EQUILIBAR® PRECISION FLUID CONTROL

For immediate release: **August, 2022**

Equilibar® Blockage Resistant Back Pressure Regulator Used in Continuous Flow Supercritical CO2 Metal-Organic Framework Nanomaterial Synthesis Reactor

Summary

Researchers in the Mechanical Engineering department at the University of Washington in Seattle are using an Equilibar blockage resistant Back Pressure Regulator (BPR) to maintain stable pressure in a nanomaterial synthesis reactor near the critical point of CO2. The goal of this research is to study how the tunable thermodynamics of supercritical phase fluid impact the sustainable and scalable synthesis of a class of porous nanomaterials called "metal-organic frameworks" or MOFs. The high surface areas and porous structures of MOF materials offer desirable capabilities in various applications such as wound healing, hydrogen storage, and filter membranes for water and air purification. The tunable thermodynamic properties near the critical point allow for a wide range of synthesis conditions with small changes in reactor settings of temperature, pressure, and flow rate.

Challenge

A significant challenge to adapting MOFs for practical application is that the laboratory techniques used to discover MOFs generally are not amenable for scaling up the production. In 2020 Rasmussen et al. published an innovative method to scale MOF nanoparticle synthesis using continuous flow supercritical carbon dioxide (SCO2).[1] To increase production rates and robustness of the reactor, including eliminating dry ice formation due to throttling and improving pressure stability, a spring-loaded back pressure regulator was replaced with an Equilibar dome-loaded BPR which also allowed removal of the particle filter so that the nanoparticles could be collected in a flask downstream of the BPR. [2]

Solution

Elizabeth Rasmussen, PhD candidate at the time these studies were conducted, worked with Equilibar engineers to determine the best back pressure regulator design and components to meet the needs of the process. The process required an operating pressure up to 6,000 psi processing supercritical CO2, so they chose an Equilibar B6R Blockage Resistant Back Pressure Regulator with a PEEK diaphragm and Kalrez O-Rings. Figure 1 shows the schematic of the supercritical carbon dioxide reactor system with the Equilibar back pressure regulator downstream of the reactor providing precise, consistent pressure control.

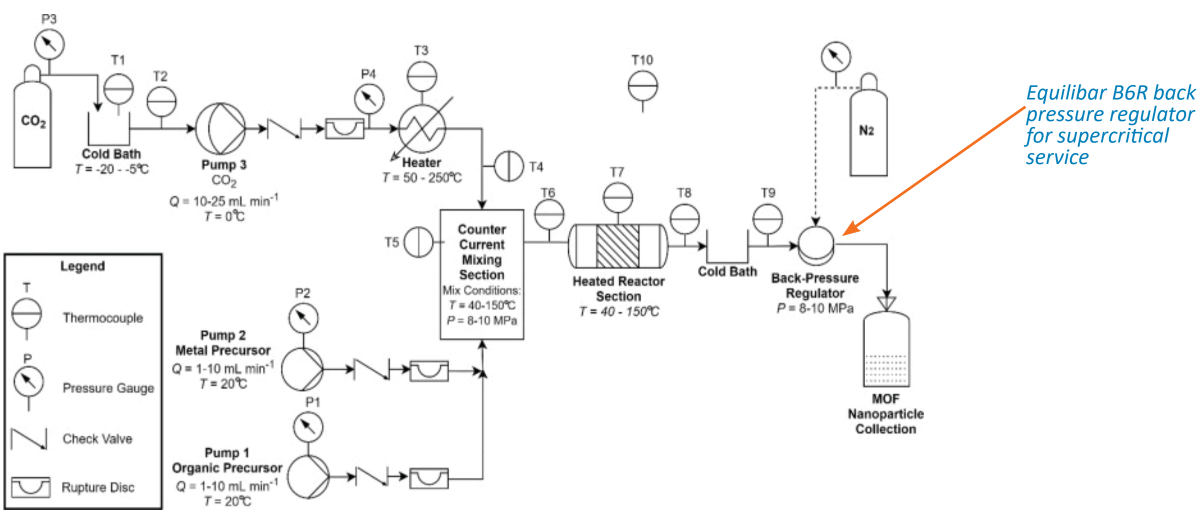

Figure 1. Schematic of the continuous flow supercritical carbon-dioxide metal-organic framework (MOF) nanoparticle synthesis reactor system.[2]

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Solution Continued:

The Equilibar® B6R back pressure regulator is a variation of the popular Equilibar research series BPR. The B6R integrates technology to optimize for supercritical applications. These features maintain high precision control while reducing the Joule-Thomson effect of supercritical process fluids passing through the outlet orifices. The key design feature of this regulator series is a downstream expansion cavity that provides a continuously increasing cross sectional area to eliminate convergence points for ice build-up during decompression. Specially designed O-ring grooves reduce stress during rapid decompression which increases O-ring life.

Figure 2. Equilibar B6R Blockage Resistant BPR

The B6R delivered a pressure accuracy of ± 2 %. Figure 3 is a photograph of the Equilibar B6R in the operating reactor system. A nitrogen gas cylinder was used to supply the pilot setpoint to the dome-loaded BPR. A video walk-through of the laboratory reactor and demonstration of the back pressure regulator operating can be seen in a YouTube video titled, "Synthesis of metal-organic framework (MOF) via continuous flow supercritical carbon dioxide reactor"(URL: *https://youtu.be/-p-SL80W8-E)*

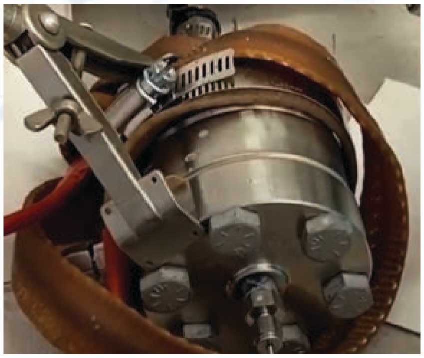

Figure 3. Equilibar B6R installed in the MOF reactor system

References

[1] E.G. Rasmussen, J. Kramlich, I.V. Novosselov, Scalable Continuous Flow Metal–Organic Framework (MOF) Synthesis Using Supercritical CO2, ACS Sustainable Chemistry & Engineering 8(26) (2020) 9680-9689. https://doi.org/10.1021/acssuschemeng.0c01429.

[2] E.G. Rasmussen, J. Kramlich, I.V. Novosselov, Synthesis of metal-organic framework HKUST-1 via tunable continuous flow supercritical carbon dioxide reactor, Chemical Engineering Journal 450 (2022) 138053. https://doi.org/10.1016/j.cej.2022.138053.

Contact Equilibar

Equilibar is a provider of unique and innovative pressure control solutions based near Asheville North Carolina. Equilibar's patented pressure regulator technology is used in a wide array of processes including catalyst, petrochemical, sanitary, supercritical and other industrial applications. For more information please contact an Equilibar applications engineer at inquiry@equilibar.com or 828-650-6590.

About the Author

Elizabeth Rasmussen, PhD is a former graduate student at the University of Washington – Seattle in the mechanical engineering department. While at the University of Washington, she was an National Science Foundation data science accelerator award recipient and a graduate research fellow at the Clean Energy Institute. Rasmussen's research interests center around creating instruments to understand and optimally apply a material's thermophysical properties.

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