Organic Process Research & Development **2008,** *12,* 771–777

High Efficiency Nondispersive Reactor for Two-Phase Reactions

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Abstract:

A novel continuous static reactor concept improves mass transfer in two-phase chemical reactions between 1 and 2 orders of magnitude. The current paradigm for chemical reactions between two liquids requires energy-intensive mixing to disperse droplets of one phase in the continuous phase. The new reactor contacts a continuous free phase with a continuous phase constrained to a multitude of fibers. Phases come out of the reactor cleanly separated. An etherification reaction, a dehydrochlorination reaction, a transesterification reaction, and a vegetable oil neutralization were evaluated in fiber reactors. High conversions of starting materials to products were observed in minutes. Benefits of the new reactor complement the benefits of phase transfer catalyzed reactions.

1. Introduction

A novel reactor design will advance the state of the art for two-phase chemical reactions.[1] Current chemical reactors create interfacial surface area by energy intensive mixing to disperse droplets about 100 μm in diameter of one phase in the other phase (Figure 1).[2] Preliminary experiments indicate fiber reactors (FR) will generate superior contact area for two-phase reactions without creating these energy-consuming dispersions.

Dispersion is the current paradigm for chemical processing equipment for both reaction and for washing. While good dispersion is desired to generate the surface area for high production rates, there is the problem of how to separate that dispersion to obtain purified product. After reaction and washing, the dispersed particles must reform (coalesce) into a bulk phase for further processing. When surfactants or other surface active species are involved, coalescing may require long settling time, mechanical separation methods, or chemical separation aids. Dispersions can form that are very difficult to handle in a continuous production environment causing water pollution problems and worker exposures. Separations can also be energy intensive. The fiber reactor should have compelling advantages for energy and material-efficient chemical processes.

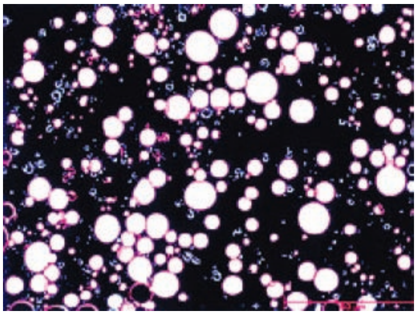

Figure 1. **Dispersion droplets.**

A recent report by Clark et al. described one pharmaceutical pilot plant process with 12 phase separations.[3] The complexity of medicinal syntheses makes it imperative that efficient processes are used wherever possible. Problems of dealing with two-phase reactions are common for process chemists. Leng recently discussed transition of specialty chemical processes from laboratory to plant and observed that two aspects of the scale-up of two-phase reactions and solute extractions are:[4]

- "If you do not see an emulsion in the lab, you will likely see one in the plant."
- "If you see an emulsion in the lab, it will likely be worse in the plant."

A new, nondispersive paradigm will benefit chemical and pharmaceutical processing. The fiber reactor results reported here challenge the existing paradigm that dispersion of small droplets is the only commercially feasible way to cause reactions between molecules in different phases.

The major barrier that prevents significant technical advances in the bioenergy, bulk chemical, specialty chemical, and pharmaceutical industries is the poor mass transfer between phases in current reactor designs. This limits the rate of overall production. Current industrial processes for contacting two immiscible phases apply mechanical energy to reduce particle size. This increases the production rate of the desired species in the two phases. Reaction between two phases is mass-transfer limited, meaning that the production rate is limited by the transfer of a reactant from one phase to the other phase where it may react rapidly. The smaller the particle size, the greater the contact area and the greater the production rate—unless an emulsion forms and the particles stop moving, at which time reaction can virtually cease. New reactor designs attempting to

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10.1021/op800010m CCC: $40.75 © 2008 American Chemical Society
Published on Web 06/11/2008

Table 1. **Two-phase process improvements**

	PTC reactions[11]	fiber reactions
increase productivity	increase yield reduce cycle time reduce unit operations increase reactor volume efficiency	continuous reactions eliminate separation time 60× interfacial area 60× faster reaction
improve environmental performance	reduce or replace solvent reduce nonproduct output	better phase separation eliminates rag layer
increase quality	improve selectivity reduce variability	continuous process produces consistent quality
enhance safety	control exotherms use less hazardous raw materials	low chemical inventory in fast continuous reactor
reduce other manufacturing costs	eliminate workup unit operations use less expensive raw materials	less complex process

solve the poor mass-transfer problem make microcontact between two phases.[5]

A second technical barrier is slow settling dispersions. Slow settling arises as a result of dispersion of liquids into micrometer-sized particles that coalesce slowly, or not at all, because of density or similarity.

Several undesirable side effects occur in liquid−liquid mixing processes because of the tendency of certain chemical reaction mixtures to form stable dispersions:

(1) Separation of the two liquid phases becomes difficult, and each phase can be contaminated with the other phase. Crystal clear phases are desirable to reduce contamination and excessive washing.

(2) Phase contact time is extended by slow settling times, causing yield loss in processes with hydrolyzable reactants. Minimum phase contact time is desirable for minimum loss.

(3) Extra washing steps may be needed, causing more environmental pollution and using more energy. The fewer steps the better.

(4) Frequently, a third layer of unseparated dispersion occurs between layers. This is called a rag layer, a region of stable or semistable emulsified reactants between two immiscible layers, which may not break during the process time frame, causing yield loss and waste disposal costs. Elimination of rag layers is highly desirable.

The time and effort needed to handle dispersions and emulsions generated in today's processes is very inefficient and costly for the industry. Conventional chemical and engineering approaches to solve the problem of poor mass transfer create other problems. Mass-transfer limitations can be reduced by using cosolvents to give homogeneous reactions that are much faster.[6] This requires solvent distillation, a recycle loop, larger reactors to accommodate solvent, and more energy. Plus, the dilution itself reduces the ultimate reaction rate. Higher temperature/high-energy processes cause faster reactions, but may require expensive equipment or high-pressure reactors.[7]

A third technical barrier hampering the specialty and pharmaceutical chemical industries is the need of some reactions for exotic and expensive reagents to effect a transformation. Phase transfer catalyst (PTC) organic reaction is a rapidly growing two-phase method for producing specialty organic compounds commercially because of significant benefits and capabilities.[8,9] PTCs are surface-active species that transfer reactants between immiscible phases. Being surface active by nature they also tend to help disperse one phase in another. PTC-based process phases may separate relatively slowly and may also form "rag" layers. Proper selection of PTC and use of solvents for density and hydrophobicity control can reduce the magnitude of phase separation problems. The fiber reactor can eliminate phase separation problems.

The benefits of PTC to industrial chemical companies could be enormous. Significant benefits and cost savings may be achieved by using PTC with an inexpensive inorganic base in place of expensive and hazardous sodium methoxide, sodium amide, sodium hydride, or *tert*-butoxide[10]

Table 1 summarizes the many potential process improvements available by converting to PTC reactions and to fiber reactors. The new nondispersive paradigm will complement PTC chemistry. Industry will have incentive to switch to PTC and the new fiber reactor simultaneously.

1.1. Fiber Reactor Principles. How can two liquids be brought into intimate contact without dispersing one in the other? Fibers can be made out of hydrophilic materials such as glass and metals. If aqueous caustic is poured over a bundle of parallel hydrophilic fibers, the fluid will form a film around the individual fibers because of surface tension and hydrophilicity (Figure 2). When a hydrophobic phase is induced to flow between the fibers, the phases do not mix, but they do have a large area of interfacial contact.

The fiber reactor is based on Merichem's Fiber−Film Contactor.[12] This design was introduced in the 1970s as an emulsion-free method to remove corrosive cresylic acids, naphthenic acids, and sulfides from petroleum products at the beginning of the refining process. It is conceptually similar to falling film reactors for gas−liquid contactors. It operates by contacting two immiscible films instead of dispersing discrete

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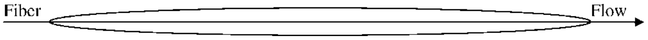

Figure 2. **Fiber wetting.**

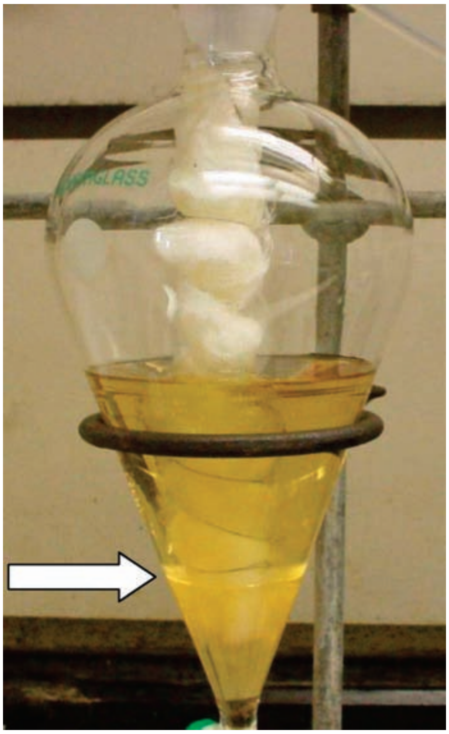

Figure 3. **Clear separation out of reactor.**

droplets of small size in a phase. The filming device, in a simple economical method, creates a very large interfacial surface area that is continuously renewed for efficient reaction between components of the two phases. Separation of the two phases at the end of the procedure is simple and effective because there are very few dispersed small droplets. This innovative process equipment combines the effect of mixing and phase separating into one simple step.

For contacting an aqueous phase in the fiber reactor, the fibers are first wetted by the aqueous phase. The aqueous phase is constrained to the hydrophilic fiber by surface tension. The aqueous phase may exist as a sheath around the fiber as shown in Figure 3.

The reactor consists of a tube that is filled with fibers oriented along the length of the tube.

If a fiber bundle is secured at the top and suspended tightly in a pipe, one will have the ability to pump aqueous caustic over the fibers continuously. With appropriate fittings at the top of the fiber bundle, one now has the ability to pump a hydrophobic petroleum fluid between the wetted fibers. Clonts discovered that this construction with a separator attached provides for rapid neutralization and extraction of acids from petroleum streams without dispersions.[12] The hydrocarbon free-flowing phase exits the fiber bundle as soon as the bundle exits the pipe. The constrained aqueous phase stays on the fibers until the fibers reach the aqueous interface in a separator and becomes part of that phase. Both phases are crystal clear (Figure 3) when the reactor is operated properly. Phase separation is instantaneous upon exiting the pipe, thus avoiding long settling times for these oil−water−surfactant systems. Suarez reported a design for removing HCl from reformer hydrocarbon streams that used a 2175 ton per day Fiber−Film unit.[13]

The Fiber−Film petroleum refining technology was a disruptive technology and has been installed in 72% of all new caustic refining installations built over the last 19 years.[14] Acid−base reactions are rapid compared to organic reactions, but still, the elegance and simplicity of the technology made the author wonder if this fiber technology could be adapted for some industrial organic chemical reactions which are plagued by stable dispersions.

2. Materials and Methods

2.1. Materials. Epichlorohydrin (EPI), glycidol, glycerin monochlorohydrin, glycerin, tetrahydrofuran (THF), diethylene glycol monobutyl ether (DB), tetrabutyl ammonium hydroxide, tetraethyl ammonium bromide, benzyl alcohol, and benzyl bromide were obtained from Sigma-Aldrich. Epoxidized soybean oil (Vikoflex^{TM} 7112, Atofina - iodine value: 1.35, acid value: 0.08 mg KOH, and oxirane oxygen: 7.0%) was used as supplied. Bisphenol A (157) was obtained from Shell Chemical. A 23% sodium hydroxide solution was made with 97% sodium hydroxide pellets obtained from EM Science and deionized water. Graphite fibers were #292 from FiberGlast and had a nominal diameter of 7 μm. These fibers were reacted with nitric acid to improve their hydrophilicity. Fiberglass was Pyrex with a diameter of 8 μm from VWR International. Stainless steel fibers (type 316), nominal diameter 8 μm, were obtained from Bekaert Fibre Technologies. Stainless steel tubing, fittings, valves, and pressure gauges were obtained from Swagelok. Pumps were obtained from Fluid Metering, Inc.

2.2. Fiber Reactor Construction. Fiber reactors (FR) were constructed of stainless steel tubing and tube fittings from Swagelok as shown in Table 2 and Figure 4. A fiber bundle half as thick as needed and twice as long was folded in the middle. The folded fiber bundle was hooked at the midpoint by a rod or tube longer than the reactor and pulled carefully through the tube using soapy water as a lubricant. The rod or tube was secured at the top of the reactor with Swagelok drilled to pass fittings. Small 0.0635 cm o.d. reactors were estimated to have about 100,000 fibers and a liquid volume available of about 2 mL/30 cm.[12] FR with larger diameters were constructed with fiberglass or 316-type stainless steel fibers. Both fibers were a nominal 8 μm in diameter. The liquid volume of reactor #3 was determined by injecting organic phase containing a dissolved crystal violet indicator. It took 7.83 min for the color to exit the reactor at an organic phase flow rate of 4 mL/min. The caustic rate was 0.5 mL/min, for a total flow rate of 4.5 mL/min. Thus, the reactor liquid volume is 7.83 min × 4.5 mL/min = 35.25 mL. Table 2 describes the fiber reactors used.

2.3. Fiber Reactor Operation. None of the reactions in this feasibility study were optimized for contact time, catalyst, catalyst concentration, or temperature. All runs were continuous and ran for 0.5 to 6 h.

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Table 2. **Fiber reactor parameters**

reactor #	tube i.d., mm	tube length, cm	fiber μm	fiber diameter, fibers	no. fibers	reactor volume, mL
1	10.9	30.5	glass	8	570,000	18
2	10.9	30.5	SS	8	540,000	18
3	10.9	91.4	SS	8	540,000	35
4	25.4	61	SS	8	2,160,000	144

The polar phase flow into the reactor and out of the separator was controlled with FMI QG20-Q1-SSY-Q485 stainless steel and ceramic pumps. Organic flow into the reactor was controlled with a FMI QG20-Q2-SSY-Q485 stainless steel and ceramic pump. For heated reactions the FR was wrapped with a silicone-coated tape heater and insulated with fiberglass insulation. Temperature was controlled with a J-Kem model 210-T controller. Temperature was measured with a type T thermocouple. Ambient temperature reactions were not temperature controlled and were typically 22.5 °C. Pressure was measured with an Ashcroft electronic 0−1000 psig pressure gauge.

The fibers were wetted first with polar phase, and then the organic reactant solution was pumped through the fiber bundle. Reactor parameters set at this time include:

- caustic/organic ratios
- flow rates
- temperature
- catalyst
- catalyst levels.

After the reactor was stabilized, samples of the organic products were collected, neutralized, chilled, or diluted in THF, and analyzed by chromatography.

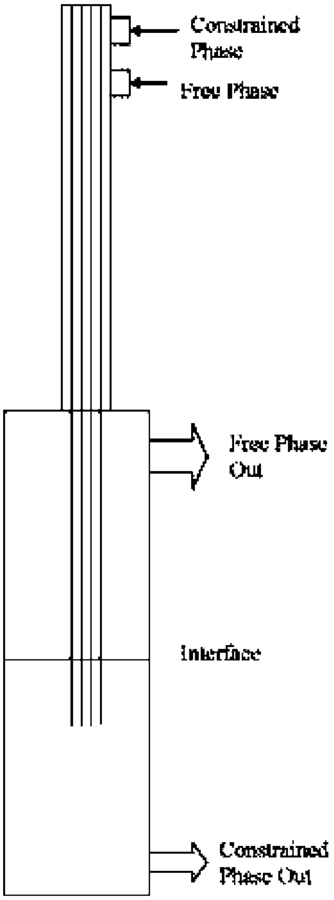
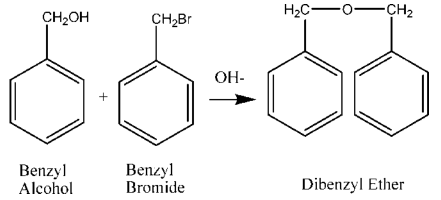


Figure 4. **Fiber reactor diagram.**

Scheme 1. **Dibenzyl ether synthesis**



2.4. Analytical. Analysis of the reactants, coupling products, epoxidation products, and hydrolysis products were determined either by gel permeation chromatography (GPC) or by gas−liquid chromatography (GC). GC/MS was performed on an Agilent Technologies model 6890N gas chromatograph coupled to a model 5973N mass selective detector. The GC injector was in the split mode with a split ratio of 1:10, and the analytes were separated on a 30 m × 0.25 mm (i.d.) capillary column (HP-5MS, Agilent Technologies) with a stationary phase of poly(phenylmethylsiloxane). GPC was performed using a Waters 600 controller, a Waters 2410 refractive index detector, and Millennium 32 software with column series of a Waters Styragel HR 4E, a Polymer Laboratories PLgel 3 μm 100 Å, and a PLgel 5 μm 50 Å. Each of the columns was 300 mm × 7.5 mm. Flow rate of tetrahydrofuran was 1 mL/min. GC was performed using a Varian 5000 using a 6 in. × $^1/_8$ in. OV-101 substrate on 100 mesh Chromosorb packing. All chromatographic analyses except GC/MS are based on area %.

2.5. Theoretical Basis and Theoretical Calculations. The exceptional mass-transfer performance of the FR is due to the large increase in surface area compared to that obtained by conventional dispersion techniques. Calculations of the surface area generated by 1 mL/min of aqueous phase pumped into the 1 cm i.d. × 30 cm long laboratory reactor with 540,000 8-μm steel fibers indicate that this unit had 1 mL of aqueous phase spread over about 40,000 cm^2 of fiber surface with a phase thickness of about 250 nm. For comparison, a stirred reactor with 100 μm particle sizes[15] would only generate about 600 cm^2 of surface area from 1 mL of dispersed liquid. This difference in surface area accounts for the approximately 60-fold decrease in reaction time observed for biodiesel reactions.

Hydrophilicity and surface tension are scientific principles that explain the strong attachment of constrained phase to fiber. The attachment has worked for water, alcohols, and mixtures of water or glycerin with alcohol.

3. Results

An etherification reaction, a dehydrochlorination reaction, a transesterification reaction, and a neutralization of vegetable oil were evaluated in fiber reactors. Preliminary unoptimized results are reported.

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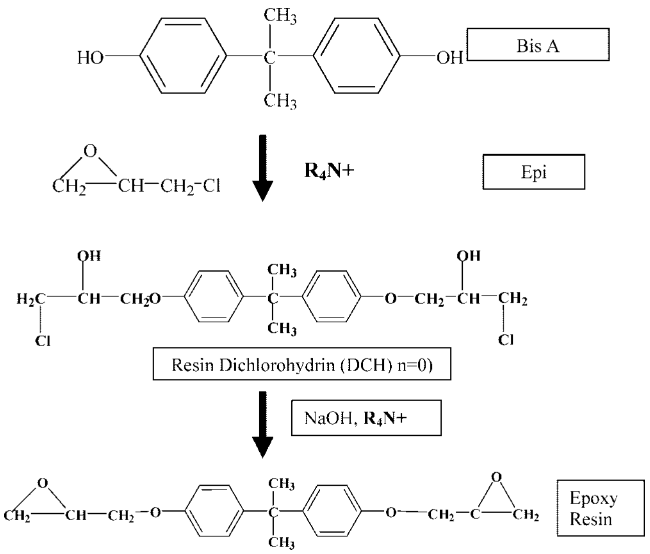
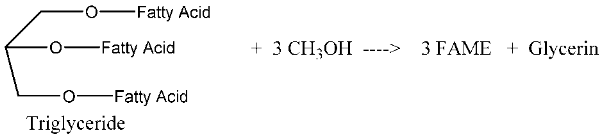
***Table 3.* Etherification reactions in the fiber reactor**

rxn	process conditions	% conversion to BzEther	mole ratio of BzBr: NaOH	excess NaOH (%)	flow rate (org:aq)	organic contact time, min	run duration, mins
1	75 °C, no PTC, 70% toluene	4.6	1:1	0	4.5:1.0	4	30
2	75 °C, no PTC, no solvent	13.3	1:1.1	10	2.0:1.5	8	60
3	75 °C, 2% TEAB, no solv.	64.8	1:1.1	10	2.0:1.5	8	60
4	75 °C, 2% TBAB, 70% toluene	72.2	1:1	0	4.5:1.0	4	30

***Scheme 2.* Coupling of bisphenol A and epichlorohydrin**



***Scheme 3.* Transesterification of triglyceride with methanol**



3.1. Etherification. Preparation of dibenzyl ether from benzyl alcohol/benzyl bromide using PTC catalyst (Scheme 1).

Dibenzyl ether was prepared continuously using FR #2 at a reaction temperature of 75 °C. Table 3 summarizes the results of four experiments and indicates that solvent, excess caustic, and PTC affect the chemical conversion achieved in this reaction. To avoid salt precipitation, a solution of 23% NaOH solution was used. When 23% NaOH is totally converted to NaBr (or NaCl), it will form a near saturated solution. It is important to ensure that a super-saturated solution of salt in water does not form so that the reactor is not plugged by salt precipitation in the reactor. Reactor pressure was constant in these reactions. To start, the aqueous caustic was pumped through the reactor at 1.5 mL/min to coat the fibers. Then an organic solution consisting of a 1:1 molar mix of neat benzyl bromide and benzyl alcohol containing 2% tetraethyl ammonium bromide (TEAB, based on reactants) was pumped at 2.0 mL/min (Reaction 3). Both phases coming out of the reactor were collected in a separatory funnel, with the fibers extending to the bottom of the funnel. Phase separation was practically instantaneous. Product was separated and analyzed using GC/MS. Result: 65% conversion of benzyl bromide to the ether in about 8 min contact time. For comparison, in experiment 2 without PTC, conversion to dibenzyl ether was only 13%. Another experiment using tetrabutyl ammonium bromide (TBAB) in the aqueous phase was evaluated in FR #2. The fibers were wetted with a 23% NaOH solution containing 2% TBAB, and the flow was adjusted to one mL/min. Then a 1:1 molar mix of benzyl bromide and benzyl alcohol in toluene (30% mix) was pumped through the wetted fibers at 4.5 mL/min. This experiment had 72% conversion (GC/MS) of benzyl bromide to benzyl ether in about 3.5 min contact time. For comparison, reaction 1 without PTC gave low 5% conversion.

Joshi and Sawant studied the conversion of benzyl chloride to dibenzyl ether with caustic and PTC.[16] About 85% conversion of benzyl chloride to dibenzyl ether was obtained in about 2 h at 80 °C and 2500 rpm at high sodium hydroxide concentration ($>$8.75 M). At low concentration of sodium hydroxide, the yield of dibenzyl ether was low.

3.2. Dehydrochlorination/Oxirane Synthesis Using PTC Catalyst. Bisphenol A (Bis A) couples with epichlorohydrin (Epi) in the presence of a quaternary ammonium catalyst to give resin dichlorohydrin (DCH, Scheme 2). Epi is used in 10-fold molar excess of stoichiometry in order to reduce oligomers.[17] DCH is converted to epoxy resin by dehydrochlorinating with caustic.

3.2.1. Step 1. Homogeneous Coupling of Bisphenol A and Epichlorohydrin. Epi (1000 g, 10.8 mol), Bis A (143 g, 0.63 mol, 1.2 equiv hydroxyl) and TEAB (2.5 g, 2182 ppm) were stirred in a 1400 mL flask with a large magnetic stirring bar. The mixture was heated to 70 °C for 12 h while stirring. Bis A conversion to coupled species was determined using GPC. Reaction was stopped at 99% conversion of Bis A to resin species.

3.2.2. Step 2. Dehydrochlorination in the Fiber Reactor with PTC Catalyst. The glass fibers in FR #1 were wetted first with 23% caustic phase, and then the coupling reaction mix from above was pumped at room temperature through the fiber bundle. Reactor parameters set at this time included total flow rates, caustic/organic ratios, temperatures, and catalyst level. One set of conditions was caustic flow, 0.2 mL/min. (0.006 equiv/min); coupling mix flow 4 mL/min. (0.0068 equiv Bis A/min); room temperature, 22 °C. The TEAB concentration in the coupling mix was 0.2%.

After the reactor stabilized, samples of the effluent were collected, chilled on ice until diluted in THF, and analyzed by gel permeation chromatography for DGEBA ($n = 0$ and $n = 1$), Bis A monochlorohydrin ($n = 0$ and $n = 1$), and DCH ($n = 0$ and $n = 1$). Chlorohydrin conversion to oxirane was 98.5% in about 4 min contact time.

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(16) Joshi, S. R.; Sawant, S. B.; Joshi, J. B. *Org. Process Res. Dev.* **1999**, *3*, 17–27.

(17) Perry, W. O. Process for reacting a phenol with a vicinal epoxy compound in the presence of phosphorous or carbon containing acid, ester, or acid ester. (The Dow Chemical Company). U.S. Patent 3,948,855, 1976.

Table 4. **Transesterification of refined soybean oil**

reactor	fiber phase, mL/min	oil phase, mL/min	pressure, psig	reactor volume. mL	contact time, min	run duration, h	conversion to FAME
1	1	2.5	150	18	7.2	4	99.6
1	3	7.5	205	18	2.4	3.5	100
2	2	5	29	18	3.6	3	98.8
2	4	10	68	18	1.8	4	99.3
4	10	25	27	144	5.8	7	99.0

3.3. Transesterification: Triglyceride to Biodiesel. Biodiesel is produced by catalytic transesterification of triglycerides in oils and fats with alcohols such as methanol to produce fatty acid methyl esters (FAME, biodiesel) and glycerin (Scheme 3).

In the process of base-catalyzed transesterification, two liquid phases must be separated. The heavier phase mainly consists of methanol, glycerol, and some catalyst, intermediate products, and may contain soap (from free fatty acids (FFA)) in the oil. The lighter phase mainly contains fatty acid methyl esters containing residual methanol, some monoglyceride, some soap, and some glycerin. Washing with water removes these residual impurities from crude biodiesel. Acid-catalyzed transesterification does not have the same magnitude of separation problems because soap is not formed, but the acid-catalyzed reaction rate is much slower than the base-catalyzed reaction rate.

Commercial multistage transesterification processes are slow (one to many hours) because the reaction mixture consists of two phases, the reaction is mass-transfer limited, conversion per stage is limited in batch and continuously stirred tank reactors (CSTR), and settling is poor because of byproduct soap. The transesterification reaction is actually quite fast if the components can be brought into contact with each other and the catalyst. Inert cosolvents have been used to generate an oil-rich homogeneous system which allowed 95% conversion in 10 min at ambient temperatures.[18] Process intensification methodology involving novel oscillatory reactors and microreactors is being developed to attain more rapid reaction rates using milder, less energy-intensive reaction conditions, and for enhanced selectivity to produce biodiesel with minimal byproducts.[19] The oscillatory flow reactor reported by Harvey, et al. gives approximately 95% conversion in 15 min at 60 °C. An ultrasonic biodiesel process has given 99% conversion in 15 min.[20] A two-stage continuous stirred tank reactor process was designed to produce 99% conversion in 2 h at 60 °C, with separation of byproduct glycerin between stages.[21]

Transesterification reactions were evaluated in FR #2. The fiber phase was methanol containing 2% sodium methoxide based on the oil, the free phase was refined soybean oil, and the reaction temperature was 60 °C. The results were surprisingly good (Table 4).

The phases coming out of the reactors were well separated. Crude biodiesel was washed simultaneously with D.I. water in another FR. ASTM specifications for biodiesel were met.

3.4. Neutralization of Vegetable Oil. Washing acids from vegetable oils is similar to the original use of the fiber−film contactor. Vegetable oils need to be neutralized by removal of 0.5−1.5% free fatty acids (FFA). Extraction of FFA from triglycerides is complicated in current dispersion processes by the formation of oil/water/soap dispersions that separate slowly. A mixture of 30% vegetable oil/70% hexane containing 5% FFA (16% FFA in the oil) was pumped into FR #2 at 9 mL/min. (This 30:70 mixture is called miscella in processes that extract oil seeds with hexane). The fiber phase was an aqueous stream (1 mL/min) containing 10% NaOH and 90% ethanol. FFA salts were cleanly extracted (99%) into the aqueous/ethanol phase with about 1.8 min contact time and instantaneous phase separation at room temperature. Total run duration for the reaction was about 2 h. Ethanol was used to solubilize the FFA sodium salt (soap) in the water phase. Solubility of soap in water is limited.

4. Discussion

The FRs were easy to build and operate. They gave rapid reactions, high conversions in one pass, and separation of phases exiting the reactor.

The ether synthesis reactions in Table 3 indicate that diluent solvent and catalyst affect the conversion that can be obtained at close to stoichiometric caustic utilization. Optimization of reaction parameters would be expected to achieve better conversions.

The base-catalyzed soybean oil transesterification with methanol to make biodiesel proved that methanol can be used as the constrained phase. We also found that transesterification is a fast reaction and is compatible with the FR. Extremely high conversion rates were achieved in one stage instead of the multiple stages anticipated from our review of commercial processes. Soap is a byproduct of the water in the raw materials as well as the caustic catalyst. Phase separation coming out of the FR was virtually instantaneous despite the presence in early reactions of high levels of soap. The FR results exceeded biodiesel results of the oscillatory flow reactor and all reported processes we found for simplicity, manufacturability, operability, short reaction time, separation time, and reduced energy consumption. ASTM specification biodiesel was produced after washing in a second fiber unit.

The neutralization of vegetable oils proved that the FR can handle high (5%) soap concentrations without the formation of "rag" layers of emulsified materials.

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5. Conclusions

The postulate that the Fiber−Film concept would work for two-phase organic reactions, including phase transfer catalyzed reactions, was confirmed. The FR gave high conversions of starting material to product within a few minutes reaction time under moderate temperature and pressure. The phases separated instantaneously because dispersions did not take place.

The large mass transfer area achieved by the FR is complemented by the very short phase contact time and instantaneous phase separation. This combination of properties allows for continuous fast reactions, high conversions, and minimal hydrolysis of byproduct.

The new nondispersive paradigm for two-phase chemical reactions has been demonstrated for the reactions reported in this paper.

Acknowledgment

We thank the National Science Foundation and the Department of Agriculture for their support of fiber technology projects: *NSF Phase I SBIR*, DMI-232067, *USDA Phase I SBIR* 2005-33610-15504, *USDA Phase II SBIR* 2006-33610-17174.

Supporting Information Available

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Received for review January 16, 2008.

OP800010M