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Specifications Continuously Stirred Tank Reactor

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1 General description

This Continuous Stirred-Tank Reactor (CSTR) will be used by the biorefinery cluster of the Biomass and Energy Efficiency (BEE) unit to research the conversion of biomass-derived components and chemicals in a continuous mode. The reactor will be used to process immiscible reagents, such as aqueous solutions, organic solvent-based mixtures, and combinations thereof, containing reagents that will be continuously fed to the stirred-tank, with contact time between 1 and 10 hours. The reactor must also include an effective liquid-liquid and gas-liquid mixing system. The stirred tank will be heated up to temperatures between 150 and 250 °C and continuously mixed by a stirring shaft to up to 2500 rpm. The reactor should be rated to pressures of up to 100 barg at 250 °C. An accurate control of the residence time, temperature and flow rates should be attainable by an independent control and programme software during any test run in the reaction rig. The heating/cooling capacity of the system should be so that short times are required to reach steady-state operation controlling temperature within ± 2 °C of the set temperature even with exothermic systems. Safety and emergency measures of the rig should cover events in which actions are taken against pressure and temperatures exceeding specified thresholds. Emergency buttons will be available to activate cooling and/or power down the system. The test rig will include two feeding vessels, one collection vessel, a fraction collector and an injection valve to link the product line to an HPLC system.

2 Technical requirements

2.1 General requirements of reaction vessel

Total volume	Max. 4 L Working volume: 70% of total volume Note: reactor volume is desired to be as low as possible, but enough to achieve accurate level and contact time control.
Operating temperatures	150-250 °C
Pressure rating	100 barg at 25 °C, and 100 barg at 250 °C
Space velocity	0.1 – 1 h ⁻¹ (contact times 1-10 h)
Mixing	By means of impeller driven by motor Low torque (0.5-2 x viscosity of water), high speed mixer (up to 2500 rpm). Impeller designed for gas/liquid applications, such as Rushton turbine and marine propellers, ideally with hollow shaft for gas dispersion (Dispersimax™ or equivalent).
Working substances	Liquids, mono- and biphasic (immiscible), including water, toluene, acetone, n-butanol, aldehydes
Material in contact with liquid	To manage reactants with pH between 0.5 and 9 at operating temperatures. Acid-resistant material (including HCl), no stainless steel, internally polished Resistant to working substances listed above.
Material not in contact with liquid	316L / 1.4404
Control system	Automated back-pressure control valve Programable parameters: Mixing velocity, liquid level, feed flows, and temperature of liquid. Set operation programme with up to 16 combinations of temperatures and flow rates.
Heating	Electric as preferred heating medium. Ideal heating rates of liquid in reactor: 5 °C/min

Ts = Set temperature
Tj = Jacket temperature
Tx = Actual temperature of the liquid in reactor

At $T_x < T_s$
 $T_j - T_s \leq 20\text{ }^{\circ}\text{C}$

At $T_x \approx T_s$
 $T_j - T_s \leq 5\text{ }^{\circ}\text{C}$

Temperature control: $|T_x - T_s| \leq 0.5\text{ }^{\circ}\text{C}$

Cooling

With cooling coil inside the jacket and around the vessel.

Rapid cooling to 60 °C with a rate of at least of 6 °C/min.

Actively controlled during exothermic/runaway reactions to keep temperature in the reaction vessel within $\pm 2\text{ }^{\circ}\text{C}$ of set temperature.

NOTE: Available service water at ambient temperature.

Solid catalyst

Catalyst basket.
Adaptable to stirring shaft or incorporated to additional stirring shaft.

Ports & valves in vessel

Total number of ports: 7

Top ports: 4

- 1 x vent
- 1 x N₂ purge
- 1 x H₂ feed
- 1 x pressure relief

Side ports: 2

- 2 x charging ports near to each other at 70% of the reactor vessel height, to connect the feed lines.

Bottom ports: 1

- 1 x product line

All ports with valves (with the exception of the pressure relief port)

**Gas feed
(Optional)**

H₂-designed sparging system
Up to 100 bar H₂ feed.

Indicator, measurements and logging

- Pressure, internal temperature of liquid, work of stirrer (for viscosity), flow rates (preferred volumetric, Coriolis acceptable), liquid level at which the reactor should be operated.
- Display and logging every one-to-few seconds of set-points, jacket temperature, liquid temperature, liquid level, pressure.
- Additional logging upon specific input signal, e.g. time, online auxiliary device.

Safety measures

- Pressure relief valve(s)
- Pressure with automatic actions
- Temperature with automatic actions
- Drip tray under vessel (TV+10%)
- Interlock on liquid level in vessel preventing to heat and activating alarm when level is lower than allowable
- Optional: Interlock on valves preventing to heat

Emergency actions

- Temperature: Exceeding threshold stops heating, starts cooling if possible
- Pressure: Exceeding threshold stops heating, starts cooling if possible
- Pressure relief valve: Exceeding max. pressure, heating already stopped, cooling already started if possible
- Loop break alarm
- Red emergency button: Power down skid
- Blue cooling button: Stops programme, starts cooling

Insulation

On all heated surfaces.
No open insulation areas.

2.2 Feeding system**Feed vessels**

2 mixing vessels with manual liquid loading (TV 20L each)

Mixing (Optional)

Low-torque, low speed.
Only required when liquids are loaded (no continuous stirring needed).

Feeding pumps

To set flows between 4 and 80 mL/min, to at least 300 barg.
HPLC-type pump, self-priming, chemical resistant
Plugged-in filter in pipeline to feed pumps.

Ports & valves in each feed vessel

Total number of ports: 8

Top ports: 6

- 1 x pressure relief
- 1 x N₂ purge
- 1 x vent
- 1 x feed return port
- 1 x charging port
- 1 x spare

Side ports: 0

Bottom ports:

- 1 x discharging port
- 1 x sampling port

All ports with valves (with the exception of the feed return ports and the pressure relief port)

Safety measures

- Drip tray under vessel (TV+10%).
- By-pass from feed vessel to product vessel and/or waste collection.
- Pressure relief valve on the feed line to the reactor vessel directed back to the feed vessel(s).

Material in contact with liquid

To manage reactants with pH between 0.5 and 9 at temperatures <50 °C.

Material not in contact with liquid

316L / 1.4404

2.3 Product/collection vessel**Vessel**

1 storage vessel (Total Volume of 20L)

Ports & valves

Total ports number: 10

Top ports:

- 1 x pressure relief
- 3 x charging ports (2 for feed vessels connection, 1 for reactor vessel connection)
- 1 x N₂ purge
- 1 x vent
- 1 x pressure control
- 1 x analytical measurement return line
- 1 x spare

	Side ports: 0
	Bottom ports: 1 • 1 x discharging port
	All ports with valves (with the exception of the pressure control and pressure relief ports)
Pressure Control	Pressure control by mean of automatic vent valve Pmax = 0.05 barg
Safety measures	Drip tray under vessel (Total Volume +10%). Pressure relief valve.
Material in contact with liquid	To manage reactants with pH between 0.5 and 9 at temperatures <50 °C.
Material not in contact with liquid	316L / 1.4404
2.4 Auxiliaries	
Pipes/lines	To manage reactants with pH between 0.5 and 9 at temperatures <50 °C
Sampling	Collector of at least 4 fractions (50 mL per fraction) on the pipe to collection vessel Programmable (optional)
Auxiliary analytical devices	T- junction on product line to connect analytical devices
2.5 Others	
IT	Centralized data logger Centralized process controller Network connection for data transfer
Power	Power connections are available (230VAC and 400 VAC).

Recommended dimensions	Up to 5 m ³ (W: 2m x L: 8m x H: 0.25m)
Max. surface loading	300 kg/m ²
Handing over	• Flow diagrams, P&ID, complete engineering and process instrumentation drawings • Complete instrumentation and control documentation • Complete material documentation list incl. certificates • Fully assembled, wired and turn-key • Operating manual • NOBO certification • PED documentation • CE declaration • FAT protocol designed in close consultation with ECN/TNO • FAT documentation incl. results
Spare parts	1 x every type of Pump 1 x back pressure control valve 1 x reactor impeller 1 x every type of valve 1 x every type of relief valve
Training	2-day training and demo at ECN part of TNO
Warranty	Included

Appendix 1. Equipment diagram

