Corning® Advanced-Flow™ Reactor Webinar Series Webinar 4: "How to Leverage Corning® Advanced-Flow™ Reactor Technology for Photochemistry Applications"

Tuesday, Jan. 26 - 10am Central European Time | Thursday, Jan. 28 - 11am Eastern Standard Time



Questions & Answers

Q1: What is the best way to start an evaluation with photochemical systems for reactions involving nitrogen compounds?

A: It depends which sort of nitrogen compounds we are talking about. If they are unstable or explosive and light could trigger them, the first thing is to investigate this effect, run a UV spectrum, and thoroughly understand the photosensitivity of the molecule.

The traditional way to approach a new system is to first identify the best wavelength for transformation. Then, identify the ideal concentration based on the absorbance of the system/light emission of the medium. Next, consider the kinetics of the system.

For a specific process, we are more than happy to discuss this in more detail. Feel free to contact us at <u>AFRwebinars@corning.com</u>.

Q2: At a particular wavelength if one reaction is exothermic and another reaction is endothermic, then in that case how do you match chemistry and wavelength?

A: Our reactors are equipped with a heat exchange system allowing you to accurately control the temperature of your medium. You also have the option to have multiple heat exchange zones allowing you to control exothermic and endothermic reactions within the same reactor. Also, the flexibility of our photo reactor allows you to choose the wavelength to be used for each part of the reactor.

Q3: We have Corning Low-Flow Reactor and are thinking of buying a Lab Photo Reactor. Does it contain LED of all categories? For any new reaction, what would be the starting point?

A: Each Lab Photo Reactor panel integrates six different wavelengths. We have two different panels available that are easy to exchange. The first, standard panel is equipped

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with 365 nm, 385 nm, 405 nm, 470 nm, 610 nm and 4000 K (white). The second panel is equipped with 340 nm, 375 nm, 395 nm, 420 nm, 455 nm and 530 nm. The selection of wavelengths has been made based on which are the most commonly used.

To answer the second question, when starting in photochemistry, the first step should be to measure the absorption spectrum of your molecule in order to learn which wavelength should be used for your chemical reaction.

Q4: Do you have other wavelengths available on top of the ones you showed in the presentation?

A: In principle, as soon as an LED is available on the market, we may have the opportunity to build a custom-made panel with the corresponding wavelength. Considering the current glass limitations and the available LED technology we currently do not provide wavelengths below 340 nm.

Q5: Based on your experience with industrial customers, how do photochemistry systems impact CapEx ratios compared to usual approaches?

A: It really depends on the type of process and on how the technology can improve the process parameters. Generally speaking, the CapEx ratios are improved if it is possible to shorten or simplify the synthesis process, and consequently less equipment is needed for the process.

Also, the costs linked to the downstream processes, separation and purification steps need to be considered as they will be impacted by the quality of the product stream in a flow process.

Other cost factors to consider are all costs linked to safety equipment and procedures, the footprint of the full process, energy consumption, reduced waste handling, and higher levels of automation.

Q6: What are the limitations in terms of glass and Heat Exchange (HE) fluid absorption?

A: Each flow module has three layers and the reaction pathway is sandwiched between the Heat Exchange pathway layers. Therefore, the HE fluid must not absorb any radiation that comes from the LED. To do this, check the UV spectrum from the oil supplier. Corning® Advanced-Flow™ Reactor Webinar Series Webinar 4: "How to Leverage Corning® Advanced-Flow™ Reactor Technology for Photochemistry Applications" Tuesday, Jan. 26 – 10am Central European Time | Thursday, Jan. 28 – 11am Eastern Standard Time

We recommend regularly checking (e.g. twice a year) that the oil does not degrade over time. A quick UV spectrum measurement should indicate if any changes occurred in the oil.

The flow modules are borosilicate glass and its absorption (peak-off frequency) is about 300 nm. However, for the time being, LED technology is lagging behind and LEDs emitting below 365 nm are scarce and pricey. The technology is evolving but not there yet.

Q7: In your experience, is there any change in wavelength choice when switching from batch to flow?

A: If no chemical is changed during the process change from batch to flow, then the chemistry will remain the same whether performed in batch or flow. Therefore, we do not anticipate that a change in wavelength will be necessary.

Swapping a process from batch to flow could, however, be a good opportunity to change the light sensitive catalyst for a more cost efficient or effective alternative. This could include a shorter lifetime and performance that is more compatible for flow chemistry.

Q8: What is the maximum pressure available in the system?

A: The maximum pressure of Advanced-Flow Reactors is 18 bars.

Q9: Is it possible to use a gas feed in the G1-G3 series?

A: Yes, it is. Advanced-Flow Reactors can handle gases very well. Gases such as O₂, HCl, H₂, etc. are commonly used with our technology. You can check some literature available on our website, such as photo oxidation using singlet oxygen: ((<u>https://www.corning.com/media/worldwide/Innovation/documents/AFR/AFR%20Public ations%20Nov.%202020_v2.pdf</u>).

Q10: How difficult is it to perform photooxidation reactions using your platforms?

A: Photooxidation is a topic that has been widely explored by Dr. Jean-Christophe Monbaliu at our AFR Qualified Lab, Citos, in Belgium using Corning reactors. You can check publication references on either our website (<u>www.corning.com/reactors</u>) or Citos' website (<u>http://www.citos.uliege.be</u>).

Q11: How effective is the system for multiphase processes such as gas and liquids?

A: The HEART shape of our fluidic modules has been specially designed to overcome multiphasic mixing issues. Our mixing behavior is about 100 times better than a conventional stirred batch reactor. You can check literature on our publication list on our website related to mixing characterization

(https://www.corning.com/media/worldwide/Innovation/documents/AFR/AFR%20Publica tions%20Nov.%202020_v2.pdf).

Also, a great example that shows the benefit of our technology for multiphasic systems in flow photochemistry can be found in the following article:

Roibu, A.; Horn, C. R.; Van Gerven, T.;Kuhn, S. Photon Transport and Hydrodynamics in Gas-Liquid Flow Part 2: Characterization of Bubbly Flow in an Advanced-Flow Reactor. ChemPhotoChem 2020, 4 (10), 5193–5200. https://doi.org/10.1002/cptc.202000066

Q12: Can Corning's software be controlled by the IKA Labworldsoft?

A: Our reactors don't require any additional software. Only the LED control panel uses software, which cannot currently be connected to any other software. Work is ongoing to update it to allow external recovery of data.

It would be better that If the LED and reactor temperature can be controlled by the Software and the data can be automatically logged and saved.

A: We agree, and believe this could be achievable as temperature control relies directly on the heat exchanger software. For the LED control, as said before, work is ongoing for a new version with an external storage option.

Q13: Is there a secondary containment option for toxic chemicals?

A: As the system is closed (the reaction takes place in the reactor), it is confined and should avoid related issues. Risk may come from using or collecting hazardous reagent as they will need to be stored and manipulate to prepare the experiment. Any of these being toxic could be a hazard, but could be mitigated by generating dangerous reagents in situ. For the products, it is important to keep the chemicals in check (in a safe collection flask) and perform a risk and safety analysis beforehand.

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The reactor should also be stored in a safe place (hood, closed cabinet, etc.) which will act as a secondary containment system. Safety aspects are related to each process and each local regulation. We have examples of energetic materials synthesis handling with our technology. Our Corning AFR Qualified Lab in the US, Nalas Engineering (<u>www.nalasengineering.com</u>) is specialized in handling such hazardous compounds and can provide more advice on this particular point.

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