Biofuels International Canada Expo & Conference

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The Importance of Biodiesel Plant Design

- Latest Technological Developments
  - Transesterification
  - Esterification
  - Newer dual reaction plants with both Transesterification and Esterification
- Examining Energy Maximization & Cost-Effectiveness
- Improving Yields
- Safety Considerations

Quick Chemistry Review

Transesterification

1 Triglycerides + 3 Methanol ←→ 3 Biodiesel + 1 Glycerin

Esterification

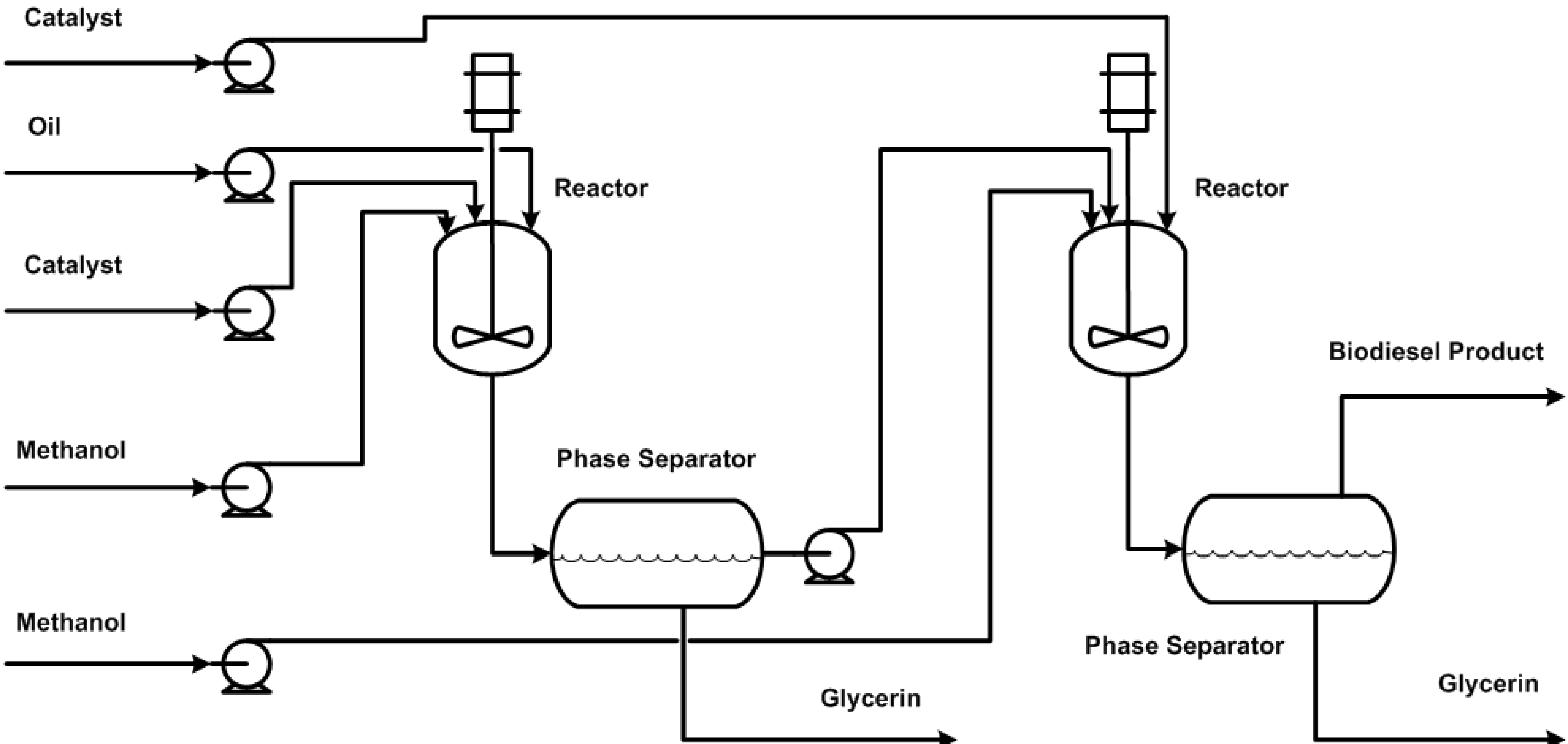
1 FFA + 1 Methanol ←→ 1 Biodiesel + 1 Water

Transesterification

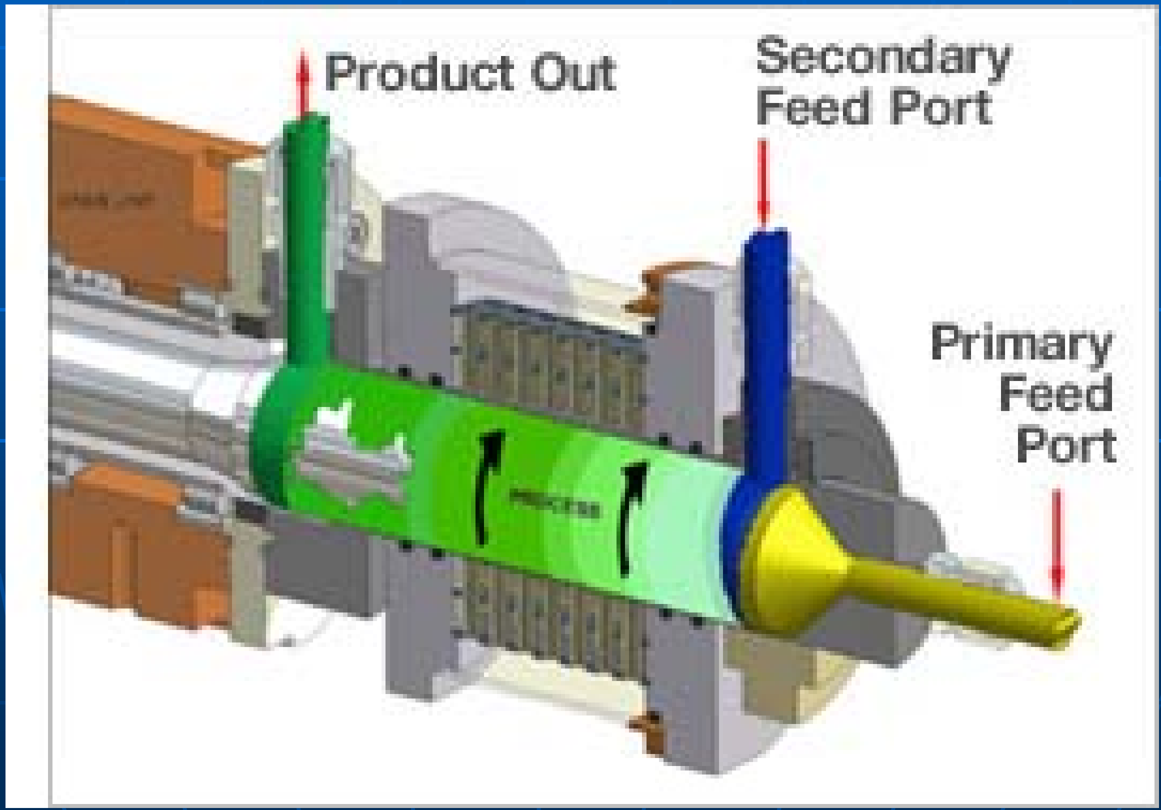
- Traditional Continuous Stirred Tank Reactors
- Spinning Tube in a Tube Reactor
- Shockwave Power® Reactors

Continuous Stirred Tank Reactors

Continuous Stirred Tank Reactors

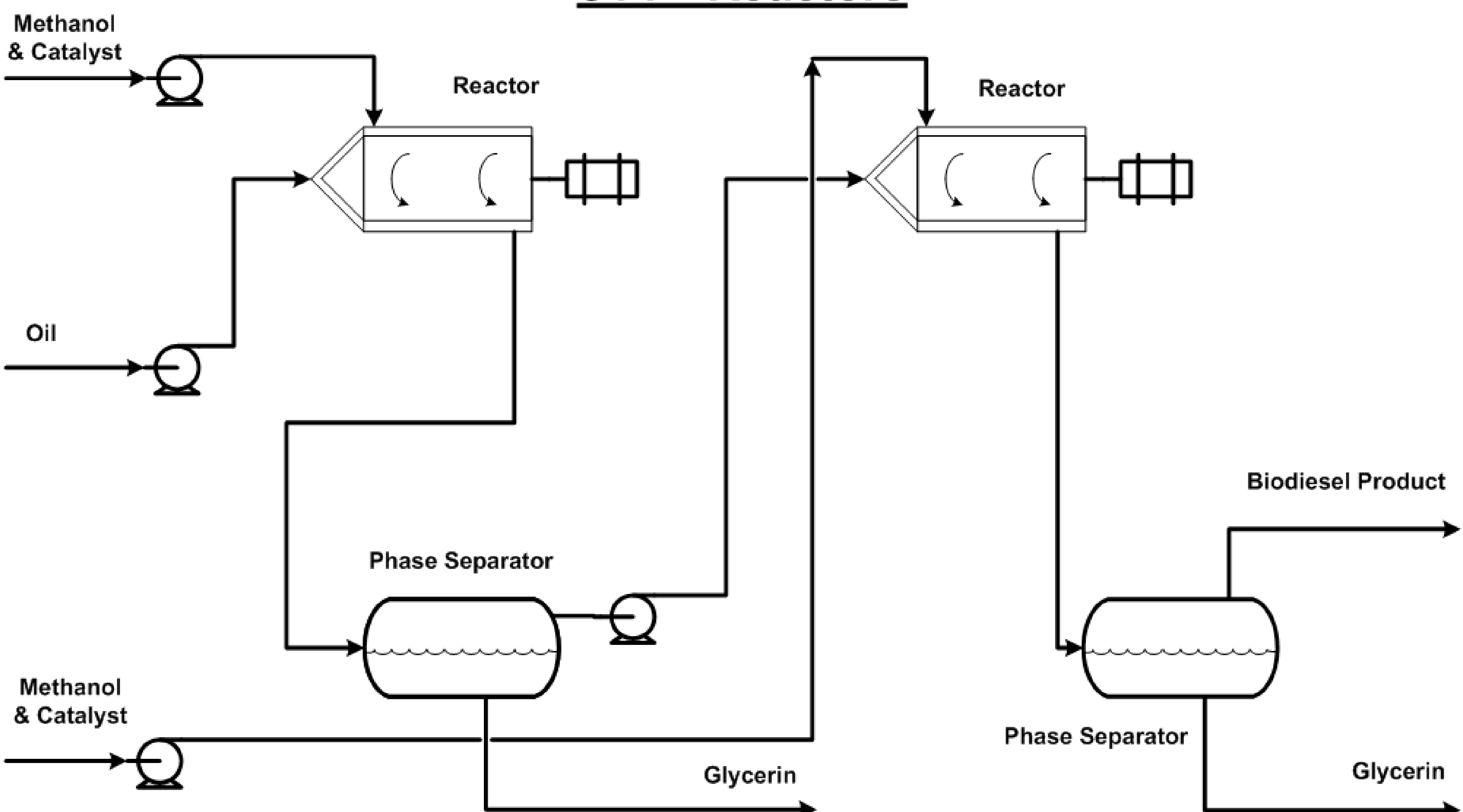


Spinning Tube in a Tube Reactor

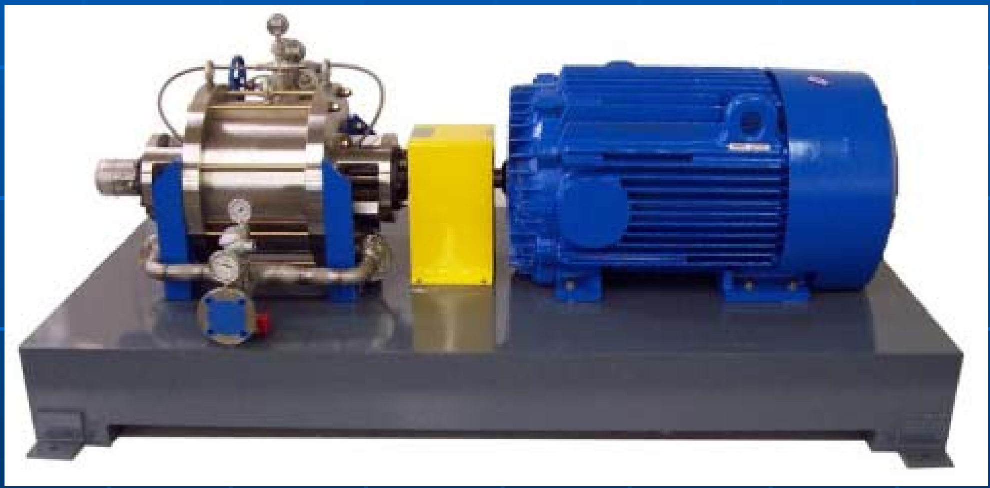


Mixing achieved by shear

STT® Reactors

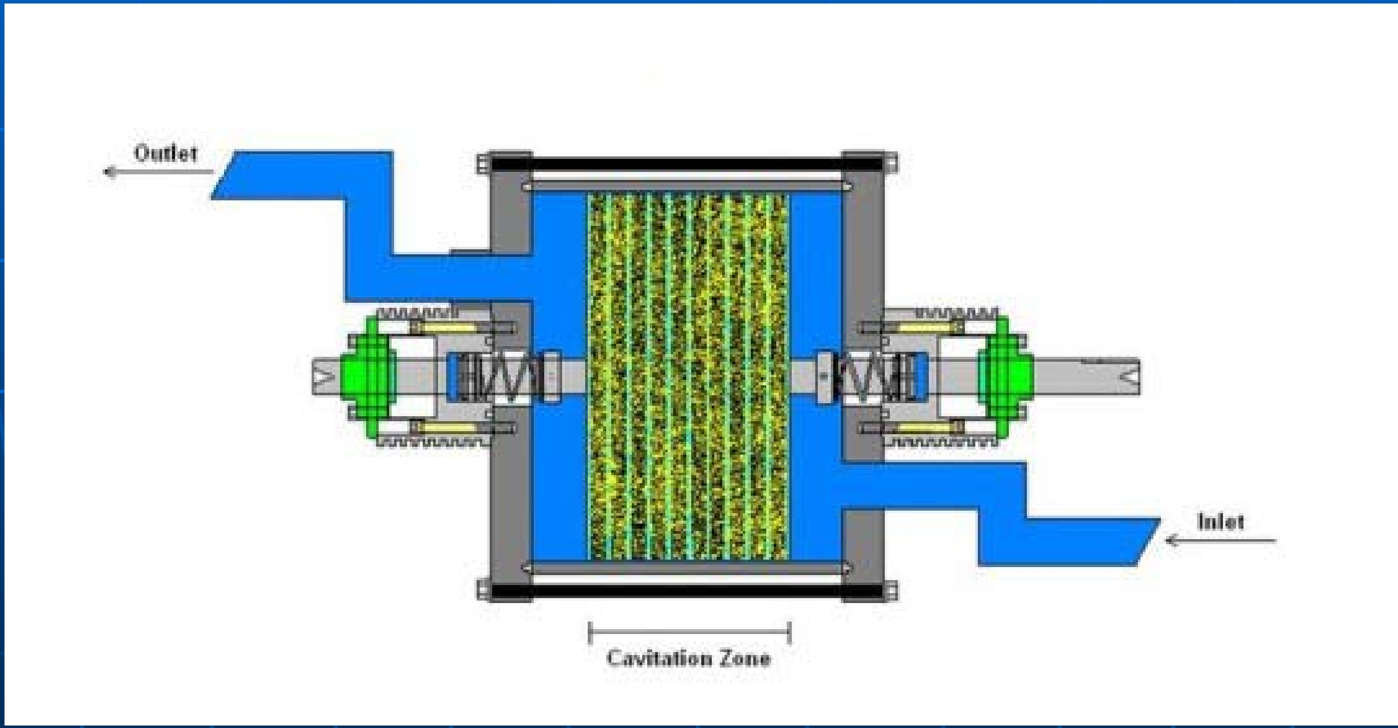


Shockwave Power Reactors

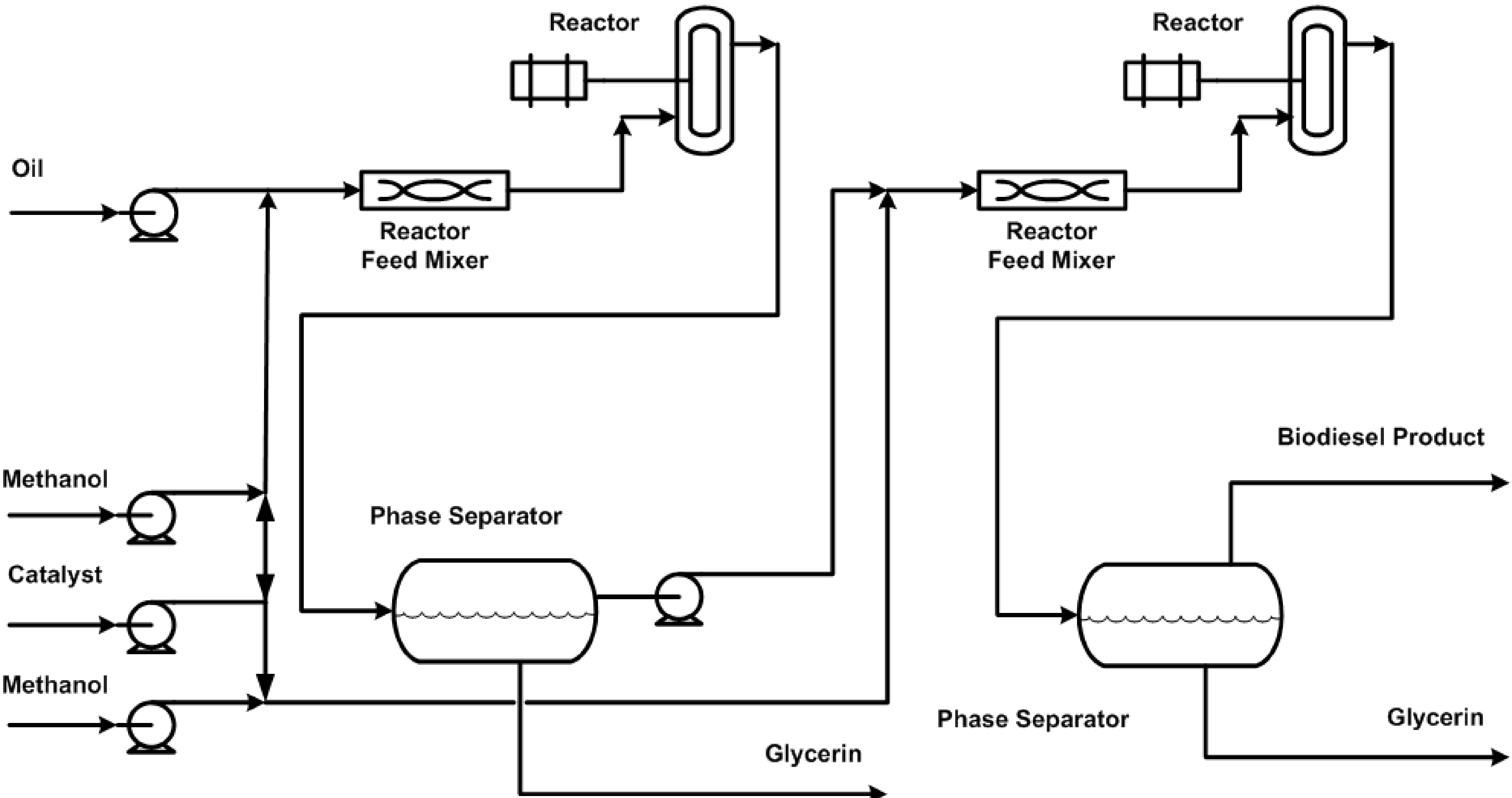


Mixing achieved by controlled cavitation

Shockwave Power Reactors



ShockWave Power® Reactors



Summarizing

• CSTRs - Residence time 20 minutes – mixing by agitation

•ShockWave Power - Reactor Residence time 3.5 seconds - mixing by controlled cavitation

•STT - - Reactor Residence time 0.5 seconds – mixing by shear

Criteria for These Latest Technological Developments

- Low Pressure Process
- Low to Medium Temperature Process
- Performs both Esterification and Transesterification together
- No sodium methylate is used that could form soap
- No sulfuric acid that requires special materials or may form stable emulsions

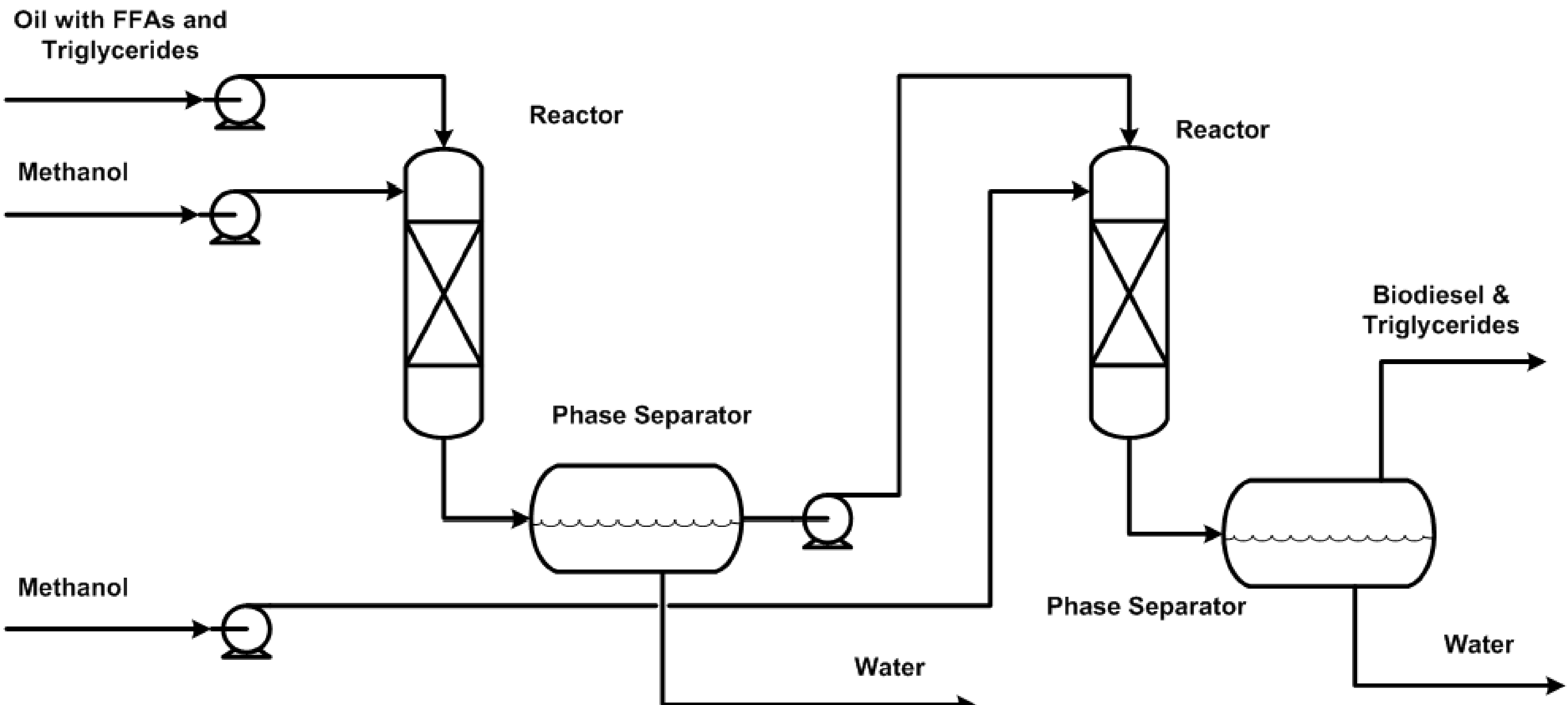
Traditional Esterification

- Typically done with sulfuric acid
- High temperature and pressure
- Corrosion resistant piping and vessels that may include plastic lined pipe and Hastelloy C vessels.

Solid Catalyst Esterification

- Ion exchange beads with the $-SO_3H$ Group
- Two columns or packed beds in series
- Placed in front of a transesterification process

Packed Tower Esterification Reactors



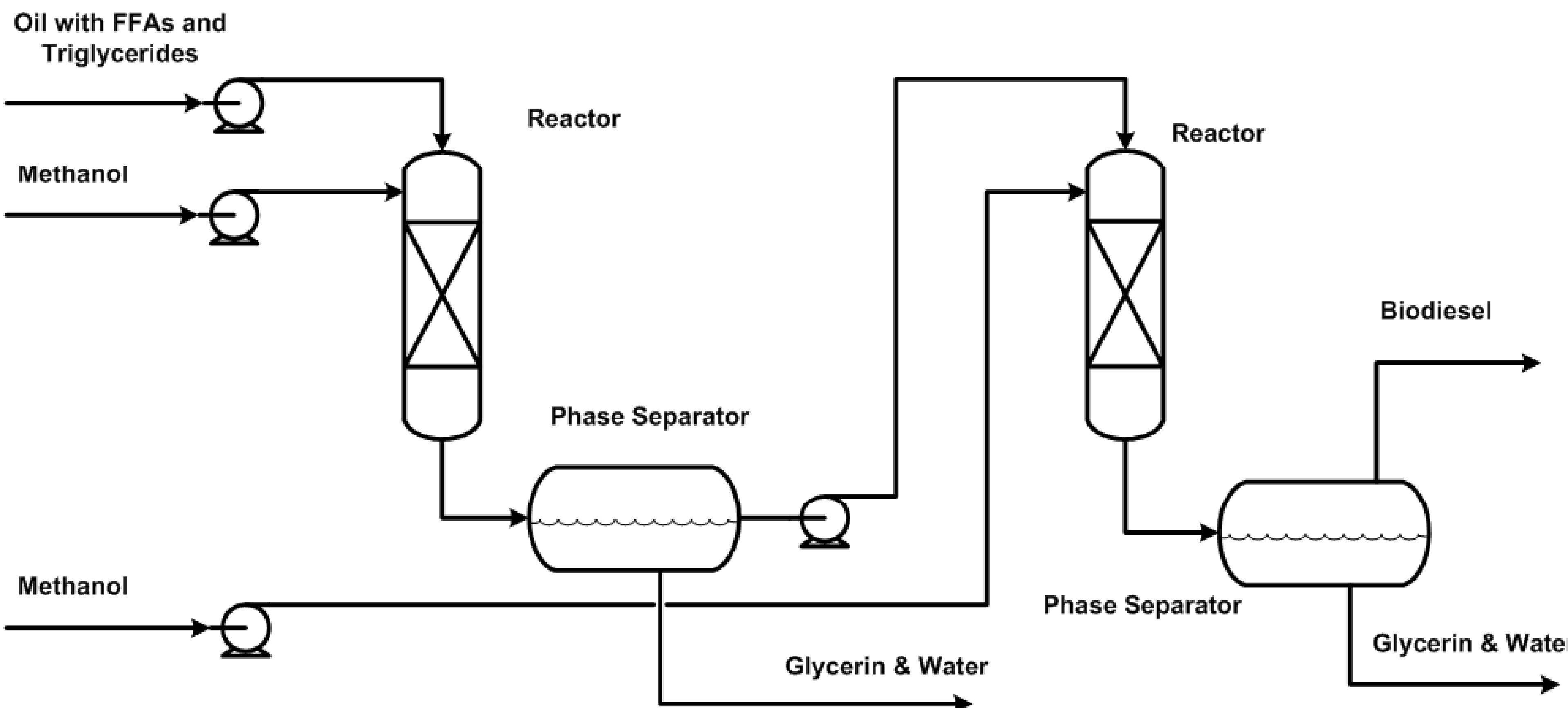
Newer Dual Reaction Technologies with Both Transesterification and Esterification

- NextCAT, Inc.
- Transbiodiesel, Ltd.
- Enhanced Biofuels, LLC

NextCAT, Inc.

- A dual catalyst packed bed reactor that performs both esterification and transesterification developed at Wayne State University in Detroit.
- Shows a very sharp separation between biodiesel and glycerin.
- No soap formation

Packed Tower Esterification & Tranesterification Reactors



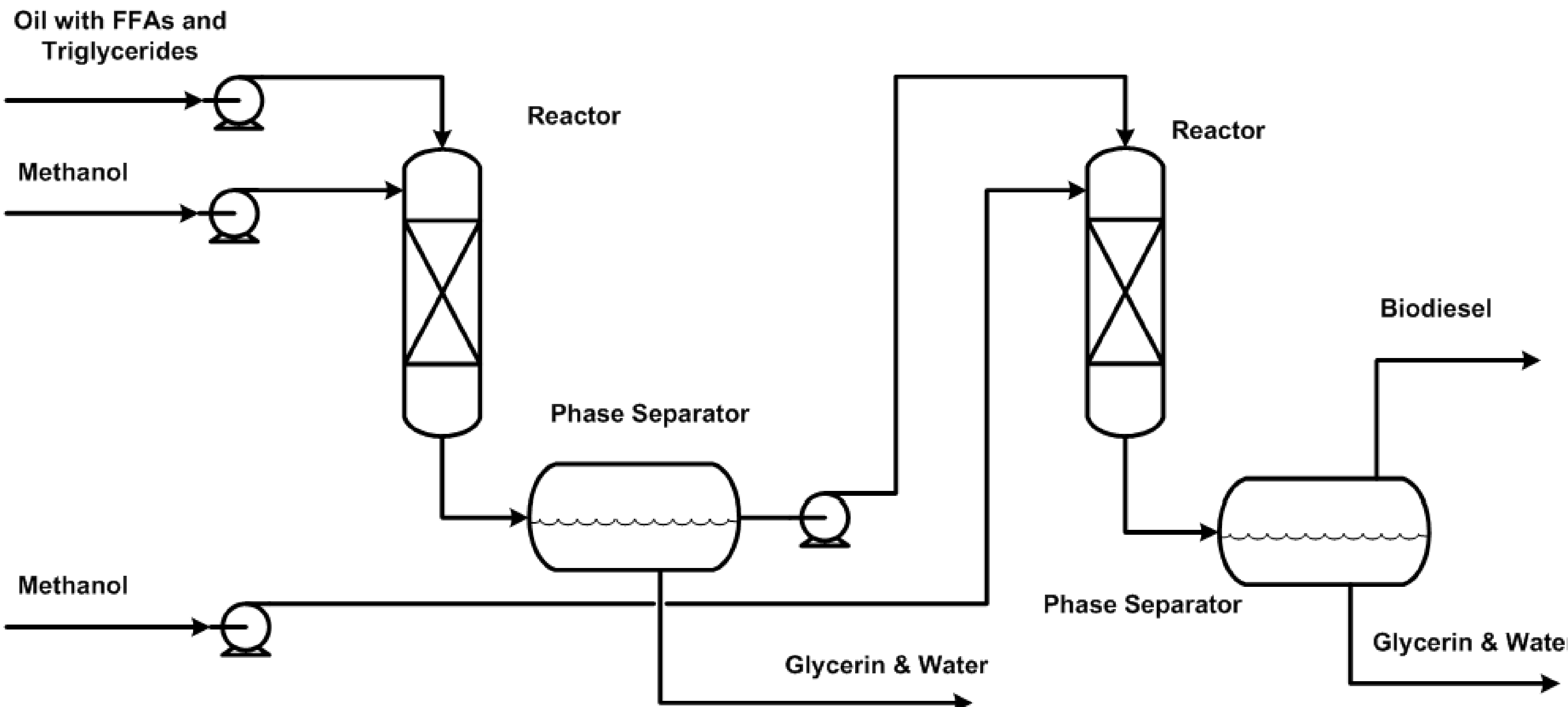
Transbiodiesel, Ltd.

- A dual lipase enzyme based packed bed reactor that performs both esterification and transesterification developed in Israel.

- Lipase enzyme is tethered to ion exchange beads. Most likely two modified lipases one for esterification and another for transesterification

- No soap formation

Packed Tower Esterification & Tranesterification Reactors



Enhanced Biofuels, LLC

Next Generation (HS Reactor System™)

What we do

- Process and upgrade high acidity feedstock
- Implementation includes add-ons and retrofits

How we do it

- Reactor design, process intensification, temperature, pressure and catalysts

Pilot Units

- 3,000 GPY and a 250,000 GPY

Examining Energy Maximization & Cost-Effectiveness

• Single largest operating cost has traditionally been feed stock.

•This will change as low cost feedstocks become available especially nonedible plant oils such as jatropha, camelina, algae oil and others.

• Energy consumption per lb gallon of biodiesel will become important and calculated on an ongoing basis.

Energy Requirements for a Biodiesel Plant

Basis

- Plant has degumming
- Dual solid catalyst esterification reactors in Series
- Dual Shockwave ® Reactors in Series
- Three distillation Columns
  - A column to strip methanol from Biodiesel
  - A column to strip methanol from glycerin
  - A column to dehydrate both methanol streams above and the methanol/ water from esterification

Energy Requirements for a 20 MGY Biodiesel Plant

Plant Size: 20,000,000 gallons per year

Unit	Motor	Steam - 150#	Steam - 30#	Condensate	Tower Water	Chilled Water	Chilled Water	Nitrogen	
	hp	lbs/hr	lbs/hr	gpm	gpm	mm btu/hr	gpm	Normal SCFM	Maximum SCFM
Connected loads									
100 - Degumming	93	1,026	0	2	113	0.0	5	0	0
200 - Esterification	52	1,804	0	4	424	0	0	0	0
300 - Transesterification	363	8,194	132	16	274	5	616	0	0
TOTAL	508	11,024	132	22	812	5	621	0	0
Operating loads									
100 - Degumming	70	1,026	0	2	113	0.0	5	0	0
200 - Esterification	26	1,804	0	4	424	0	0	0	0
300 - Transesterification	323	7,264	132	15	274	5	616	0	0
TOTAL	418	10,094	132	20	812	5	621	0	0

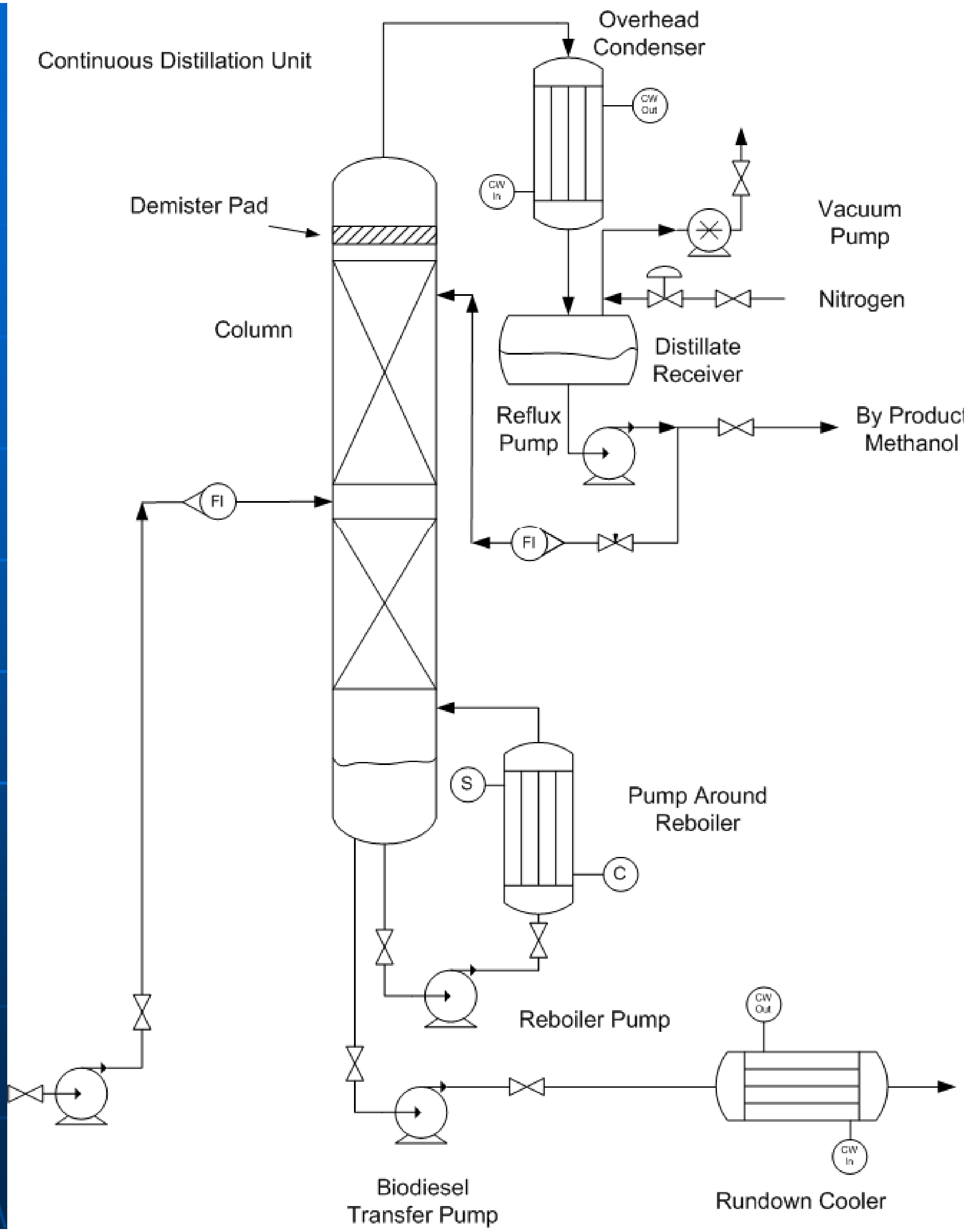
Energy Requirements for a 20 MGY Biodiesel Plant (Transesterification)

Tag	Service	PID	Motor	Steam - 150#	Steam - 30#	Condensate	Tower Water	Chilled Water	Chilled Water	Nitrogen	
			hp	lbs/hr	lbs/hr	gpm	gpm	mm btu/hr	gpm	Normal SCFM	Maximum SCFM
Columns											
C-301	BIODIESEL DISTILLATION COLUMN	MFD-341									
C-302	GLYCEROL DISTILLATION COLUMN	MFD-351			150						
C-303	METHANOL TOWER	MFD-356									
C-304	VENT SCRUBBER	MFD-370									
Exchangers											
E-303	OIL DRYER HEATER	MFD-300		2,325		5					
E-317	OIL FEED DRUM HEATER	MFD-300			275						
E-321	OIL DRYING CONDENSER	MFD-300					13				
E-301	REACTOR OIL HEATER	MFD-315		2,325		5					
E-302	REACTOR METHANOL HEATER	MFD-315		745		1					
E-320	1ST STAGE COOLER	MFD-320					167				
E-312	2ND STAGE REACTOR HEATER	MFD-325		355		1					
E-304	REACTOR PRODUCT COOLER	MFD-335					179				
E-307	BIODIESEL FEED/BOTTOMS EXCHANGER	MFD-341									
E-308	BIODIESEL PRODUCT RUNDOWN COOLER	MFD-341					292				
E-305	BIODIESEL TOWER REBOILER	MFD-342		2,790		6					
E-306	BIODIESEL TOWER CONDENSER	MFD-343						1.20	160		
E-310	GLYCEROL FEED/BOTTOMS EXCHANGER	MFD-351									
E-316	GLYCEROL PRODUCT RUNDOWN COOLER	MFD-351					24				
E-309	GLYCEROL TOWER REBOILER	MFD-352		5,120		10					
E-311	GLYCEROL TOWER CONDENSER	MFD-353						4.57	600		
E-313	METHANOL TOWER REBOILER	MFD-357		6,825		14					
E-319	WASTE WATER COOLER	MFD-357					11				
E-314	METHANOL TOWER CONDENSER	MFD-358						5.86	780		
E-315	SEAL WATER COOLER (BY VENDOR)	MFD-370									
Tanks											
T-303	MeOH DAY TANK	MFD-305									
T-304	CATALYST DAY TANK	MFD-310									
Vacuum Pump											
CP-301	DISTILLATION VACUUM PUMP	MFD-370	5								

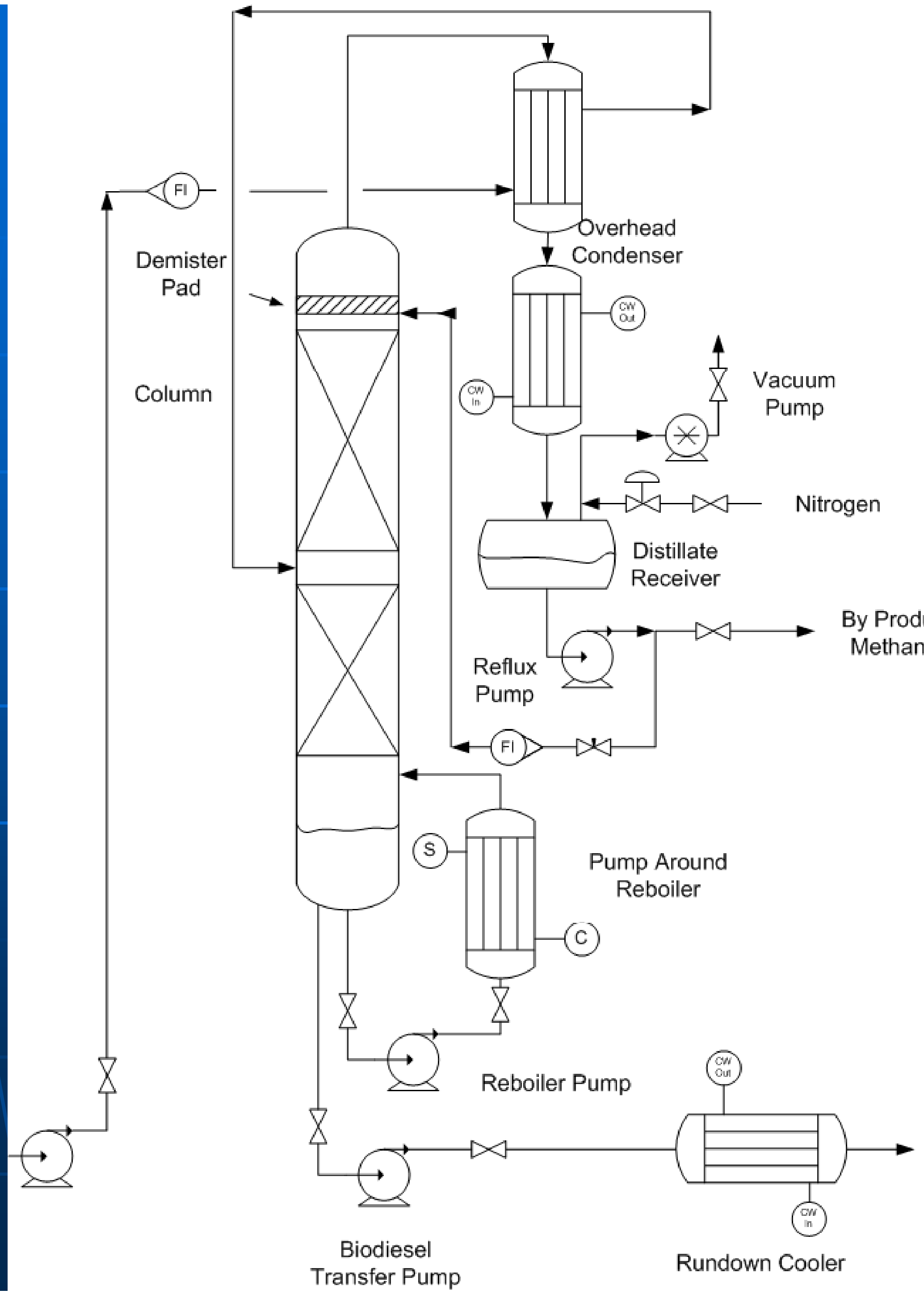
Energy Requirements for a 20 MGY Biodiesel Plant

- Transesterification has the highest energy requirements
- The three distillation columns are the biggest energy users within tranesterification
- What can be done?

Energy Conservation Schemes Around Distillation



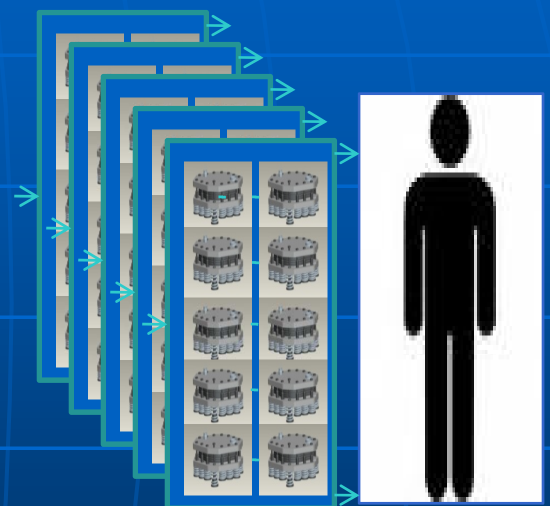
Energy Conservation Schemes Around Distillation



The iPod for distillation

Today's Distillation

fluXXion HEC: High Efficiency Contactor



Diameter 0.5-6 m
Height 5-100 m
Weight 1-100 ton

100 times smaller

Modular system
Microsieve stacks
Box system

Fast, Flexible and Safe

Today's Distillation

High energy loss

Separation inside column, heat recovery outside column

Inflexible system

Large dimensions

High installation cost

Well proven system

fluxxion

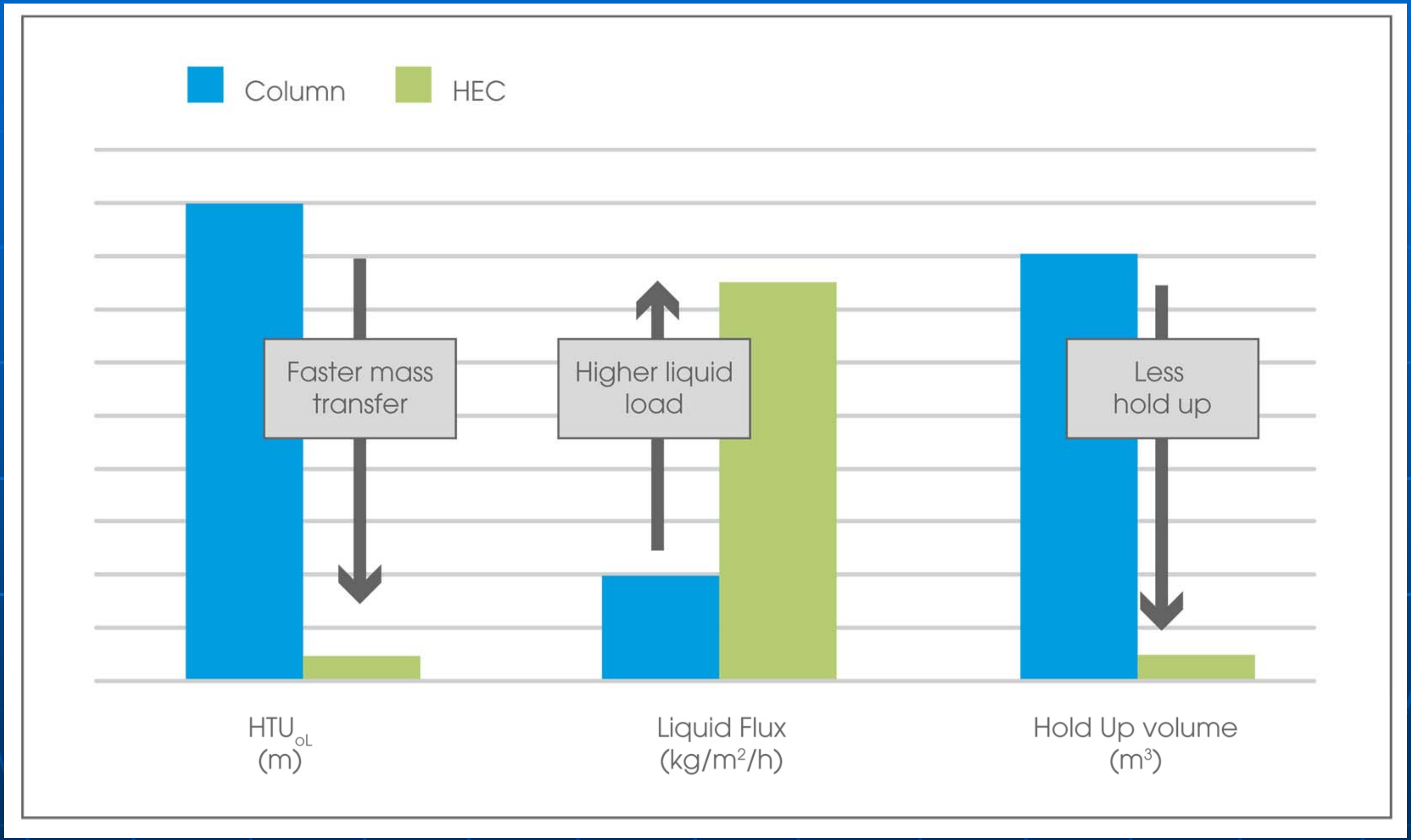
Integrated heat and mass transfer
- 100-1000x faster
- 20-40% energy savings

High flexibility
- Capacity in operation
- Modularity
- Purity
- Products

Small dimensions
- Easy to place anywhere

Enhanced safety
- small liquid hold up

Small, Flexible, Safe



- Stable operation (large pressure differential)
- No moving parts
- Individual module/stack temperature control possible
- High pressure operation possible (put module in autoclav

- Hazardous materials & pathways
- capacity & purity flexibility
- volume/height restricted situations
- low gas volume stripping

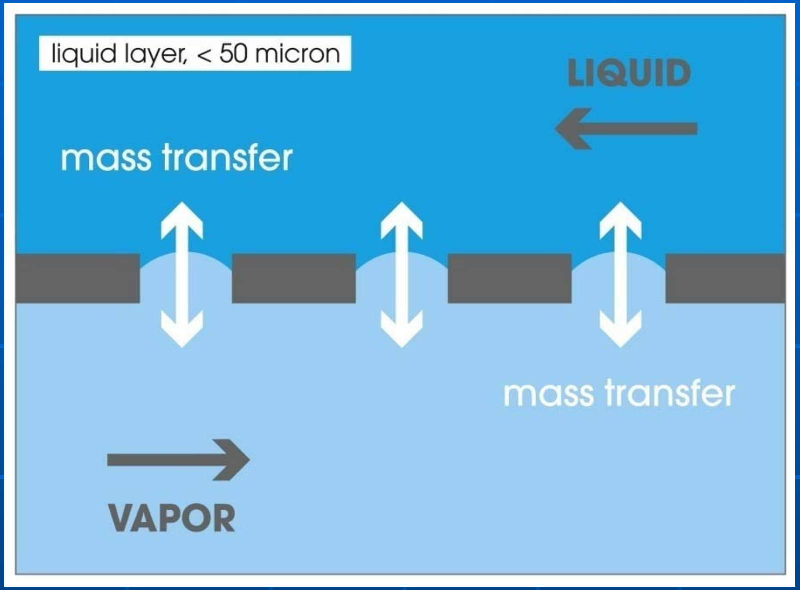
Proven Science

UNIQUE MICROSIEVE TECHNOLOGY

15 cm diameter

0,7 mm thick

High volume production



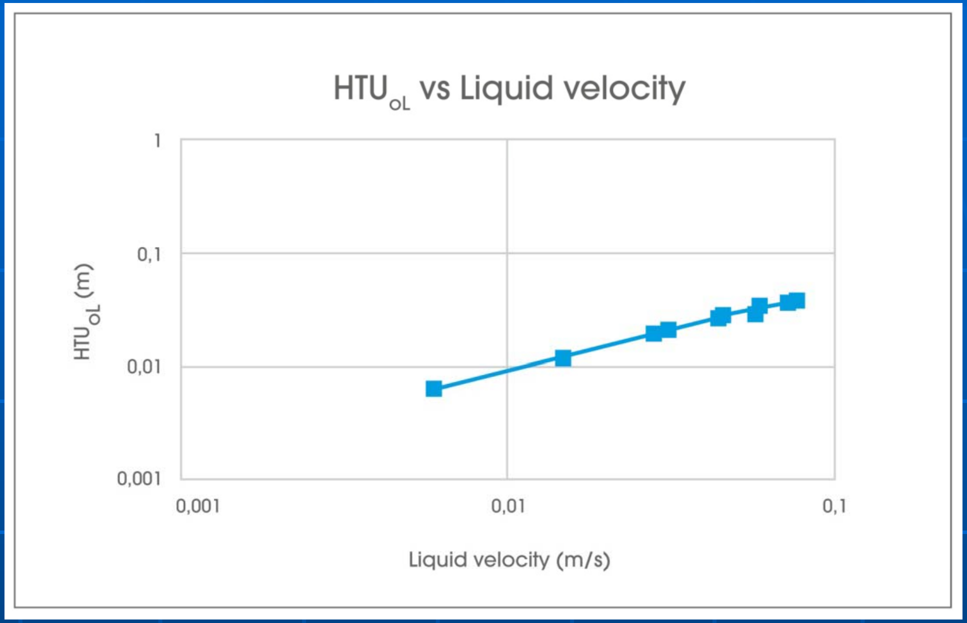
5 billion identical pores

0,45 micron diameter

1 micron membrane thickness

PROOF: ultra short HTU_{OL}

Stripping MTBE from water by nitrogen at ambient conditions was used as a test system for performance evaluation purposes. Inlet concentration of MTBE , different flow rates for liquid and gas were varied in a wide range. The best efficiency was achieved in all cases at lowest liquid flow rate, and tended to decrease progressively with increasing liquid flow rate, i.e. decreasing residence time. Gas flow rate exhibited practically no effect on separation, indicating that this system also can be considered as fully controlled by liquid side mass transfer resistance. Since the value of the overall liquid side based volumetric mass transfer coefficient was almost constant, the overall height of liquid side transfer unit (HTUoL) increased nearly proportionally and ranged 0.5-4 cm (!). The corresponding heights equivalent to a theoretical plate (HETP values) were 2 to 10 cm (!). These numbers indicate a high mass transfer efficiency, an order of magnitude above that experienced with common types and sizes of corrugated sheet structured packing in similar applications.



What Else Can Be Done?

- If your plant is large enough consider an anaerobic digester using glycerin to produce biogas.
- Remove the water and hydrogen sulfide from the biogas.
- Feed the low Btu gas (650-750 BTUs/ SCF) to the boilers that produce steam for the plant and especially the distillation columns.

Improving Yields in Tranesterification

- STT ® Reactor 99.7% conversion in one reactor
- Shockwave ® Power Reactor 99.99% conversion in two (2) reactors in series
- Better than the continuous stirred tank reactors CSTRs with (2) reactors in series.

Advantages of Improving Yields

- More biodiesel produced per lb of incoming oil
- Less Soap produced because of low residence times
- Sharper phase separation since unreacted mono and diglycerides are emulsifiers. Now they don’t exist.

Disadvantages of Improving Yields with Extra Methanol

- Use more methanol upfront to improve conversion but then you will have higher distillation costs.

- Typically 100% excess of theoretical methanol is used. That is 6 moles of methanol per mole of triglyceride.

Improving Yields in Two Column Systems

- Add a third reactor but this is costly
- If we have 99% conversion per pass
- With 15% free fatty acids in an esterification system only
  - After the first reactor 15%* (1-.9)= 1.5%
  - After a second reactor 1.5 %* (1-.9)= 0.15%
  - After a third reactor 0.15 %* (1-.9)= 0.015%

Safety Considerations

- During the years 2007 to 2009 there were 26 fires and or explosions in the US from methanol vapors in biodiesel plants.

- During this same period there were two (2) deaths

Three Explosions (1 of 3)

One consisted of a man welding on top of tank partially filled with glycerin and methanol. The methanol vapors in the top of the tank ignited causing a manway cover to hit the man and killing him.

The tank should have been emptied and dried and purged with air. An LFL meter would determine if a flammable mixture existed or not existed prior to welding.

Three Explosions (2 of 3)

Another consisted of the ignition of a methanol vapor cloud by a genie garage door opener.

An operator opened a hot vessel filled with hot glycerin and methanol vapors to check the level in the vessel.

The vapors poured into the room.

Someone opened the garage door to vent the vapors

The motor on the door opener ignited the vapors.

Three Explosions (3 of 3)

The American Biofuels plant in Bakersfield, Calif., burned to the ground when a spill from a methanol-filled transport tote was ignited.

Why?

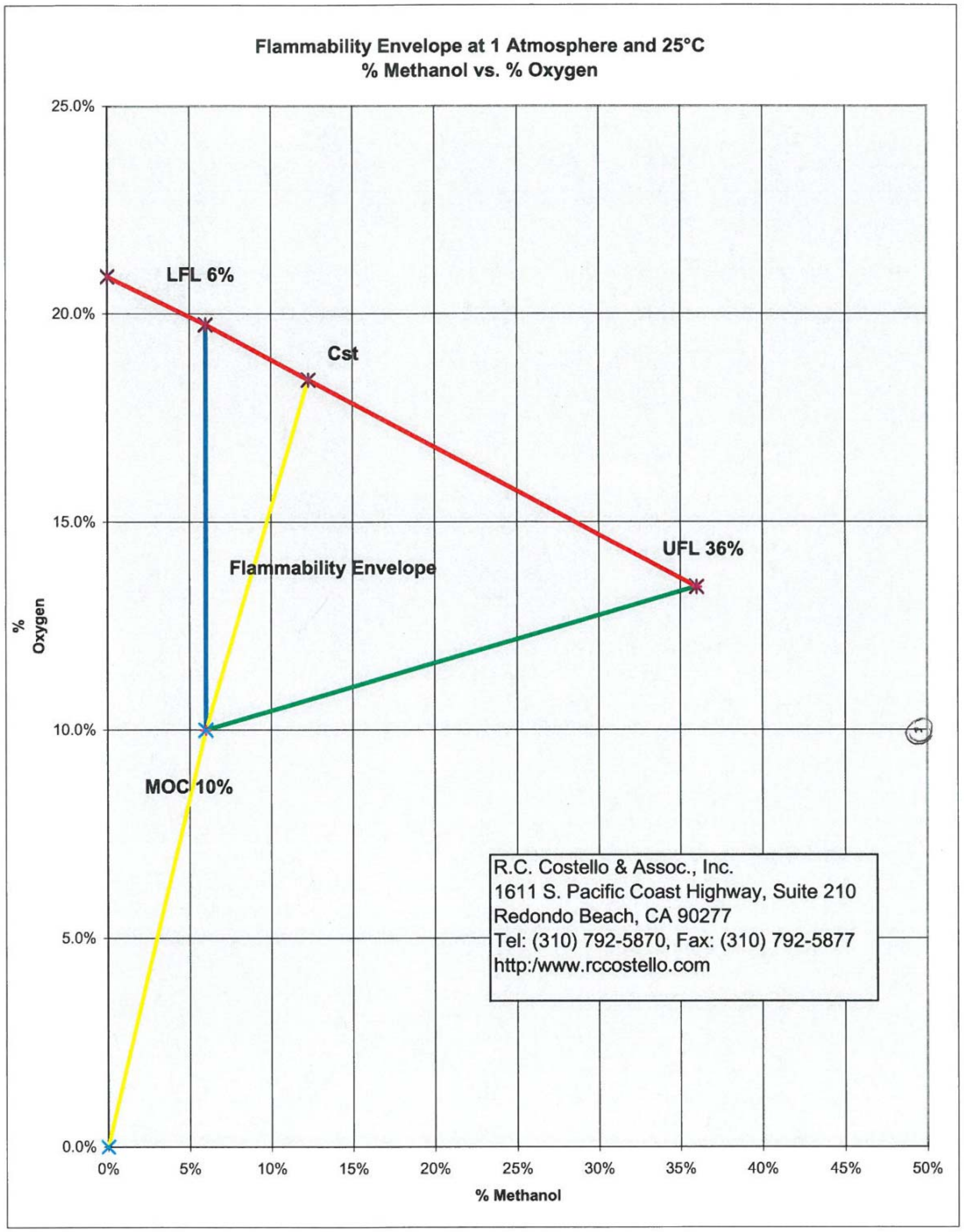
- Unsophisticated builders try to save money with lower cost plants. They have no prior experience in the chemical industry. Do not buy on price.
- Unsophisticated buyers.
- These lower cost plants have low on stream factor (Low reliability). I.e. installed spare pumps.
- These low cost plants are not safe.

What have I observed?

- Non explosion proof motors
- Vessels operating at 50 psig without an ASME code stamp.
- Non explosion proof wiring
- Non explosion proof instrumentation
- No Hazards Analysis ever performed!!! (If you have 10,000 lbs on methanol on hand OSHA requires a Hazards Analysis)

What have I observed?

- Stripping methanol out of Biodiesel with air. This creates an explosive flammable mixture. All we need now is an ignition source. Such as a an electrical spark. This is a short cut approach when no knowledge of vacuum distillation exists.


Flammability Envelope at 1 Atmosphere and 25°C
% Methanol vs. % Oxygen
25.0%
20.0%
15.0%
10.0%
5.0%
0.0%
% Oxygen
0%
5%
10%
15%
20%
25%
30%
35%
40%
45%
50%
% Methanol
LFL 6%
Cst
UFL 36%
Flammability Envelope
MOC 10%
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Things you should know and ask your plant designer / builder / supplier:

Was your plant designed by licensed professional engineers?

Is your plant design compliant with the latest version of the National Electric Code and all of the National Fire Protection Association codes for flammable liquids and vapors?

Does your plant designer understand why you must use explosion-proof motors, explosion-proof wiring and explosion-proof field instruments when using flammable liquids such as methanol?

Is your lighting in the building explosion-proof, or could it be an ignition source?

Is your plant design compliant with the state pressure vessel code for all vessels operating at pressures above 14.9 pounds per square inch gauge?

Things you should know and ask your plant designer / builder / supplier:

Are you, as the owner, compliant with local construction codes?

Does your city require civil/structural drawings and load calculations to be prepared by a licensed civil engineer, with final approval by the city prior to starting construction?

Does your city require electrical drawings and electrical load calculations to be prepared by a licensed electrical engineer, with final approval by the city prior to starting construction?

Have you performed a Hazard and Operability (HAZOP) study prior to beginning construction, as required by the Occupational Safety and Health Administration (OSHA) and most likely your state equivalent?

Have you obtained all necessary air permits?

Things you should know and ask your plant designer/builder/supplier:

Have you obtained all necessary water permits to discharge water, which would include either a Publicly Owned Treatment Works (POTW) permit or a National Pollution Discharge Elimination System (NPDES) permit to discharge to a local body of water?

Are you meeting all of OSHA's federal industrial hygiene requirements and your state's industrial hygiene requirements regarding worker exposure to methanol vapors?

Does your plant have vessel entry procedures?

Does your plant have lock-out/tag-out procedures?

Do you have written start-up and shutdown procedures?

Things you should know and ask your plant designer / builder / supplier:

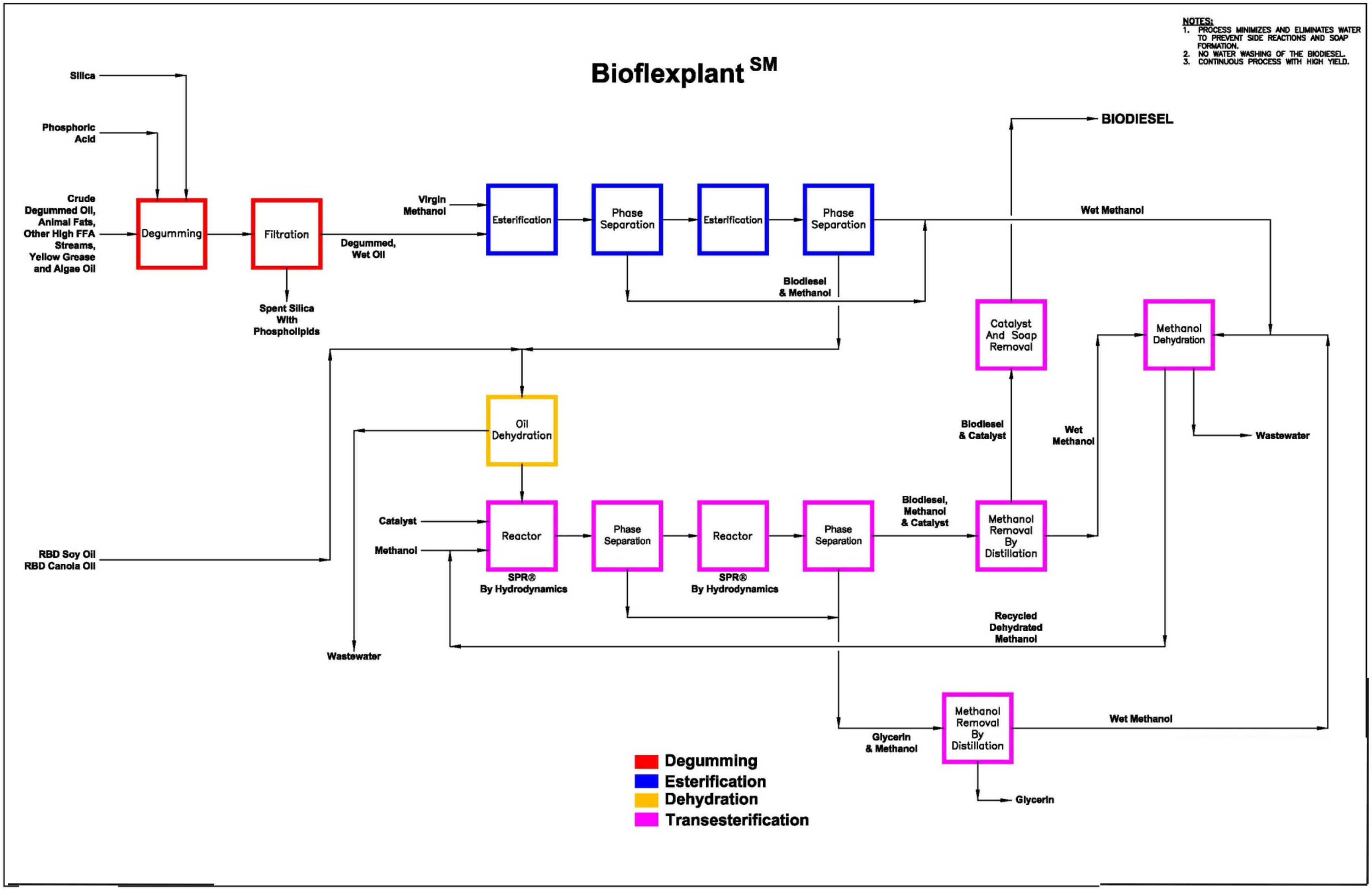
Do you have written start-up and shutdown procedures?

Are pressure vessels properly protected by safety relief valves? Do these relief valves discharge to a safe location?

Are storage tanks designed and laid out in accordance with American Petroleum Institute guidelines? Are the tanks located within containment berms designed for the event of a tank failure?

Do you have written operating procedures that include instructions for fire and explosion handling?

Source: Rocky C. Costello, R.C. Costello & Assoc., Inc.


NOTES:
1. PROCESS MINIMIZES AND ELIMINATES WATER TO PREVENT SIDE REACTIONS AND SOAP FORMATION.
2. NO WATER WASHING OF THE BIODIESEL.
3. CONTINUOUS PROCESS WITH HIGH YIELD.
Bioflexplant SM
Silica
Phosphoric Acid
Crude Degummed Oil, Animal Fats, Other High FFA Streams, Yellow Grease and Algae Oil
Degumming
Filtration
Spent Silica With Phospholipids
Degummed, Wet Oil
Virgin Methanol
Esterification
Phase Separation
Esterification
Phase Separation
Biodiesel & Methanol
Wet Methanol
BIODIESEL
Catalyst And Soap Removal
Methanol Dehydration
Oil Dehydration
Wastewater
Biodiesel & Catalyst
Wet Methanol
Wastewater
RBD Soy Oil
RBD Canola Oil
Catalyst
Methanol
Reactor
SPR® By Hydrodynamics
Phase Separation
Reactor
SPR® By Hydrodynamics
Phase Separation
Biodiesel, Methanol & Catalyst
Methanol Removal By Distillation
Recycled Dehydrated Methanol
Glycerin & Methanol
Methanol Removal By Distillation
Wet Methanol
Glycerin
Degumming
Esterification
Dehydration
Transesterification

Summarizing

- Smaller intensified reactors exist now for Transesterification.
- Packed bed reactors exist for Esterification
- Dual process packed bed reactors for Esterification & Transesterification are on the way.
- Energy usage will become more important.
- Plants that can handle multiple feedstocks will survive.
- Good safety is good business.