This presentation does not contain any proprietary, confidential, or otherwise restricted information.

Reactive Dehydration Technology for Production of Fuels and Chemicals from Biomass

KSE, Inc. with the University of Massachusetts, Amherst October, 2010 Through December, 2014

> Dr. James R. Kittrell, KSE, Inc. Dr. Carl R. Dupre, KSE, Inc. Dr. Michael F. Malone (Subcontractor)

DE-EE0004543

U.S. DOE Advanced Manufacturing Office Peer Review Meeting Washington, D.C. May 6-7, 2014



Project Objective

- Commercialize a novel reactive distillation technology using the iCARD platform (Intensified Catalytic and Reactive Distillation) for compact, inexpensive production of biomass-based chemicals from complex aqueous mixtures.
- Separation/Purification of Biomass Chemicals is Difficult and Costly:
 - Costly separation of complex mixtures of many biomass chemicals typically found in a dilute aqueous stream.
 - Excess water is costly to remove, inhibits biomass conversion, and often drives byproduct reactions.



Conventional Technologies

➢Distillation

- Highly energy intensive
- Difficult for non-ideal mixtures
- Limitations for heat sensitive materials
- Many costly columns for complex mixtures

Membrane Separation

- High investment, maintenance costs
- Severe limitations on contact material
- No economies of scale







Possible iCARD Commercial Applications

> Water removal from fuel grade ethanol

- 50% reduction in energy requirement
- Particularly superior for dilute cellulosic ethanol production
- Extensive experimentation for thermodynamic data
- Aspen-based process design using thermodynamic data
- Existing plant retrofit: less than 2 year payout

Desalination

- Dehydration of directional solvent extractant
- Water separation during reactions
 - Water retards reaction, drives byproduct reactions
 - Production of FDCA (furandicarboxylic acid) from hexoses
 - Production of THF from pentoses
 - Simple, intensified process



Technical Approach

- Convert water to easily removable chemical using reversible reaction.
- Convert biomass intermediate to easily removable chemical
- ≻ Example: THF from Biomass
 - Now: Pulp Mill: Hemicellulose containing xylan in ~5% aqueous solution; burned as waste heat
 - **iCARD**: Convert xylan to xylose to furfural to THF THF (60°C) or furan (31°C); easily distilled out. Heavy column bottoms continue to waste heat boiler

Low cost THF in compact, highly intensified process



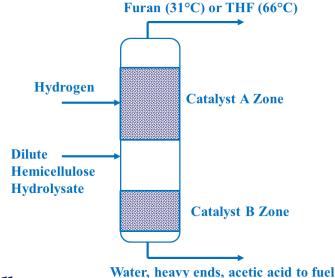
Innovation and Execution

Reactive distillation

- Process intensification
- Reduce reaction and complex separation to a single compact unit
- Low cost THF

Novel structured catalysts

- Combine catalytic functionalities for dehydration, decarbonylation, hydrogenation
- Shape, form suitable for reactive distillation
- Industry / University Team
 - KSE and University of Massachusetts





Transition and Deployment WHO CARES? **U.S. Producers of THF and Spandex (PTMEG) Conventional Manufacturing of THF** OH Natural H₃C \rightarrow C₄H₁₀ \rightarrow Gas Liquids Maleic Butane Butanediol THF Anhydride **Complex, Energy Intensive, Costly Process Manufacturing Cost: \$1.45/Pound KSE iCARD Manufacturing of THF** Hemicellulose **Hydrolysate** PTMEG THF **Spandex Process Intensification, Simplification Manufacturing Cost: \$0.70/Pound**



Transition and Deployment

The Customer Cares

U.S. Pulp & Paper Industry

- \$200 Billion annual sales
- 6% of U.S. manufacturing GDP
- Employment over 1 million
- Over 170 mills closed last 20 years
- U.S. Kraft Pulp Mills
 - Produce Xylan-rich hydrolysate
 - Currently disposed for waste heat
 - **iCARD** will utilize hydrolysate for THF
 - Pulp mill profitability enhancement







Impact: Transformational Technology

Energy Savings

Cumulative Energy Demand (CED), MJ/kg Conventional THF Manufacturing	270
Cumulative Energy Demand (CED), MJ/kg KSE iCARD THF Manufacturing	75
Energy Savings for iCARD THF Manufacturing, MJ/kg	195
Annual U.S. THF Production, Million Pounds	320
Potential Annual U.S. Energy Savings, Trillion BTU's	62

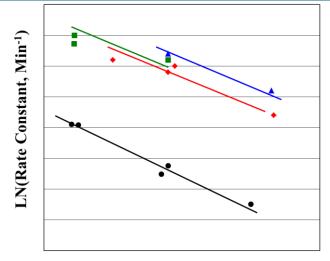
>Manufacturing cost savings

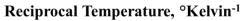
Conventional THF Manufacturing Cost, \$/Pound THF	\$1.45
KSE iCARD Manufacturing Cost, \$/Pound THF	\$0.70
KSE iCARD Savings, \$/Pound THF	\$0.75
Total Manufacturing Cost Savings for Single 32 Million PPY Plant	\$24 Million/Yr

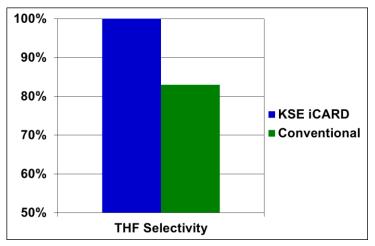


Results and Accomplishments

- Completed Phase I laboratory technical and economic demonstration from furfural
 - Novel catalyst compositions: activity order of magnitude greater than conventional catalysts
 - THF selectivity 100%
- Completed Aspen-based process design









Project Management & Budget

- Key Milestone Schedule
 - Complete patent filings
 - Selection of commercial partner
 - Prototype for xylan hydrolysate conversion to THF
 - Start up of toll processing unit
 - Completion of commercial process design
 - Startup of commercial unit

Total Project BudgetDOE Investment\$2,000,000Cost Share\$1,500,000Project Total\$3,500,000



6 months 12 Months 18 Months

24 Months24 Months36 Months

Project Contacts

- Dr. James R. Kittrell, KSE, Inc. Tel: 413-549-5506 Email: kseinc@aol.com
- Dr. Carl R. Dupre, KSE, Inc. Tel: 413-549-5506 Email: kse.carl@comcast.net
- Mr. Keith Kittrell, KSE, Inc. Tel: 615-549-9084 Email: kdkittrell@kse-online.com

