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Counting bubbles: precision process control of gas-liquid reactions in flow with an optical inline sensor

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Abstract and Figures

Quality by Design encouraged by the US Food and Drug Administration (FDA) in the continuous flow synthesis requires tight monitoring of all the reaction input and output parameters to improve reproducibility and eliminate the process rejects. The reaction monitoring, however, relies on costly (above 10,000$) analytical equipment-one of the factors preventing a wider utilisation of continuous processes. In the work, we show that gas-liquid reactions can be monitored using low-cost (10$) hardware-optical liquid inline sensors-that allows instantaneous analysis of gas fraction in the moving stream. We discuss the application of the sensor for various gas-liquid reactions. The gas-consuming reactions such as hydrogenation are the easiest to implement because the sensor without calibration provides accurate readings close to complete consumption of the gas. The gas-evolving reactions can be monitored but require sensor calibration to determine the gas fraction accurately. Operation of the sensor was demonstrated for various hydrogenation reactions self-optimised using a proportional-integral (PID) algorithm which adjusted the substrate concentration to provide high (but not full) pre-defined hydrogen consumption. The optimised hydrogen consumption agreed with the product analysis for a range of the substrates hydrogenated under various pressures and with different selectivities. The optical sensor was also proven to be an efficient tool in adapting the reaction condition to the catalyst deactivation in the reaction of 2-methyl-3-butyn-2-ol semi-hydrogenation-the autonomous reactor allowed reaching a turnover number (TON) of $2.7 \cdot 10^6$ with the value of $1.5 \cdot 10^7$ expected till a twofold decrease in the catalyst activity. The TON values demonstrated are significantly higher than those observed in batch reactors ($\sim 10^3$) even in case of catalyst re-use (10^5) demonstrating a substantial improvement of process sustainability operating with the process control.

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Counting bubbles: precision process control of gas-liquid reactions in flow with an optical inline sensor

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Abstract

Quality by Design encouraged by the US Food and Drug Administration (FDA) in the continuous flow synthesis requires tight monitoring of all the reaction input and output parameters to improve reproducibility and eliminate the process rejects. The reaction monitoring, however, relies on costly (above 10,000$) analytical equipment – one of the factors preventing a wider utilisation of continuous processes. In the work, we show that gas-liquid reactions can be monitored using low-cost (10$) hardware – optical liquid inline sensors – that allows instantaneous analysis of gas fraction in the moving stream. We discuss the application of the sensor for various gas-liquid reactions. The gas-consuming reactions such as hydrogenation are the easiest to implement because the sensor without calibration provides accurate readings close to complete consumption of the gas. The gas-evolving reactions can be monitored but require sensor calibration to determine the gas fraction accurately. Operation of the sensor was demonstrated for various hydrogenation reactions self-optimised using a proportional-integral (PID) algorithm which adjusted the substrate concentration to provide high (but not full) pre-defined hydrogen consumption. The optimised hydrogen consumption agreed with the product analysis for a range of the substrates hydrogenated under various pressures and with different selectivities. The optical sensor was also proven to be an efficient tool in adapting the reaction condition to the catalyst deactivation in the reaction of 2-methyl-3-butyn-2-ol semi-hydrogenation – the autonomous reactor allowed reaching a turn-over number (TON) of $2.7 \cdot 10^6$ with the value of $1.5 \cdot 10^7$ expected till a twofold decrease in the catalyst activity. The TON values demonstrated are significantly higher than those observed in batch reactors ($\sim 10^3$) even in case of catalyst re-use (10^5) demonstrating a substantial improvement of process sustainability operating with the process control.

1. Introduction

Gas-liquid reactions are widespread in the chemical industry and range from oil desulfurization performed on a gigaton scale to metathesis on a gram scale. Gas-liquid reactions involve numerous reaction classes and functional groups, but hydrogenations are particularly important because of versatility, and often perfect atom efficiency.[1–3]

A majority of hydrogenation reactions in the fine and pharma industries are carried out in stirred-tank batch reactors.[4,5] Low production and economic performance shown by the batch reactors comes from mass and heat transfer limitations as well as myriad operations such as substrate loading, heating, cooling, and reactor cleaning – repetitive and non-productive. The performance can be enhanced with the continuous flow chemistry.[6–8]

Reactions in continuous flow improve micro-mixing, heat dissipation, process safety and reaction control.[9–11] Sub-microsecond chemistry becomes possible in flash reactions.[12] Flow chemistry, as a result, is getting adopted in the research and manufacturing processes.[11] However, a majority of the

1

reactions already converted to flow are either exothermic or hazardous liquid-liquid reactions. Gas-liquid reactions attract disproportionally little attention.[11,13–16] The likely reason is that gas-liquid reactions, such as hydrogenations, have lower reaction rates and their hydrodynamics is more difficult to control resulting in a limited reproducibility.[17]

The gas-liquid reactions often require a solid catalyst making them three-phase reactions. High pressure drop, limited product selectivity, formation of hot spots and associated quick catalyst deactivation are usual problems.[18–20] The exact control of residence time is another problem because different two-phase flow regimes can co-exist in different parts of the reactor.[19,20] Nevertheless, there are ample examples of carrying out the gas-liquid reactions with high selectivity in a continuous fashion achieving excellent catalyst utilisation and process intensification.[21–24]

Another barrier for a wider adoption of continuous flow manufacturing for liquid-phase as well as gas-liquid reactions comes from the need for precise process monitoring, automation and control. Control of all the input operational parameters (temperature, pressure and flow rates) as well as the main reaction indicators (conversion, selectivity) is the key for reproducible process operation.

In a manufacturing process, autonomous operation of the continuous flow processes decreases the labour costs and improves process safety.[11,25–29] Product quality obtained under the precise process control is ensured and any abnormalities are detected and eliminated.[30] The US regulator, Food and Drug Administration (FDA), encourages adoption of continuous flow processes because they provide the Quality by Design.[31,32]

In a research environment, autonomous operation of a flow reactor improves efficiency allowing unprecedented testing throughput and improved reproductivity of the processes.[33] Self-optimisation of reaction conditions is one of such examples – the process where the software finds the optimum conditions without human interaction.[11,28,34–38] Months of the experimental work can be performed in days.

Monitoring the input reaction parameters is straightforward and involves standard equipment such as flow meters, temperature and pressure transducers.[13] Monitoring the output reaction parameters is, unfortunately, significantly more difficult because it often requires gaining insights into the chemical composition. The literature shows examples of using online chromatography, NMR, mass, IR, Raman and UV-vis spectroscopy.[29,33,39–44] Selecting a proper reaction monitoring tool involves finding a balance between the (i) price of the process monitoring solution, (ii) versatility for various reactions, and (iii) data quality and acquisition speed. Quality of data is, not surprisingly, prioritized and the process monitoring tools cost above $10,000 a unit – prohibitively expensive for many labs.

High equipment price and uncertainties in process monitoring impose limitations on process automation. In the current work, we show that excellent data quality in gas-liquid reactions can be achieved at high acquisition speed with low-cost equipment. A 10$ inline optical sensor can be used to control reaction conditions precisely. The sensor can also be employed in self-optimisation of reaction conditions to provide the required gas consumption and maintain the product yield regardless of the course of catalyst deactivation.

2. Experimental

The hydrogenation experiments were performed in an automated system described in our previous work.[45] Briefly, the system included two HPLC pumps (Knauer P4.1S) to control the liquid flow rates (Fig. 1). The gas flow rates were set with a set of mass flow controllers (Bronkhorst) and pneumo-actuated valves (Swagelok) placed afterwards.[45] The gas and liquid flows were combined in a T-mixer (IDEX). A reactor was placed in a convection oven followed by a back-pressure controller (Equlibar). The reaction was performed in catalyst-coated tube reactors (1.27mm inner diameter, 1.6 mm outer diameter) provided by Stoli Catalysts Ltd. The catalyst-coated tubes were used to

2

packed-bed reactors.[17] A filter (IDEX A-410) was placed after the catalyst-coated tube to protect the pressure controller from the possible catalyst particles. The liquid products were placed into vials with septa using a Zang Autosam 360 automated sample collector. The samples were analysed with an offline gas chromatograph (Shimadzu GC-2010) equipped with a flame ionisation detector and a Stabilwax column 10 m x 0.15 mm x 0.15 μm. Product conversion and selectivity were calculated using equations provided in the Electronic Supplementary Information, S1.

Fig. 1. Scheme of the hydrogenation reactor with a feedback control and process monitoring with an optical sensor.

The reactor was controlled with an OpenFlowChem software[45] and contained two systems: (i) the flow reactor until the back-pressure regulator, (ii) and a control system containing the optical sensor and the sample collector. The flow reactor was executing the pre-defined reaction conditions (flow rates, temperature, pressure). The reactions were performed by mixing a 0.5 M substrate solution with isopropanol solvent to maintain a constant liquid flow rate of 1.5 mL min^{-1} at a reaction temperature of 50-90 ^{o}C and pressure of 1.3-6.0 bar (absolute). A simplified system used for the validation of the optical sensor is described in the Electronic Supplementary Information, S2.

3.   Results and Discussion

3.1.   Head-on approach: combine equimolar quantities and let them react

First of all, we would like to discuss why semi-hydrogenation may benefit from complex process control. Indeed, the best control measures are often passive. In case of a temperature control, for example, overheating can be prevented in some cases without any process control because the heat dissipation rate increases with temperature.[46] The next step in the process control complexity might be a bimetallic strip that bends at known temperatures or a phase-change material that consumes heat – both are simple and reliable for maintaining a process temperature.[47,48] In case of hydrogenation, it is possible to avoid complex process monitoring and rely on a performance margin. The reaction can be carried out at a contact time longer than required so that deactivation and external disturbances do not affect the full conversion.

As an example, we took alkyne (MBY) semi-hydrogenation to alkene (2-methyl-3-buten-2-ol, MBE) shown in Fig. 2 as a model and the compound used vitamin synthesis.[3,23,49] A straightforward approach to control the reaction could be feeding an equimolar combination of MBY and H$_2$ with a sufficient contact time for full H$_2$ consumption – the performance margin.

3

Fig. 2. Scheme of the 2-methyl-3-butyn-2-ol (MBY) semi-hydrogenation with alkene the desired product.

Operating catalysts under hydrogen-lean conditions, unfortunately, brings a lot of problems outlined in Table 1. First of all, the performance margin introduced provides a sub-optimal reactor throughput, the unutilised performance. Secondly, it is difficult to predict when the performance margin "wears out" – the conversion may remain complete for days followed by a sudden drop resulting in contamination of the product with the feedstock. Therefore, a detailed catalyst stability study is required before using the approach. Thirdly, the catalysts tend to leach in the absence of hydrogen.[50] Leaching brings contamination of the product stream which might require additional purification. Leaching also alters and removes the catalyst resulting in faster deactivation with more frequent reactor maintenance cycles.[50–52] Lastly, any contact time beyond the minimum facilitates side reactions such as isomerisation, oligomerisation, or decomposition.[53,54] Therefore, simplicity brought with the performance margin is fraught with problems.

Table 1. Comparison of controlling a semi-hydrogenation reaction by ensuring complete consumption of a limited amount of hydrogen.

Advantages	Disadvantages
Simplicity	Underutilised performance
Quick to set-up	Stability optimisation required
No need for process control equipment	Accelerated catalyst deactivation by leaching
	Product contamination by catalyst leaching
	Side-reactions

Simplicity, nevertheless, might be a substantial incentive to try this simple process control approach. We performed an experimental study of MBY semi-hydrogenation in a 1 m tube reactor wall-coated with a 2.3 wt% Pd/C catalyst. Because the extent of the required performance margin cannot be predicted in advance, we gradually increased the reaction pressure and collected the products. The initial H_2 to MBY molar ratio was kept at 105% to ensure high MBE yield despite unavoidable over-hydrogenation to MBA (Fig. 2) that consumes twice as much H_2 as MBE.

Fig. 3 shows the effect of reaction pressure on the MBY conversion and MBE selectivity. On increasing pressure from 1 to 11 bar, the MBY conversion increased because of (i) the higher reaction rate which is proportional to the H_2 pressure[21,55] and (ii) a longer liquid residence time due to compression of H_2 bubbles at higher pressure. The MBE selectivity in the 1-11 bar range was 93-95% which is typical for supported Pd catalysts.[21,56–59]

4

Fig. 3. Effect of reaction pressure on MBY semi-hydrogenation performance over a 2.3 wt% Pd/C catalyst-coated tube: (■) MBY conversion, (♦) MBE selectivity, and (●) H_2 consumption calculated from product composition. Reaction conditions: 0.5 mL min^{-1} 5M MBY, 35 ℃, 5% molar excess of H_2 to MBY.

At a pressure of 12 bar, the conversion reached the maximum value of 98.1% and decreased the MBE selectivity to 93%. The decrease in selectivity at a high conversion is known to appear from the competitive adsorption of the dominant alkene and minor alkyne species onto the Pd sites.[22,23,57,58,60] A single phase (liquid) flow was observed at the reactor outlet confirming full H2 consumption. The product composition confirmed that MBY consumed 103% molar equivalent of H_2, in agreement with the introduced feed ratio.

Above 12 bar, H_2 had already been fully consumed. A further increase in pressure was expected to bring changes neither in the MBE selectivity nor in conversion. On the contrary, the experimental data showed a decrease both in the MBE selectivity and the MBY conversion (Fig. 3). The performance margin introduced by the higher reaction pressure did not provide the desired control over the reaction. Study of this effect is beyond the scope of this work, but it was found to be reproducible over a range of catalysts, pressures and concentrations. This simple model reaction clearly demonstrates that the "performance margin" may bring anything but performance and some form of "complex" process control is beneficial.

3.2. Applicability of an optical sensor

We used a commercially-available 10$ Optek liquid optical sensor for the process monitoring. The sensor consists of an infrared light-emitting diode and a photo-transistor with a transparent 1/16" tube in between (Fig. 4). The sensor detects liquid based on light refraction and allows analysing optically-transparent liquids such as water. The output is a binary signal – the presence or absence of liquid in the detection volume of below 0.2 μL. The sensor has an excellent response speed typically an order of 50 μs; therefore, the measurements can be reliably performed on quickly-moving feeds.

5

Fig. 4. Photograph of the Optek optical sensor with 1.59 mm OD tubing.

We used the optical sensor to determine liquid fraction (LF) – the fraction of liquid in the gas-liquid flow by reading the liquid presence every 100 μs and averaging the data over 10 s. The response time of 10 s is considerably faster than chromatography and is comparable to the spectroscopy.

The accurate LF measurements are obviously possible only for the Taylor flow regime because the relative abundance of liquid slugs is expected to provide the LF.[61,6263] The annular or slug-annular flow regimes, where the liquid film moves along the reactor wall as a film,[22,64–66] seem unsuitable for the detection. This is because the light refraction properties of the film may depend on the media, film thickness and velocity to result in false readings.

The accuracy of the sensor was first verified with a model N_2-isopropanol flow with the technical details shown in Electronic Supplementary Information, S2. Fig. 5 shows the relation between the introduced and the measured LF in the model flow with the dashed line corresponding to the expected measurement. All the LF readings were consistently overestimating the correct value by 10-25%. The likely reason is that in the meniscus at the gas-liquid boundary introduced strong light refraction so the sensor considered the slug boundary as filled only with liquid underestimating the gas content.

When the gas-liquid flow was formed with a T mixer (Fig. 5), a broad "hump" appeared at the 30-60% LF for the liquid flow rates of 200 and 500 μL min^{-1}. At higher liquid flow rates, the "hump" disappeared and the LF readings were consistent (within 2%).

The T mixer, however, generated Taylor flow with constant gas and liquid bubble sizes – not always the flow observed in the hydrogenation.[18,21,22,57] Therefore, we placed a large-volume filter (300 μL) after the T mixer to introduce flow irregularity. With the filter, no "hump" was observed and the LF readings became considerably closer to the expected LF values likely because the slugs were longer resulting in a smaller number of inter-phase boundaries. However, there was a significant discrepancy between the LF readings observed with and without the filter.

6

Fig. 5. Comparison of the liquid fraction introduced and determined by the optical sensor in the model isopropanol-N_2 flow.

Therefore, the LF readings of the optical sensor depend significantly on the liquid flow rates and slug lengths. The difficulty in controlling the slug lengths after the reactor outlet is a limiting factor in the applicability of process monitoring with the optical sensor. The zoomed-in area in Fig. 5, however, demonstrates that the LF readings above 95% become consistent. Nevertheless, there is also a risk of falsely obtaining 100% LF readings when a small amount of gas is present in the flow – at the introduced LF above 95%. This phenomenon comes, likely, from the sensor inability to detect gas in the bubbly flow – the regime where the gas bubbles are substantially smaller than the channel diameter.[66–69]

Fig. 6. Summary of the optical sensor applicability for monitoring various types of gas-liquid reactions.

Fig. 6 summarises the findings of the sensor applicability for gas-liquid reactions based on the gas behaviour during the reaction: equimolar substitution, evolution, or consumption. Gas-"neutral" reactions (Fig. 6A) such as carboxybenzyl hydrogenolysis deprotection (where 1 mol of H_2 is consumed and replaced with 1 mol of CO_2) seem unfeasible to monitor with the optical sensor because the LF readings do not change during the reaction. The nature of the gas, however, may be used to overcome the problem. A possible solution in this example is dissolving CO_2 in the liquid media at a high pressure or adding a gas-absorbing alkaline solution. In these cases, the simplicity of the optical sensor may be counterbalanced by complexity introduced by the additional gas-absorption system.

Gas-evolving reactions (Fig. 6B) such as decarbonylation can be monitored easier with the optical sensor because the LF changes substantially during the reaction. The strong dependence of the LF readings on the liquid flow rate and slug length (Fig. 5), however, require additional efforts for

7

process monitoring. The optical sensor must be calibrated for the required flow rates with, possibly, slug length control by image analysis.

Gas-consuming reactions (Fig. 6C) such as hydrogenation seem the most suitable for monitoring with the optical sensor. Reliable readings in the LF range of 95-97%, possible without sensor calibration, are beneficial in ensuring almost complete gas consumption. A few remaining hydrogen bubbles saturate the solution and minimise side-reaction and catalyst leaching.[50,51] A minor hydrogen content in the product feed maximises residence time and the reactor throughput. In summary, the negative consequences discussed in Table 1 are eliminated.

In the work, we used the optical sensor to monitor hydrogenation reactions because these gas-consuming reactions are vital in many fine chemical syntheses.[1,3] The experimental system presented in Fig. 1 combined the flows of (i) H_2, (ii) a solvent and (iii) a substrate solution. Following the unsuccessful attempts to control the MBY semi-hydrogenation by complete H_2 consumption in Fig. 3, we used the optical sensor to monitor and maintain high (but not complete) H_2 consumption with the same 2.3 wt% Pd/C catalyst-coated tube.

Optimisation was performed using a proportional-integral (PID) algorithm. The LF reading was a process variable (setpoint of 95%), and the MBY concentration was a control variable adjusted to reach the setpoint. Compared to conventional optimisation algorithms such as simplex,[28,29,70] the PID algorithm does not assume implacable experimental reproducibility and can handle slow changes in the reaction performance caused by usual phenomena such as catalyst activation/deactivation.[21,71–73] The flow rates of H_2 and solvent were also adjusted by the algorithm, but these were linked to the MBY flow rate by the pre-defined H_2 to MBY molar feed ratio and the total liquid flow rate. After reaching the LF setpoint, the system collected the 4 liquid samples for the offline GC verification.

Fig. 7A shows the change in the MBY and H_2 flow rates over the time on stream in the system controlled with an optical sensor. The PID algorythm, using the LF readings, automatically adjusted the MBY flow rate to maintain the LF at 95%. During the experiment, the H_2/MBY feed ratio increased stepwise and the LF values dropped momentarily to about 80%. The diminishing LF was compensated by the system with decreasing MBY flow rates allowing for a longer MBY residence time to consume more H_2.

Once the LF readings stabilised, the system collected 3 liquid samples for each specified H_2/MBY feed ratio. The conversion and selectivity for the samples collected was reproducible within ±0.4% which shows excellent process control achieved with the optical sensor. The pre-defined H_2/MBY feed ratio agreed with the experimental consumption calculated from the product composition (Fig. 7A). The increasing H_2 consumption resulted in a higher MBY conversion but the MBE selectivity decreased due to depletion of MBY species in the solution. Importantly, the MBE yield was constant at around 92% in the H_2/MBY feed ratio range from 100 to 109% because the increasing conversion was compensated by the decreasing MBE selectivity. The H_2/MBY feed ratio above 109% resulted in the declining MBE yield because the conversion could no longer be increased (being close to 100%), but the MBE selectivity decreased due to over-hydrogenation with the available H_2.

8

optical sensor and PID reactor control in maintaining the product Liquid Fraction to 95 %. (B) The effect of the H_2/MBY feed ratio on the product composition. Concentration 0.39-0.43 M, 70 °C and 2.5 bar.

To validate the process control with the optical sensor, we studied hydrogenation of 3-hexyne-1-ol, a widely used fragrance compound and a model molecule for internal alkyne semi-hydrogenation.[74–76] The possible hydrogenation reactions are shown in Fig. 8A.

Fig. 8. Schemes of (A) 3-hexyne-1-ol, (B) 1,4-butyndiol, and (C) dehydrolinalool hydrogenation reactions.

Hydrogenation of 3-hexyne-1-ol was studied in 2.3 wt% Pd/SiO_2 and 2.3 wt% Pd/ZnO catalysts-coated tubes. The Pd/ZnO catalyst often provides a higher alkene selectivity in the alkyne semi-hydrogenation.[22,23,55,56,77] Compared to MBY (Fig. 7B), the range of 3-hexyne-1-ol hydrogenation products was wider and a significant E-alkene formation was observed caused by Z/E isomerisation.[74–76] The increasing H_2/substrate feed ratio over the Pd/SiO_2 (Fig 8A) catalyst resulted in a higher alkyne conversion (from 80 to 85%) but lower Z-alkene selectivity (from 64 to 55%). The highest yield of the Z-alkene was 50.5 % at the H_2/substrate feed ratio of 100 %. The results observed over the Pd/ZnO catalyst (Fig. 9B) confirm the expected Z-alkene selectivity increase, which reached 70 % with the maximum yield of 62 % at the H_2/substrate feed ratio of 100%.

9

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Quality by Design encouraged by the US Food and Drug Administration (FDA) in continuous flow synthesis requires tight monitoring of all the reaction input and output parameters to improve reproducibility and eliminate the process rejects. Reaction monitoring, however, relies on costly (over 10000$) process analytical technology (PAT)-one of the factors that prevents the wider utilisation of ... [Show full abstract]

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Hydrogenation of an imine (N-Cyclohexyl(benzylidene)imine) into a secondary amine (N-Benzylcyclohexylamine) was studied in catalyst-coated tube reactors to utilize the advantages of continuous-flow processes. Tetrahydrofuran (THF) was found to be an optimal solvent providing high reaction and low catalyst deactivation rates compared to toluene and isopropanol. Even in THF, however, the ... [Show full abstract]

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Active site isolation in furfural (FA) hydrogenation was studied by poisoning a Pd catalyst with bismuth. A solution of FA in water was hydrogenated over a 5 wt% Pd/SiO2 catalyst in a batch reactor at various reaction temperatures and pressures. Furfuryl alcohol (FAL) was an intermediate product which was further hydrogenated into tetrahydrofurfuryl alcohol (TFAL) or cyclopentanone (CPA) and ... [Show full abstract]

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