“Direct fluorination of toluene using elemental fluorine in gas/liquid microreactors”

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(paper review)

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Overview

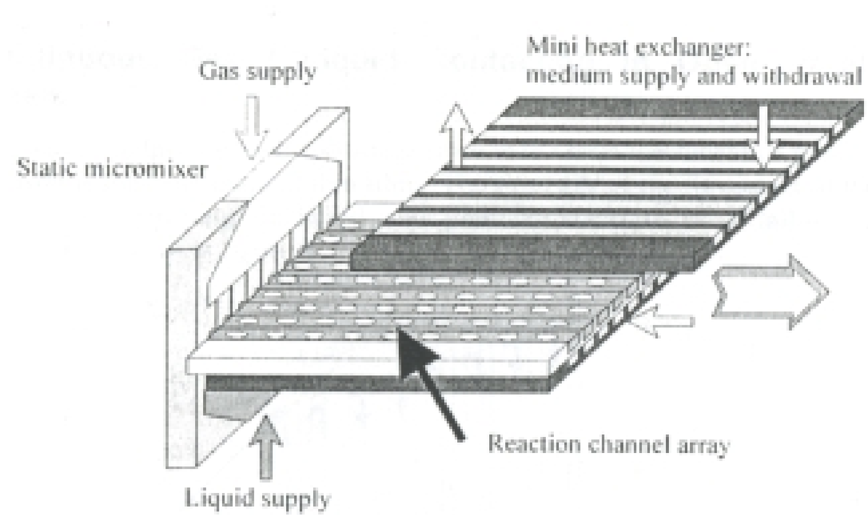
- Motivation for microreactor studies
- Micro fabrication of reactors
  - Micro bubble column
  - Falling film microreactor
- Comparison between micro/macro reactors
- Advantages of using micro technology
- Improvements to design for future work
- Summary

Technological Benefits For Microreactor Use

- Process safety
- Simple processing
- Low waste generation
- Reaction performance improvements
  - Control over reaction temperature
  - Control over reactant mixing

Micro Bubble Column (MBC)

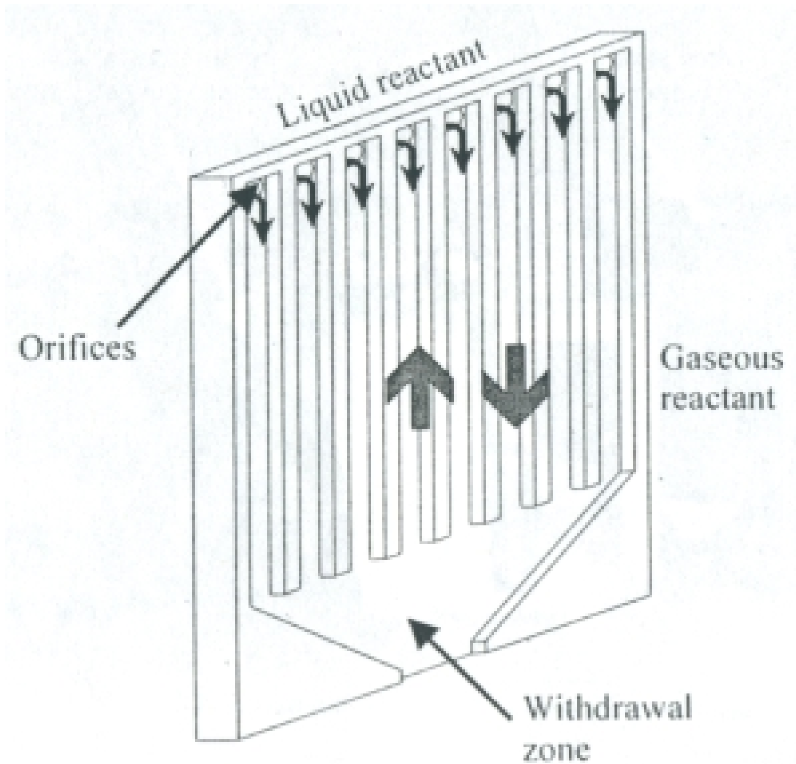
- Stainless steel
- Mixer (UV- LIGA)
- Platelet with narrow channels (UV-LIGA)
  (50×50×50000 μm)
- Platelet with wide channels (wet chemical etch)
  (300×150×50000 μm)



Schematic of micromixer with gas and liquid channel configurations

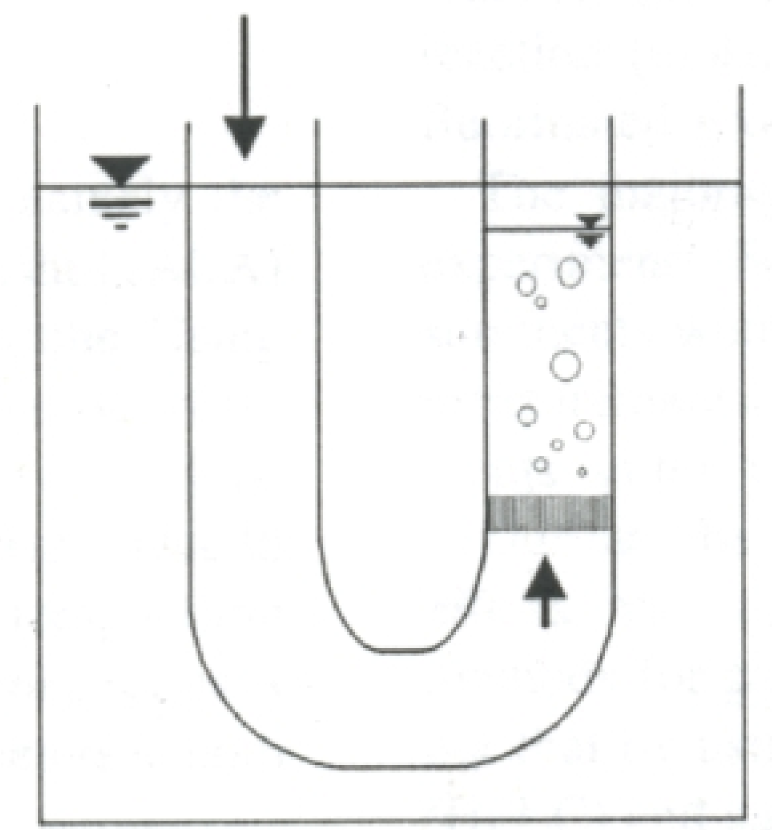
Falling Film Microreactor (FFMR)

- Stainless steel
- Reaction plate (micro electro discharge machining)
- Channels on reaction plate (die sinking) (300×100 μm)
- Thin metal foils (wet etching)



Laboratory Bubble Column (LBC)

- U-tube
- Gas dispersion into liquid through porous frit
- Thermostatic cooling bath



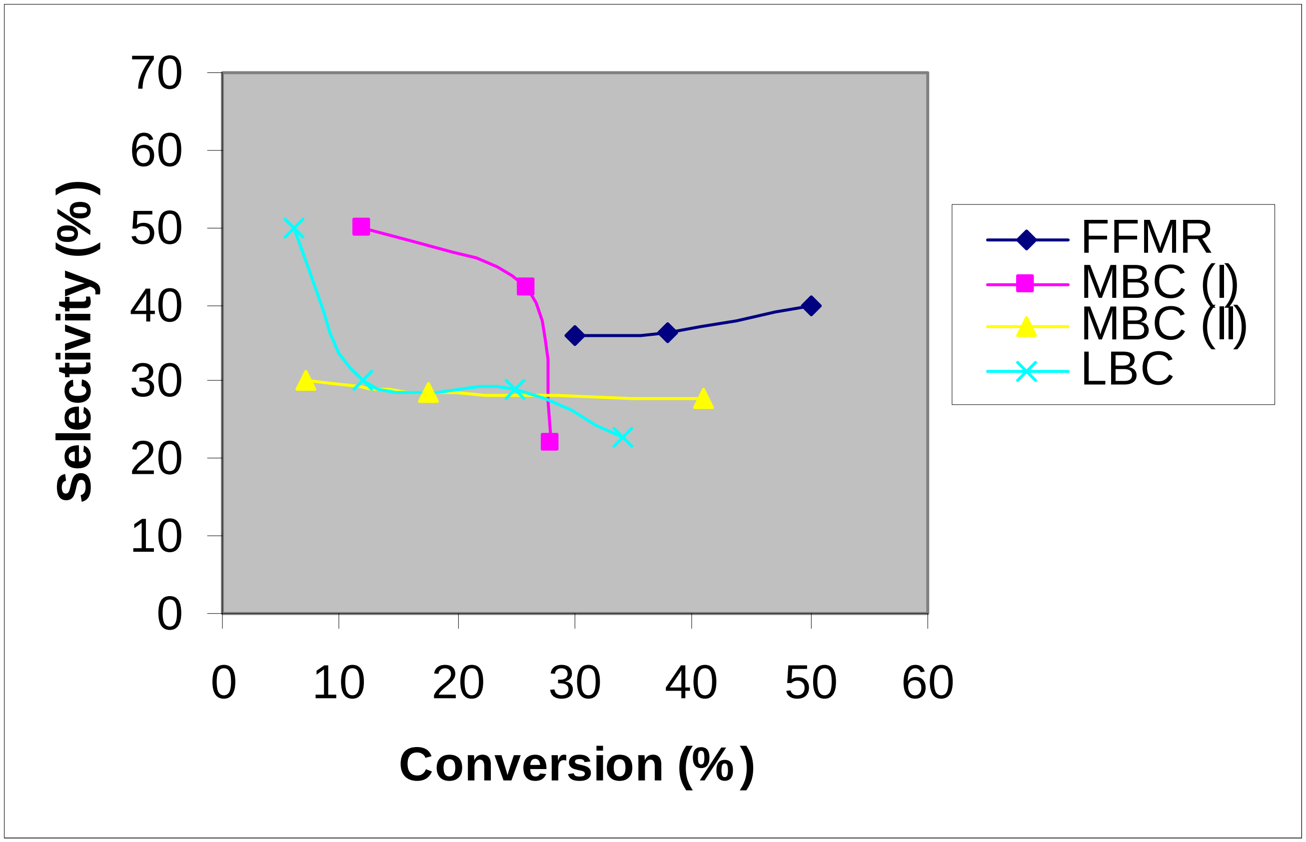
Fluorination to Mono-Substituted Compounds

- Experimental conditions
- Performance of reactors
  - Selectivity versus conversion
  - Space-time yield
- Variation of fluorine to toluene ratio
- Comparison between FFMR, MBR, and LBC

Experimental Conditions

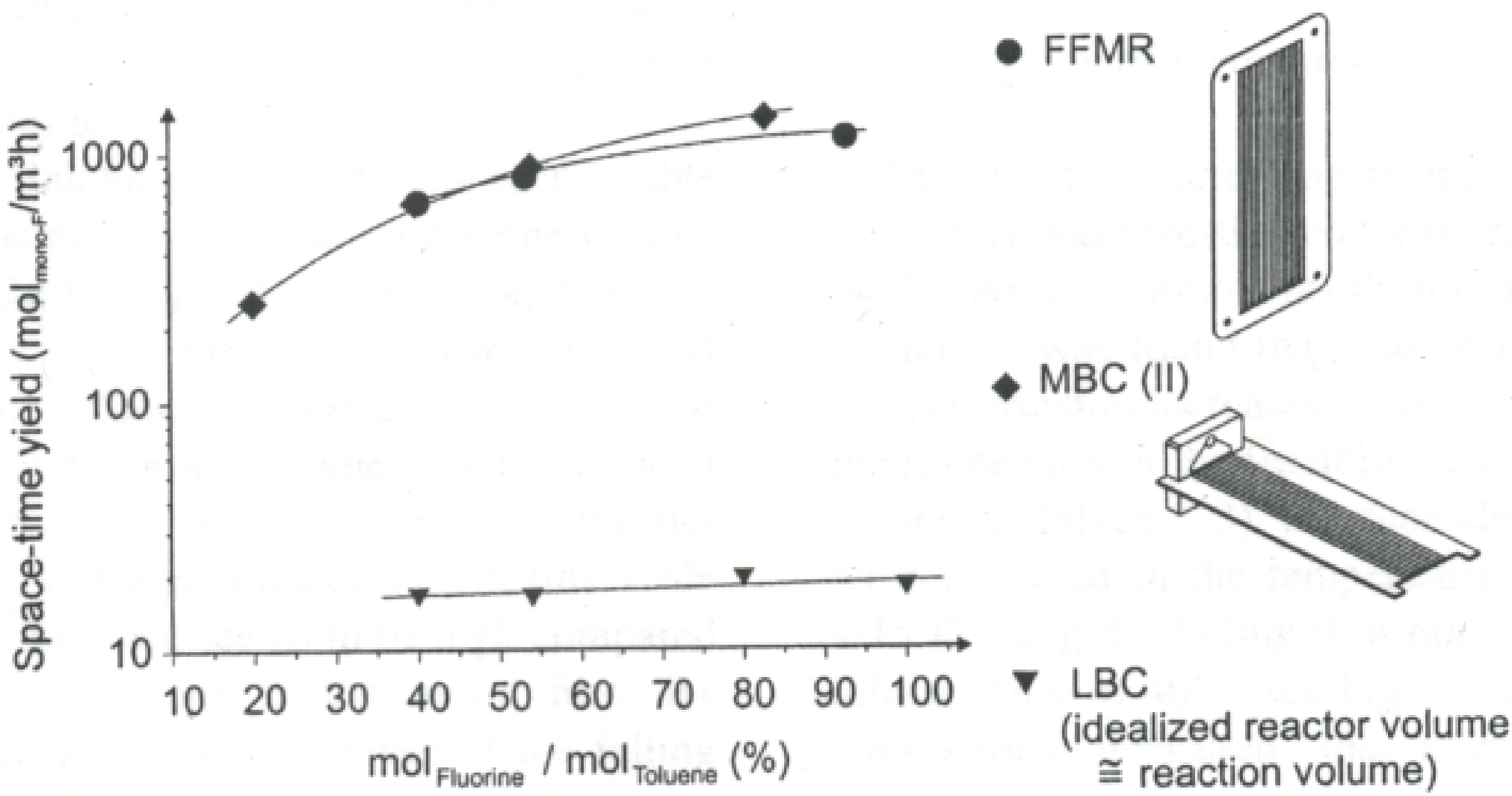
- Gas: 10% F_2 in N_2 carrier
- Liquid: toluene 1.1 M in acetonitrile or methanol
- Explore parameter space
  - change temperature
  - change F_2/C_7H_8 inlet molar ratio
  - change liquid residence time

Selectivity versus Conversion

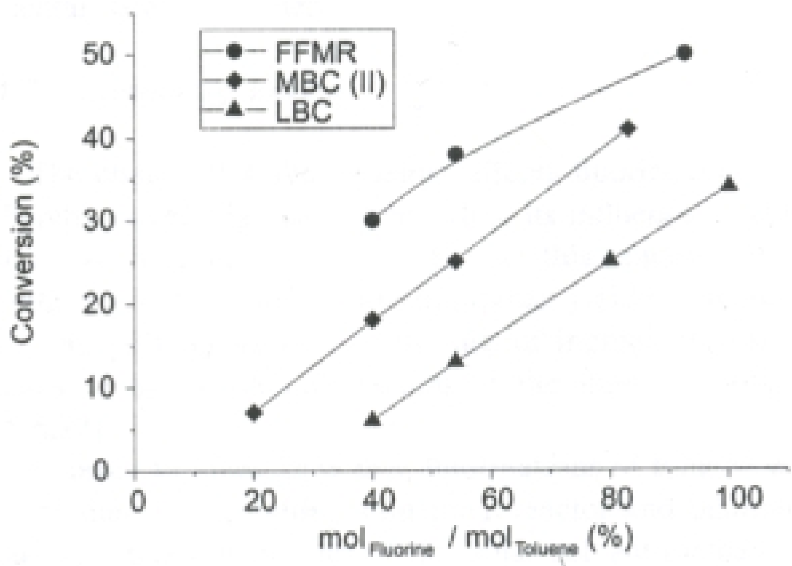


* This graph was made in Excel using values reported by the literature

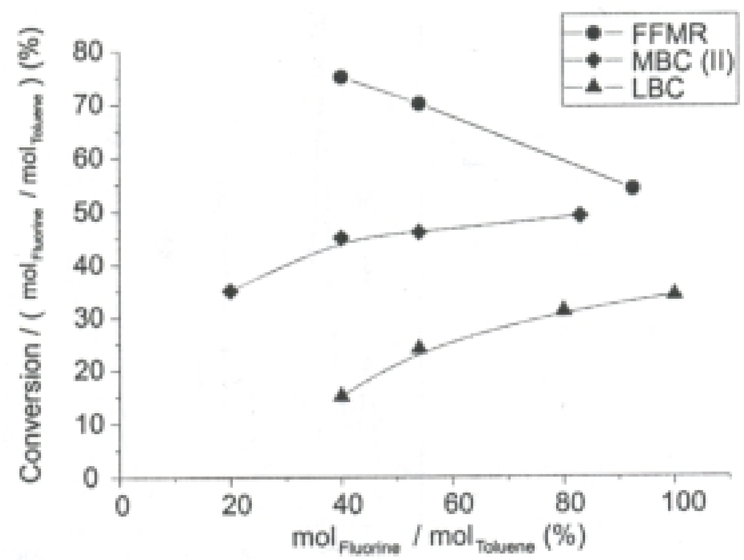
Space-Time Yield



Variation of Fluorine to Toluene Ratio



Conversion of fluorination of toluene



Reactor efficiency

Results

- MBC
  - 41% conversion with a yield of 11%
- FFMR
  - 50% conversion with a yield of 20%
  - * 76% conversion with a yield of 28%
- LBC
  - 34% conversion with a yield of 8%

* This experiment ran under different operating conditions with a 200% fluorine to toluene ratio and 10% toluene to CH3C ratio.

Advantages of Micro Technology

- Enhanced heat and mass transfer
- High specific interfacial areas
- Increased conversion and selectivity
- Higher yield
- Safety issues are less problematic
- Fast reaction times

Potential Design Modifications

- Role of specific surface area
- Flow equipartition among channels
- Numbering-up concepts (parallel reactors)
- Improved ratio of reaction to construction volume

Summary

- Microreactor performances superior to laboratory benchmark
- FFMR outperformed MBC
- Process safety non-issue for microreactors
- Microreactor fluorination in a matter of seconds

References

- V. Hessel et al., Microreaction Technology: Industrial Prospects, IMRET3, Springer, Berlin, 2000, p. 526