Angewandte Chemie International Edition

**Catalysis in Flow**

International Edition: DOI: 10.1002/anie.201500841
German Edition: DOI: 10.1002/ange.201500841

# Composite Polymer/Oxide Hollow Fiber Contactors: Versatile and Scalable Flow Reactors for Heterogeneous Catalytic Reactions in Organic Synthesis**

*Eric G. Moschetta, Solymar Negretti, Kathryn M. Chepiga, Nicholas A. Brunelli, Ying Labreche, Yan Feng, Fateme Rezaei, Ryan P. Lively, William J. Koros, Huw M. L. Davies,* and Christopher W. Jones**

**Abstract:** *Flexible composite polymer/oxide hollow fibers are used as flow reactors for heterogeneously catalyzed reactions in organic synthesis. The fiber synthesis allows for a variety of supported catalysts to be embedded in the walls of the fibers, thus leading to a diverse set of reactions that can be catalyzed in flow. Additionally, the fiber synthesis is scalable (e.g. several reactor beds containing many fibers in a module may be used) and thus they could potentially be used for the large-scale production of organic compounds. Incorporating heterogeneous catalysts in the walls of the fibers presents an alternative to a traditional packed-bed reactor and avoids large pressure drops, which is a crucial challenge when employing microreactors.*

Organic synthesis by flow chemistry is an emerging concept in fine chemistry and pharmaceutical production.[1–6] Commercial flow reactors for organic synthesis are available from numerous vendors and range from μL to L in volume.[2,3,7,8] These reactors offer advantages over batch reactors, such as operation at high temperatures and pressures, improved heat and mass transfer, and safer handling of toxic and explosive materials.[2–4,9] As the surface area-to-volume ratio of the reactor increases, the heat and mass transfer performance of the reactor improves, which is critical for consistent product yield and selectivity in a continuous process.[2] A drawback associated with some commercially available flow reactors and microreactors is the potential for a high pressure drop along the length of the reactor, because of poor handling of solid species, which can clog the flow channels and reduce the rate of production.[4,9–15] Therefore, alternative reactor types that avoid large pressure drops but accommodate supported catalysts for complex organic reactions while maintaining superior heat and mass transfer properties are desired.

We have begun a program to explore the possibility of using flow chemistry in the emerging field of C–H functionalization. The goal of the program is not only to demonstrate that C–H functionalization reactions can be conducted in flow,[6,16–31] but also to design new types of flow reactors to handle supported catalysts that are often used for such reactions (Figure 1). Herein, we demonstrate that hollow composite fibers offer great flexibility for catalyst immobilization and application in flow chemistry. This is demonstrated by creating hollow fibers that contain three different types of catalysts: 1) active solid oxides (zeolites), 2) silica-tethered organocatalysts, and 3) silica-tethered organometallic catalysts (Scheme 1), and use of these fibers in reactions important in organic synthesis.

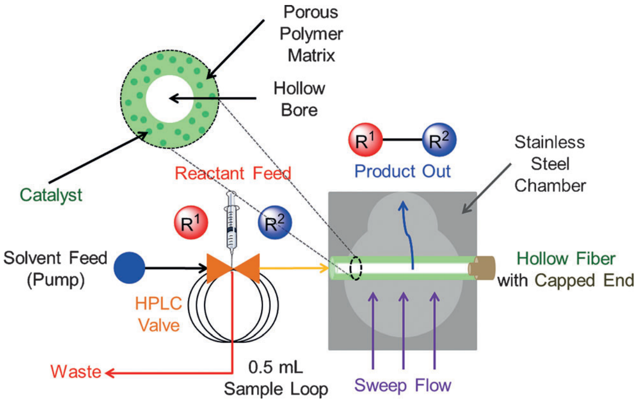

***Figure 1.*** Schematic description of the radial flow hollow fiber flow reactor.

The two proof of concept studies employed simple catalysts in straightforward reactions: the Brønsted acid catalyzed deprotection of benzaldehyde dimethyl acetal and a Lewis base catalyzed Knoevenagel coupling reaction. The main focus of the study was then directed at enantioselective dirhodium(II)-catalyzed cyclopropanations, a method used for making important ligands in C–H functionalization chemistry, and then to a key C–H functionalization reaction useful in organic synthesis. A fiber-supported analogue of the

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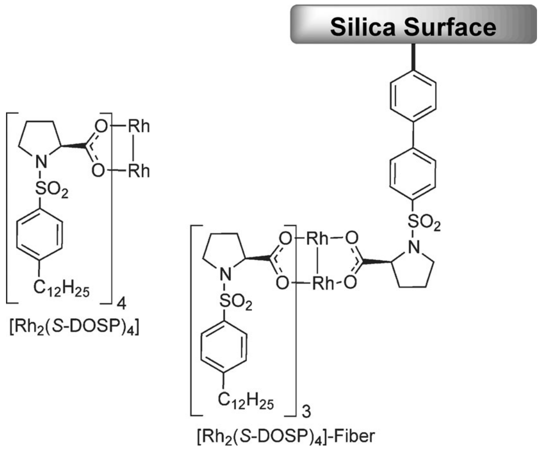
[*] Dr. E. G. Moschetta, Dr. N. A. Brunelli, Dr. Y. Labreche, Dr. Y. Feng, Dr. F. Rezaei, Prof. R. P. Lively, Prof. W. J. Koros, Prof. C. W. Jones
School of Chemical & Biomolecular Engineering
Georgia Institute of Technology
311 Ferst Drive NW, Atlanta, GA 30332 (USA)
E-mail: cjones@chbe.gatech.edu

Dr. S. Negretti, K. M. Chepiga, Dr. N. A. Brunelli, Prof. H. M. L. Davies
Department of Chemistry, Emory University (USA)
E-mail: hmdavie@emory.edu

[**] We thank the NSF under the Center for Selective C-H Functionalization (CCHF), No. CHE-1205646.

Supporting information for this article (including details of the flow reactor setup, residence time and space time yield calculations, fresh and used fiber characterization, diazo synthesis, and product characterization) is available on the WWW under http://dx.doi.org/10.1002/anie.201500841.



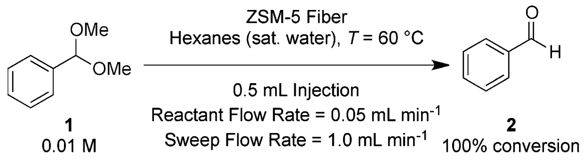
***Scheme 1.*** Homogeneous $[Rh_2(S\text{-DOSP})_4]$ and its immobilized analogue on silica. The other catalyst structures are shown in the Supporting Information.

powerful $[Rh_2(S\text{-DOSP})_4]$ catalyst is the centerpiece of this investigation. While cyclopropanations/cyclopropenations have been investigated using flow systems,[18–20,26,28] no such investigations have occurred using hollow fiber reactors, nor have there been studies of dirhodium(II)-catalyzed asymmetric intermolecular C–H functionalization reactions in flow, to the best of our knowledge.

Polymeric hollow fibers are commercially used for industrial gas separations, namely separation and purification of $CO_2$, and provide high surface areas at low cost.[32] The polymeric hollow fibers used here were prepared in a continuous process whereby the solid oxide particles were incorporated into the porous polymer matrix during the fiber spinning, thereby allowing fibers to be made rapidly at low cost using commercially available materials.[32] The small inner (ca. 300 μm) and outer (ca. 1100 μm) diameters of the fibers ensure that the walls are thin, which imparts excellent heat and mass-transfer properties.[33] In this case, the thin wall presents an extremely short reaction bed length when radial flow through the fiber wall is used. Recently, composite hollow fibers (organic polymers and inorganic fillers such as zeolites,[34] silicas,[35] and metal–organic frameworks (MOFs)[36]) have emerged as separation membranes and sorbents that offer highly tunable material properties. Previous work on hollow fiber reactors focused on Pd-catalyzed reactions and alkene/alkyne hydrogenations,[37–46] although, to the best of our knowledge, no examples of asymmetric reactions using well-defined organometallic complexes embedded in hollow fiber reactors in flow have been reported. In general, the few examples of flow catalysis using hollow fiber catalysts/reactors are for simple catalysts and reactions, with no examples in organic synthesis using well-defined asymmetric organometallic catalysts.

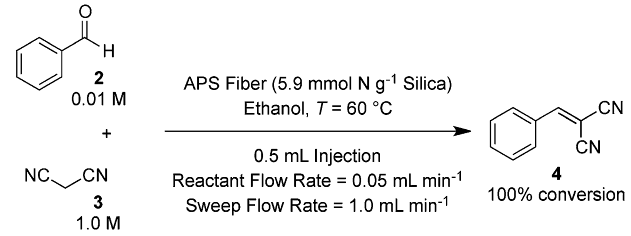
The polymer/oxide hollow fibers used herein were comprised of cellulose acetate, a common polymer used in commercial hollow fibers for gas separation. Two inorganic fillers were also used, an aluminosilicate MFI zeolite (ZSM-5) for Brønsted acid catalysis as well as commercially available porous amorphous silica on which molecular catalysts were tethered. The composite fibers were prepared by the well-established dry-jet, wet-quench, nonsolvent-induced phase-separation spinning method.[32] For Lewis base catalysis, 3-aminopropyl (APS) groups were grafted to the silica particles by a recently developed post-spinning amine infusion technique after spinning the hollow fibers.[35] Similarly, an analogue of the well-known $[Rh_2(S\text{-DOSP})_4]$ catalyst was grafted to the silica particle surface after fiber formation by using previously developed techniques, in which a $[Rh_2(S\text{-DOSP})_4]$ derivative containing a styryl group on a prolinate ligand was tethered to styryl-functionalized silica in the fiber wall through a radical coupling initiated by 2,2′-azobisisobutyronitrile (AIBN).[47] $[Rh_2(S\text{-DOSP})_4]$ was chosen because it is stable, and a highly active catalyst for a wide range of reactions of donor–acceptor diazo compounds, including stereoselective C–H functionalizations.[48]

The reactants are fed through the bore of the hollow fiber reactor (Figure 1). The fiber walls, composed of cellulose acetate and inorganic particles, are highly permeable. The walls are approximately 300–400 μm thick and can be considered an ultrashort catalyst bed through which the reactants pass. Outside the fiber is a sweep flow of an organic solvent that facilitates product collection. In this initial study, reactant pulses were flowed through the fiber, although continuous flow operation can also be conducted. The fiber was capped at the opposite end to ensure the reactants flow through the walls and contact the catalyst (tubular dead-end flow). To evaluate the viability of the reactor system, the Brønsted acid catalyzed deprotection of benzaldehyde dimethyl acetal (**1**) to benzaldehyde (**2**) was examined using hollow fibers containing the proton-exchanged form of zeolite ZSM-5. Scheme 2 demonstrates the conditions used to drive the reaction to completion. Water-saturated hexanes were used as the solvent to provide the trace amount of water needed for deprotection. Control experiments using bare fibers without ZSM-5 exhibited almost no conversion, thus demonstrating the ability of the catalytic fibers to achieve substantial conversion, even over a short catalyst bed.



***Scheme 2.*** Acid-catalyzed deprotection of **1** using zeolite ZSM-5 embedded in the walls of hollow fibers. Conversions were determined using GC and are the averages of three different fibers.

Next, APS-functionalized silica particles embedded within the fiber walls were used as catalysts for the Knoevenagel condensation of benzaldehyde (**2**) and malononitrile (**3**). Scheme 3 demonstrates that the reaction can be driven to completion by using an excess of malononitrile. Control experiments using bare fibers without APS-functionalized silica showed that the conversion decreased by a factor of about two in the absence of the amine catalyst, thus suggesting that the bare cellulose acetate/silica fibers may provide some background activity for this reaction as well.

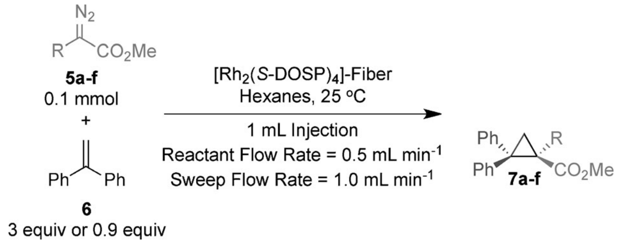


***Scheme 3.*** Base-catalyzed Knoevenagel condensations of **2** and **3** using APS-functionalized silica embedded in the walls of hollow fibers. Conversions were determined using GC and are the averages of three different fibers.

In these reactions, the residence time was estimated to be 10–42 s at a flow rate of 0.05 mL min$^{-1}$. In this initial test—reactions employing simple acid and base catalysis for straightforward reactions—the hollow fibers enable quantitative conversion of the reactants at short residence times.

Having established the catalytic capability of the composite fibers in simple reactions, the main focus of the study was applying the hollow fiber concept to a silica-tethered version of the well-known [$Rh_2(S\text{-DOSP})_4$] catalyst. The first reaction examined was the asymmetric cyclopropanation of 1,1-diphenylethylene (**6**) with various donor–acceptor diazo compounds (**5a**–**f**).[49] These substrates were chosen because of the importance of the triarylcyclopropane carboxylate products in the design of novel dirhodium(II) catalysts that promote high asymmetric induction in carbenoid reactions.[50–52] The data in Table 1 demonstrates the breadth of diazo compounds that were successfully used to give the products with comparable, but slightly diminished, enantioselectivities (e.r.) and yields to the complementary batch reactions.[47,50]

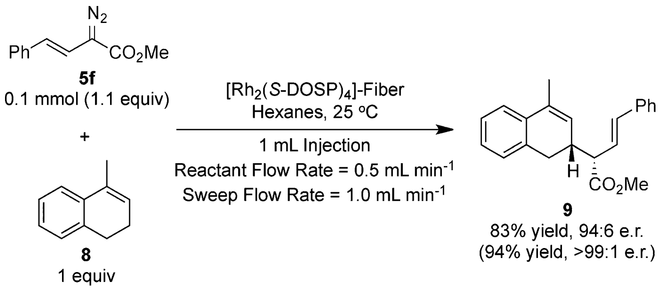
***Table 1:*** Scope of cyclopropanations of donor–acceptor diazonium compounds (**5a**–**f**) and **6** using supported Rh catalysts embedded in the walls of hollow fibers.



| Entry | 5 | R | Yield [%][a–c] | e.r.[a–c] |
|---|---|---|---|---|
| 1 | **5a** | Ph | 77 (87)[d] | 96:4 (98:2)[d] |
| 2 | **5b** | $p$-$BrC_6H_4$ | 77 (88)[e] | 94:6 (99:1)[e] |
| 3 | **5c** | $p$-$MeOC_6H_4$ | 61 (70) | 98:2 (>99:1) |
| 4 | **5d** | $p$-$CF_3C_6H_4$ | 64 (59) | 97:3 (99:1) |
| 5 | **5e** | $o$-$ClC_6H_4$ | 66 (69) | 93:7 (98:2) |
| 6[f] | **5f** | $PhCH{=}CH_2$ | 68 (81) | 96:4 (96:4) |

[a] Yield of isolated **7a**–**f**. [b] Values without parentheses refer to [$Rh_2(S\text{-DOSP})_4$]-fibers. [c] Values in parentheses refer to homogeneous [$Rh_2(S\text{-DOSP})_4$]. [d] From Ref. [47]. [e] [$Rh_2(R\text{-DOSP})_4$].[50] [f] 0.9 equiv of **6** used.

To further demonstrate the utility of the fiber-supported [$Rh_2(S\text{-DOSP})_4$] catalyst, a classic C–H functionalization, the sequential combined C–H functionalization/Cope rearrangement (CHCR)[53]/retro-Cope rearrangement[54] of 1-methyl-3,4-dihydronaphthalene (**8**) and styryl diazoacetate (**5f**), was conducted (Scheme 4). A similar enantioselectivity was obtained as reactions in batch,[47] with slightly lower yield in flow. Successful completion of such robust organic transformations in flow is promising for the construction of natural products and other organic compounds[55] using the hollow fibers.



***Scheme 4.*** C–H insertion of **8** by **5f** using the [$Rh_2(S\text{-DOSP})_4$]-fibers compared to homogeneous [$Rh_2(S\text{-DOSP})_4$] (values in parentheses).[54] The product (**9**) is formed by CHCR and subsequent retro-Cope rearrangement.

To examine the stability of the dirhodium(II) catalyst, the use of the Rh-functionalized fibers over multiple reactions was probed (Figure 2). Although these Rh carbene catalysts are known to deactivate,[56] good e.r. values were obtained over more than 1000 turnover numbers (TON) in the cyclopropanation of methyl phenyldiazoacetate (**5a**) and **6**. Initially, the e.r. was 94:6 for both fibers studied, but gradually decreased to 92:8 as the TONs exceeded 1000.

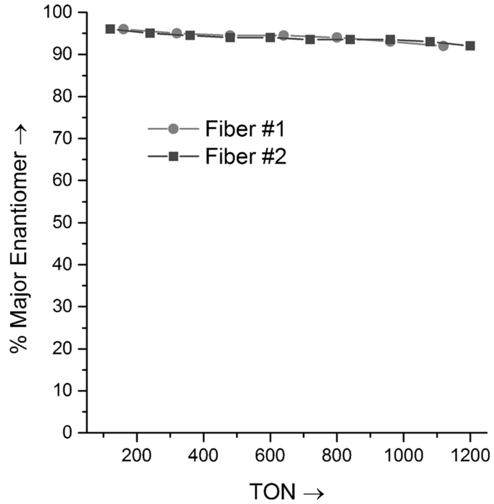


***Figure 2.*** The effect of increasing the TON on the e.r. value of the dirhodium(II)-catalyzed cyclopropanation of **6** by **5a**. Two different [$Rh_2(S\text{-DOSP})_4$]-fibers were used to demonstrate the reproducibility. Each data point refers to a 1 mL injection of 0.1 M of **5a** and 0.3 M of **6** at a flow rate of 0.5 mL min$^{-1}$ and a sweep flow of 1.0 mL min$^{-1}$. Reactions were performed at 25 °C in hexanes.

These examples illustrate three types of active sites (oxide particle surface, oxide-grafted organic site, oxide-grafted organometallic site) covering three types of reactions important in organic synthesis (deprotections, simple C–C couplings/condensations, and highly selective asymmetric reactions). The versatility of the types of catalysts incorporated into the hollow fibers as well as the reactions catalyzed within the hollow fiber flow system demonstrates that the composite hollow fiber platform may be a useful new approach to conducting flow liquid-phase synthesis of organic molecules. Future studies will be directed towards the application of the composite hollow fibers to a variety of C–H functionalization methods, with the eventual goal of enhancing the transformations compared to the current batch reactions and to develop more practical procedures when highly reactive reagents are involved.[57–62]

**Keywords:** flow chemistry · heterogeneous catalysis · hollow fibers · organic synthesis · rhodium

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Received: January 28, 2015

## Communications

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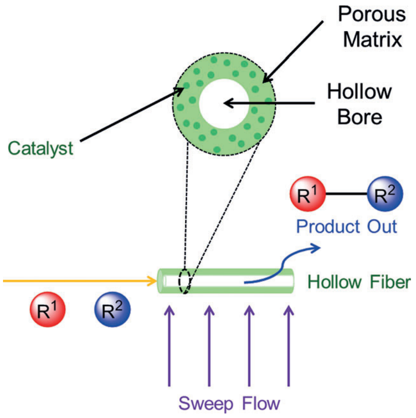
### Composite Polymer/Oxide Hollow Fiber Contactors: Versatile and Scalable Flow Reactors for Heterogeneous Catalytic Reactions in Organic Synthesis



**Go with the flow**: Composite polymer/oxide hollow fibers have been used as a flow reactor platform for heterogeneously catalyzed reactions in organic synthesis. Embedding catalysts in the fiber walls avoids clogging of the flow channels and provides a short catalyst bed. Three different catalysts and reaction types were successfully performed in flow, thereby demonstrating the versatility of the fibers as reactors.