Advanced BUSS Loop® Reactor Technology

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Abstract

This paper highlights the operating principle of the BUSS Loop® reactor technology and its theoretical background. Data comparing the loop reactor (LR) technology with other reactor technologies is provided to demonstrate that LR's are also in practise yielding the highest specific mass transfer performance momentarily available. Typical areas of application and possible operating concepts of the Advanced BUSS Loop® reactor technology (ABLR) are discussed, as well as the benefit of an early involvement of an external specialist during process optimisation.

Introduction

Process intensification has recently received widespread attention because of its potential to achieve significant reduction in capital costs as well as its ability to improve reaction yields. Furthermore, many of the safety, health and environmental concerns can benefit from the inventory reduction, improved containment possibilities, enhanced selectivity, and increased heat and mass transfer rates achieved by using process intensification technologies. Process intensification is no longer just the realm of high volume chemicals production, but can be equally applied to small volume specialty production. As well as reducing capital cost, process intensification also offers other potential benefits.
Matching the mixing rate and residence time to the required reaction rate, and removing product from the reaction zone will improve the yield and reduce the possibility of by-product formation or degradation of reactant or product, which also has clear environmental benefits. Because of fewer byproducts, product quality increases and constant product quality can be achieved.

All of the above mentioned factors are influenced by the reactor technology chosen and hence will affect initial investment cost and operational cost of the process as a whole. The ABLR technology (as shown in Fig. 1), is made up of a reaction vessel with a high performance gas/liquid ejector to achieve high mass transfer rates, and is globally in operation since many years.

It provides a significant improvement of intensification for heat- and mass transfer, and has been successfully applied to a range of processes including hydrogenation, oxidation, phosgenation, alkoxylation, amination and sulphonation among others.

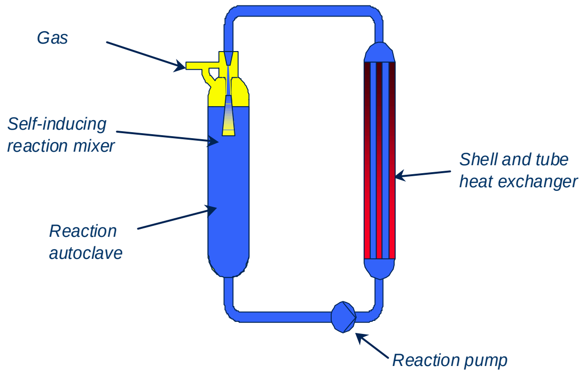


Fig. 1: Advanced BUSS Loop® reactor

In the following, the operating principle of the Advanced BUSS Loop® reactor technology and its theoretical background is outlined. Based on this more in-depth understanding, chemical engineers are in the position to select the right reactor technology matching their mass transfer and heat transfer requirement, by using process intensification technologies.

Reaction kinetics and mass transfer

Some chemical reactions are carried out at conditions where the mass transfer from the gas to the liquid (and/or liquid to the solid phase, i.e. heterogeneous catalyst) is not limiting at all and where only the conversion rate will dictate the type and size of the reactor system. Actually, most chemists in the first trials will choose conditions in such way that in their laboratory autoclaves adverse effects due to mass transfer limitations will be negligible.

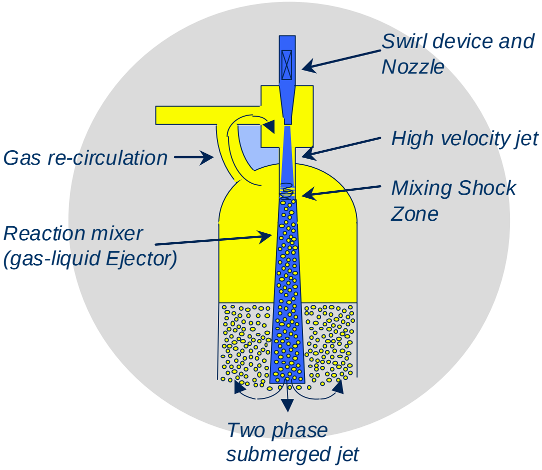
For developing a new chemistry and to investigate the kinetics thereof, the chemists normally choose:

- low substrate concentrations
- high stirring speeds
- low temperatures
- high pressures
- low catalyst concentrations

After finding the specific kinetics, they will start to change parameters in order to improve the economics of the process. That's when reactions are mostly identified as mass transfer controlled. Changing the reaction parameters will probably result in problems such as undesired side reactions, difficulties in temperature control or catalyst deactivation effects. However, it is important to find the conditions where mass transfer starts to play a role, because mass transfer could be a limiting factor on the larger scale reactors.
Where the chemical reaction is solely controlled by the kinetics, the scale-up to industrial size reactors becames a question of expertise and is releated to the desired capacity. The aspects of scaling-up stirred vessels and loop reactors have recently been reported by L.L. van Dierendonck, et al. From this publication, it may be concluded that the scale-up mass transfer limited reactions in stirred vessels can be very complex and difficult. The scale-up of the ABLR, is much easier and more reliable if you are experienced with these systems.

Operating principles of Advanced BUSS Loop® reactor technology

The ABLR consists of a reaction autoclave, a circulation pump, a heat exchanger and a reaction mixer (gas-liquid ejector). This system requires the same number of elements as a stirred vessel system but is arranged in a completely different way.



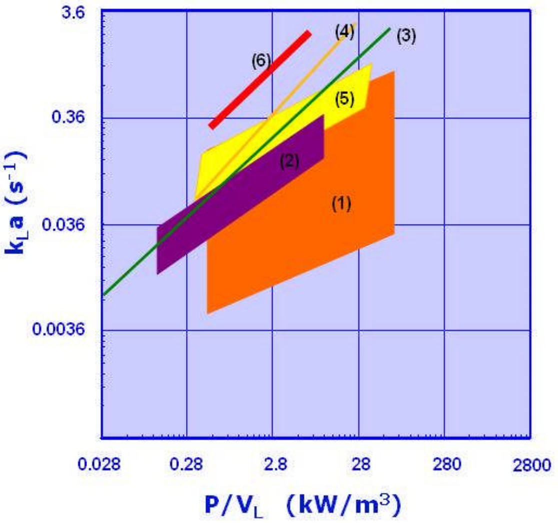
(1) The **reaction mixer** (instead of a sparger or other gas distribution system) is a high performance-gassing tool. A gas-liquid ejector consists of four main sections. A optional swirl device directs, orientates and stabilises the pumped liquid flow. Then it passes through a nozzle that provides a high velocity jet of fluid to create suction of the gas in the gas suction chamber and entrain gas into the ejector. In the following mixing tube the liquid jet attaches itself to the mixing tube wall resulting in a rapid dissipation of kinetic energy. This creates an intensive mixing shock zone where the high turbulence produces a fine dispersion of bubbles. The ability to generat and finely disperse very small gas bubbles to the liquid (30 to 70 µm) with a gas-liquid ratio between 0.5 and 2.0, or even more, makes this an ideal tool as primary dispersion device for gas-liquid reactors. The two-phase mixture created in the reaction mixer is then injected into the fluid of the reaction vessel.

(2) The **reaction vessel** of an ABLR reactor does not need baffles. It is normally built with a larger L/D ration than the stirred vessel and is thus lower in costs, especially for high-pressure reactions. The two-phase mixture that "jets" into the reaction autoclave causes an intensive secondary mixing and a very high mass transfer rate due to the small bubbles, which were created in the reaction mixer. The average bubble sizes in the reaction autoclave are in the range between 0.2 and 0.7 mm (larger than the primary bubbles due to coalescence phenomenon).

(3) The **external heat exchanger** (instead of coils or internal exchangers) can be built as large as required and is not limited by the reactor’s working volume. The full heat exchanger area is available, even if the reactor is operated with reduced working volumes (e.g. semi-batch operation).

(4) The **circulation pump** (instead of an agitator) allows high power input per working volume (kW/m^3) in those cases where high mass transfer rates have to be achieved. New pump designs are now available which allows pumping of liquids with high solid (catalyst) contents (up to 8 wt %) and high gas loads (up to 30 vol %).

The hydrodynamics and the mass transfer characteristics of loop reactors have been investigated and reported by several authors. (Henzler (1981/1982/1983), Zahradnik et al. (1982/1991), Dutta et al. (1987), van Dierendonck et al. (1988), Cramers et al. (1992/1993/2001) and Havelka (1997)) The effect of the reaction mixer not only results in a high gas fraction in the vessel, but also offers a high mass transfer rate within the reaction mixer. The reported mass transfer characteristics of loop reactors and the typical relation of the mass transfer coefficient to the power input per unit of volume is shown in Fig. 3.



1. Stirred reactor (STR) with external cooling
2. Conventional stirred vessel equipped with six bladed turbine stirrer
3. Self-aspirating stirrer in a vessel
4. Self-aspirating stirrer in combination with EKATO phasejet in autoclave

5 „Schlaufenreaktor“- loop reactor (LR)

6. Advanced BUSS Loop® reactor (ABLR)

Fig. 3: Mass transfer rates in various reactor systems vs. power input per unit volume.

In Fig. 3 it is shown, that the newer design of the Advanced BUSS Loop® reactor achieves higher mixing and gassing rates to the reaction vessel by using a reaction pump that is able to circulate liquids with a high gas load (up to 30 vol. %) which also enhance even more the mass transfer in the reaction vessel and external loop. In order to generate and improve mixing and/or mass transfer, the reactor equipment must direct the energy most efficiently into the fluid system. In a stirred tank reactor, the energy input clearly comes through the impeller, but this arrangement suffers high-energy losses through frictional and other losses. The energy that remains is focused mainly upon the fluid directly in contact with the impeller. This means that while power inputs at the impeller tip may be relatively high, the majority of the fluid is unaffected and the average power input across the whole tank is low (0.1 – 0.7 W/kg).

Considering these facts one may conclude that the ABLR reactor is the most interesting alternative to the stirred vessel if one or more of the following conditions apply:

- reactions at higher pressures
- mass transfer controlled reactions
- strongly endothermic or exothermic reactions
- requirements for flexible operating volumes
- requirements for product equivalence at different reactor sizes
- requirements for gas treatment

Flexibility in working volume

A multi-purpose facility for producing pharmaceutical chemicals or specialty chemicals is normally set-up with different types and sizes of reactors in order to be flexible to the required production volumes for each product.

The following parameters may affect the choice of equipment:

- type of reactions to be performed
- required temperatures and pressures
- maximum viscosity
- required maximum solid content
- required material of construction
- minimum/maximum working volumes

Considering the fact that stirred vessels can only be operated in a narrow range of operating volume without losing mixing efficiency, heat transfer surface and mass transfer rates, it is assumed that the working volume may only be varied between 60 and 110 % of the nominal capacity. The Buss Loop® reactors may be operated between 30 and 110 % of the nominal capacity, still offering the same heat transfer area, the same mixing efficiency and almost the same mass transfer rates.

This example illustrates how important the right choice of reactor type and size can be for a multi purpose facility.

Continuous operation

As discussed, the reaction mixer generates very fine dispersed gas bubbles and offers very high local mass transfer rates. The highest energy dissipation takes place in the mixing shock zone within the reaction mixer, resulting in extremely fine gas bubbles and very high mass transfer coefficients. The concept of a continuous operated Advanced BUSS Loop® reactor is shown in a simplified flow diagram (Fig. 4).

An excellent example for a reaction carried out in continuous ABLR reactor is the conversion of Nitro-compounds to the corresponding Amines. While maintaining a high loading of active catalyst in the reaction suspension, the Nitro-compound is fed continuously into the mixing zone of the reaction mixer. The conversion of the Nitro-compound will take place immediately and completely because of the presence of catalyst, the presence of hydrogen and the high energy dissipation in the mixing shock zone. The product is continuously filtered through the cross-flow filter system out of the reactor system. In a continuous stirred vessel system, this reaction would require a cascade of stirred vessels with a total residence time of 3 to 4 hours, whereas the ABLR required a total residence time of ½ hour only. Further, due to the high exothermic reaction stirred vessels would require the use of a solvent in order to dilute the solution and to reduce the heat released. This example shows that when using a stirred autoclave the total reaction volume would be at least a factor of 4 to 6 higher than when using the BUSS technology. That's why a continuously operated ABLR is a typical example of a high performance reactor using the concepts of process intensification.

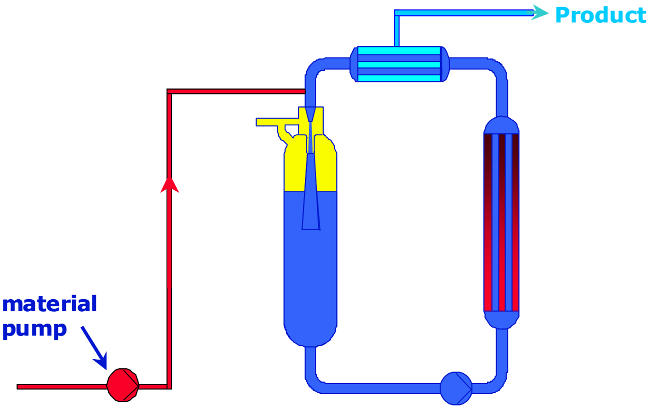


Fig. 4: Simplified diagram of a continuous operated Advanced BUSS Loop® reactor system

Benefits when using the BUSS gas-liquid reactor technology

1. *highest mass transfer rates and unconstrained heat transfer area proven in industrial plants*

2. *significant savings in catalyst cost:*

a) The percentage of slurry phase catalyst added to the reactants is less than in a STR, and this still gives a shorter reaction time and better yield. In some cases, the reduction is 50 to 70 %. This means that the capital tied up in catalyst stocks can be correspondingly reduced and will generate major savings in operational costs.
b) Catalyst lifetime can be increased when reaction time is shortened, reaction conditions are milder and byproduct formation is reduced. This results in a further reduced consumption.
c) Furthermore, lowering the quantity of catalyst present also reduces losses during handling and filtration which tend to be a fixed percentage of the total present.
d) Lower catalyst concentrations also mean that catalyst filters can be smaller and therefore less expensive.

3. *no scale-up problems on an industrial scale and guaranteed process and performance:*

The initial development of a chemical reaction is often done in the time honoured laboratory autoclave. As soon as the process optimisation and development process starts it is important to work with larger batch sizes. It is important not to underestimate kinetic effects when it comes to the step from laboratory autoclave to industrial scale. The best and most reliable results are normally obtained in one of two BUSS pilot plant scale loop reactors (15 or 50 litre) which allow to achieve the highest selectivity and yield, to reach a high capacity and throughput, demonstrate an excellent reproducibility and allows flexibility with respect to production parameters (turn down ratio, variation of grades, etc.). The results thus obtained can easily be scaled up to any size of industrial reactor. A scale up factor 500 to 1 or more presents, according our longterm experience, no risk. Based on the pilot plant trials BUSS provides full process and performance guarantees.
In the case of a stirred tank autoclave, scale up is more difficult due to changes in mixing pattern and area to volume ratios between a pilot plant scale and a production scale vessel. Mixing intensity cannot be scaled up so readily as temperature and concentration gradients arise which often lead to longer reaction times and lower yields.

4. *reduction of reaction pressure* (superior mass transfer rate)

This consequently allows reduction of the initial investment cost of the reaction section and gas supply system

5. *excellent cleaning features:*

The autoclave of the BUSS Loop® reactor has no internals besides from the ejector, which is located in the top of the autoclave. There are no cooling coils and/or plates, sparging tubes or baffles and no agitators, which are dipped in the reaction fluids and which are difficult to clean. The whole reactor can be completely drained, so that product losses are low when a product change is made and solvent washing (when required) is relatively quick and easy. That's why loop reactors are also particularly suitable for multi-purpose plants and increase the operational availability and flexibility.

Conclusions

- The product equivalence to be achieved in different sizes of reactors is becoming increasingly important not only for the pharmaceutical and fine chemical industries. This requirement can easily be fulfilled with loop reactors, because of the identical performance at all sizes.
- The BUSS loop® reactor has clear advantages in the field of continuous reactions utilising the high energy dissipation as well as in batchwise operations.
- Loop reactors originally developed for hydrogenations are used today also for phosgenation, alkylation, amination, carbonylation, oxidation and other gas-liquid reactions. This trend will continue.
- The Advanced BUSS Loop® reactor is a proven and a state of the art reactor design for industrial performed slurry phase reactions.
- The inherently safe design of BUSS fulfills the highest required environmental and safety regulations.
- An early involvement of external specialists gives security in process optimisation, shortens the time to market and minimises your costs.
- The BUSS Loop® reactors are designed to increase your efficiency, productivity, utilized capacity and process flexibility to operate safely with high product quality to cut down on your operational costs.

Literature

Cramers, P.H.M.R. et al., Hydrodynamics and Mass Transfer Characteristics of a Loop-Venturi Reactor with a down-flow liquid jet ejector, Chem. Eng. Sci. (1992), 47, 3557-3564.

Cramers, P.H.M.R. et al., Hydrodynamics and local mass transfer characteristics of gas-liquid ejectors, Chem. Eng. J. (1993), 53, 67-73.

Cramers, P.H.M.R. et al., Influence of the ejector configuration, scale and the gas density on the mass transfer characteristics of gas-liquid ejectors, Chem. Eng. J. (2001), 3770, 1-11.

Dierendonck L.L. van, et al., Scale up of Gas-Liquid reactions made simple with loop reactors, 6th European Conference on Mixing (1988), Pavia, Italy, 287-295.

Dierendonck L.L. van, et al., Loop Venturi Reactor-a feasible alternative to stirred tank reactors? Ind. Eng. Chem. Res. (1998), 37, 734-738.

Dutta N.N. et al., Mass Transfer and Hydrodynamic Characteristics of Loop Reactors with Downflow Liquid Jet Ejector, Chem. Eng. J. (1987), 36, 111-121.

Havelka, P. et al., Effect of ejector configuration on the gas suction rate and gas holdup in ejector loop reactors, Chem. Eng. Sci. (1991), 52, 1701-1713.

Henzler, H. -J., Das Sogverhalten von Strahlsaugern f Stoffsysteme: flVT-Verfahrenstechnik. (1981), 15(10), 738-749.

Henzler, H. -J., Zur Auslegung von Strahlsaugern f einphasige Stoffsysteme. Chem.-Ing. -Techn. (1982), 1, 8-16.

Henzler, H.J., Design of ejectors for single-phase material systems. Ger.Chem.Eng. (1983), 6, 292-300.

Zahradnik et al., Hydrodynamic Characteristics of Gas-Liquid beds in Contractors with Ejector type gas distributors, Coll. Czech. Chem. Comm. (1982), 47, 1939-1949.

Zahradnik et al., Design and scale-up of Venturi-tube gas distributors for bubble column reactors, Coll. Czech. Chem. Comm. (1991), 56, 619-634.

BUSS ChemTech AG company profile:

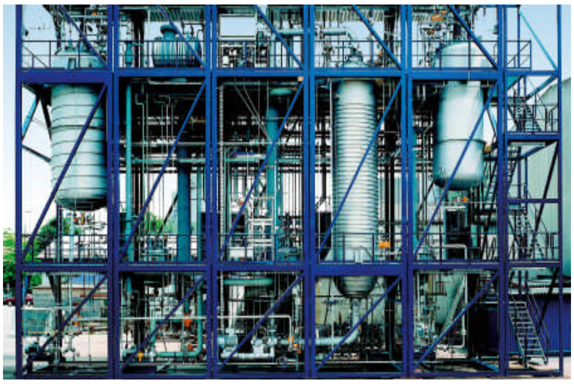
BUSS ChemTech AG, a Switzerland-based chemical engineering company was founded in 1884. We are specialized in optimising and developing chemical reactions following your requirements and supplying you with engineering together with full performance guarantees and equipment.

Since 1950, when the first BUSS LOOP® reactor ever was set into operation, we developed BUSS LOOP® Process Technologies for more than 800 different reactions and several reaction types and provided the whole range of chemical engineering services from process development to basic engineering to key equipment and to complete production plants.

BUSS ChemTech AG has state-of-the-art laboratories and - unique for an engineering company - three pilot plants on site in Pratteln. Therefore, our scale-up is not a theoretical calculation but experimentally proofed and reliable data (e.g.: yield, selectivity, throughputs, reaction time and, if present, catalyst consumption).

That is why we feel so confident when offering you process and performance guarantees to support you with the most profitable and productive process technology.

As a reliable partner with widespread expertise in chemical process technology, developed during the past decades and more than 250 plants put into operation, we would like to serve you and welcome you to benefit from our expertise.



The BUSS LOOP® REACTOR

Designed to increase your efficiency, productivity, flexibility and to cut down on your costs!

- Aminations
- Alkylations
- Carbonylations
- Chlorinations
- Ethoxylations
- Hydrogenations
- Nitrilations
- Oxidations
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