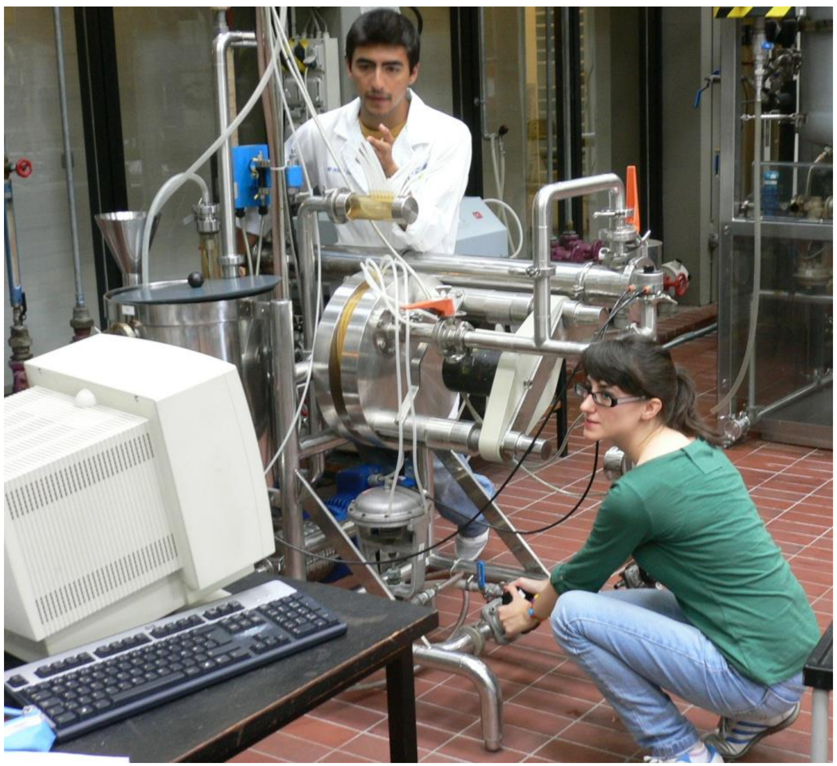
The equipment is a continuously operated falling film evaporator with full datalogging, computer touch screen, separate condensor, vacuum pump, CIP nozzles etc. The evaporating capacity is up to 100 kg of water/h.

The heat supply can be hot water or steam, and the plant can be operated from vacuum (~ 0.1 bara) to over-pressure.

The routine teaching application deals with concentrating whey permeate, a residual product from cheese manufacturing.

Membrane separation by ultrafiltration

One of the important membrane processes is ultrafiltration, used to separate polymers/macromolecules from smaller molecules and to concentrate by water removal. The pilot plant is equipped with two different kinds of membrane – a plate-and-frame system and a spiral-wound type.



Membrane properties such as retention and permeability and process parameters like osmotic pressure, permeate flux and concentration polarization can be studied. Also mass transfer relative to various operating conditions can be determined.

CIP-cleaning procedures are introduced when handling mem-branes.

Reverse osmosis

The plant is equipped with a spiral wound membrane module and designed for desalination purposes. A multi staged centrifugal high pressure pump can deliver more than 25 bar at high flow.
The membrane has a high salt retention and is able to produce clean water at low conductivity.
The pump, membrane and vessel modules are designed as mobile units.

Fixed bed reactor

The plant is designed for multipurpose applicability for any kind of low temperature/low pressure fixed bed reaction. At present it is used for immobilized enzyme catalysed conversion and for ion exchange operation, but many other chromatographic processes can be performed.
The flow direction in the bed can be either upwards or downwards using liquids preheated up to 60 ^{o}C.
The bed size is 10 cm in diameter and max. 1 m in height.

Organic synthesis

The plant is a multipurpose design for various organic synthesis. Originally build for homogenously catalyzed biodiesel production it can also operate with immobilized enzymes in backmix and fixed bed reactors.
The flexibility of the setup is due to the installation of 3 feed vessels, several reactors, a distillation column, holding vessels, separators and evaporators.
The largest vessel is 50 liter.
The plant is operated under ATEX requirements and can be inertizised by nitrogen. Full datalogging using Labview.
The plant is also used for research experiments.

Transport and separation of solids

In almost all chemical industries handling of bulk solids, powder, granules, crystals etc takes place.

The theories covering many of the relevant unit operations are not very well developed and the practical difficulties are obvious and un-predictable.

Pneumatic transport of fines and separation in cyclone/bagfilter is tested and pressure drop, solids load and gas velocities compared to theory.

High temperature processes

The department has a long tradition for working within the field of high temperature technology. At this plant some aspects can be tested: Running a gas burner under varying conditions, operating and calculation high temperature heat exchangers, operating a SCR de-NOx catalyst and using gas analyzing equipment constitutes parts of the application possibilities.

CIP cleaning

Clean-in-place (CIP) is a technology especially used by pharma industries to clean process equipment without dismantling. In addition to a mobile CIP unit the setup includes a transparent large scale model of a process plant. Using artificial and coloured dirt it is possible to follow the cleaning process as a function of various operating parameters.

Process control laboratory

On three plants it is possible to study various process control aspects.

A. On the 2-tank experiment it is possible to work with level and flow control using PID controller settings on the computer.
B. The HTST – High Temperature Short Time - pasteurizer is for work with temperature control using heat exchangers and holding tanks.
C. The distillation column control shall ensure the right concentration of product also taking thermodynamic aspects into account.

Tray column stripper

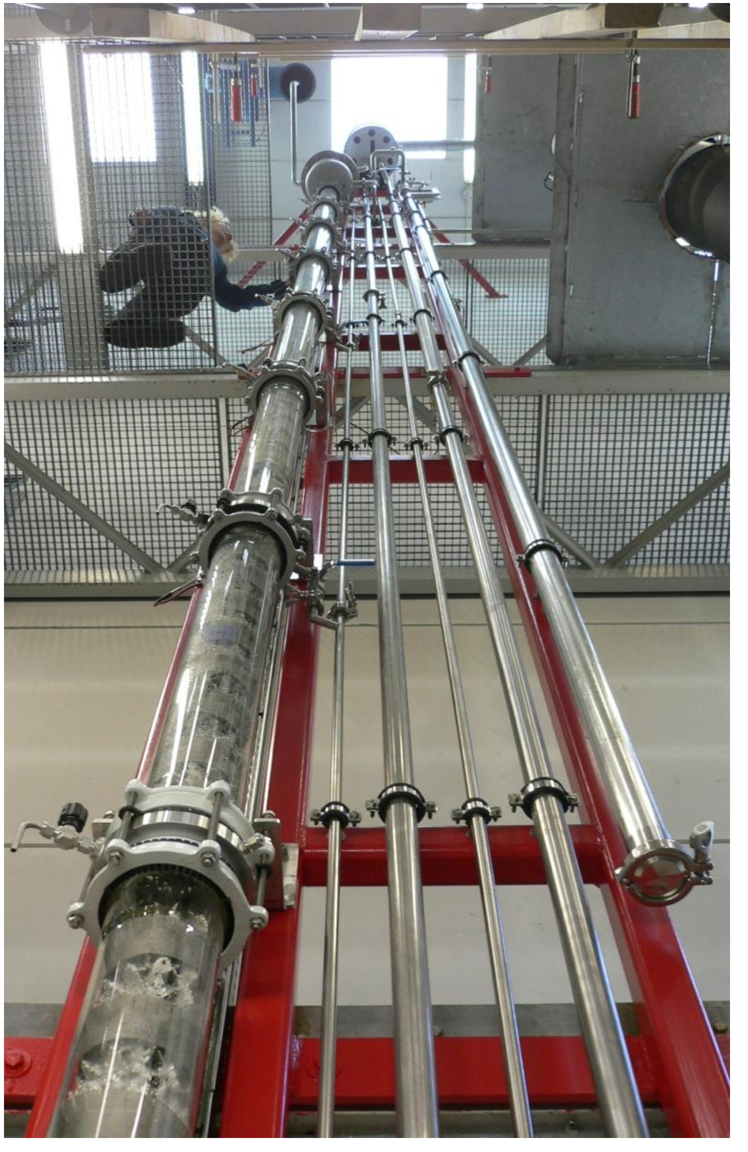
Ammonia dissolved into water is stripped off in a counter-current process using water.
The trays in the column are of special design and can be taken out and exchanged with other designs.
Since the plant is rather complex to use it is mostly used in our advanced course in process technology.

CO_2 absorption/desorption plant

One method of removing CO_2 from fluegasses is to use a liquid absorbent in a scrubber. A common absorbent is an aqueous solution of alcanol-amine, which however has to be regenerated in a desorber in order to be recycled. The process is a part of the CCS concept – Carbon Capture and Storage.

The absorber is more than 10 m high and used for CCS research with MEA – monoethanolamine and simulated fluegasses in counter-current flow.

The desorber is not yet implemented.



Fermentation

At the department we have 3 pilot scale fermentation setups. The plant on the picture comprises two fermentors – one 50 liter unit in stainless steel and one 15 liter unit in glass. They are both connected to one control panel, so they can only be operated one at a time.

The plant is connected to the steam and cooling water system, and has a separate hot water supply for temperature control. All data including supply of nutrients and acid and bases, pH, temperature and oxygen concentration etc. are recorded.

The other setups are a 100 liter U-loop fermentor and a standard 150 liter unit.

Various mobile equipment

Pilot plant courses

Course 28121: Laboratory in chemical unit operations. Basic course for 2nd-3rd year students, 5 ECTS. 4 experiments with reports performed in groups of 2 students. Spring and fall semester.

Course 28125: Same as 28121, but for the 3-weeks period in January

Course 28231: Advanced master course in process technology laboratory. 5 ECTS. 3-weeks period in June. Two or three assignments selected from a large number of individual projects given by teachers at KT and Food.

Course 28344: Biotechnology and process design. Mandatory course for diploma engineering students comprising fermentation and down-stream processing experimental activities including reports and a final design project. 15 ECTS, spring + June and fall + January.

Course 28122: 3-week summer university course in July for foreign and Danish students. Identical with course 28121.

Course 28123: 4-week special summer university course in July for International students (designed for American universities). In groups of 2 students 7 experiments with reports and a non-technical presentation are performed. 8 ECTS.

Course 28924: PhD/graduate course. 5 experiments with reports + utility report and oral presentation. 5 ECTS.

Course 28346: Advanced master course in fermentation technology in June. 5 ECTS

Special projects: Ba and Master projects or shorter R&D assignments.

Learning philosophy

- Hands on semi-industrial/industrial equipment
- Focus on plant and process design, scale up etc.
- Plan and perform large scale experiments, including application of various utilities (steam, compressed air, cooling water etc.)
- Critically search the necessary information
- Compare experimental results with theoretical expectations
- Utilize many different scientific subjects in one assignment, for instance analytical chemistry, thermodynamics, heat and mass balances, unit operations, kinetics, process control, uncertainty calculations, samp-ling techniques, safety and environmental aspects.
- Teach the students to write short and clear targeted technical reports
- Learn the students to think like an engineer through critically interpreting and evaluating results, finding errors, making conlusions.

Plant application

Most setups are usually applied for well defined experiments based on an operation manual. This is the case for the basic courses 28121, 28122, 28123, 28125 and 28344.

In the advanced course 28231 the equipment is used in a more free and holistic context, usually as a number of single steps constituting a complete simulated production process. In this case the mobile equipment is of special relevance.

The same may be the case for special projects.

For research purposes all equipment can be used as stand-alone units for a certain part of the research, or constitute the main device as is the case for the organic synthesis plant for biodiesel research and the CO_2 absorber for CCS research (carbon capture and storage).