Pretest

What industry has the largest fermentation capacity?

What industry has the largest fermentation capacity?

- Pharmaceuticals

What industry has the largest fermentation capacity?

- ☐ Pharmaceuticals
- ☐ Wine

What industry has the largest fermentation capacity?

- ☐ Pharmaceuticals
- ☐ Wine
- ☐ Beer

What industry has the largest fermentation capacity?

- ☐ Pharmaceuticals
- ☐ Wine
- ☐ Beer
- ☐ Wastewater treatment

What industry has the largest fermentation capacity?

- ☐ Pharmaceuticals
- ☐ Wine
- ☐ Beer
- ☒ Wastewater treatment

What industry uses mixed-culture fermentation?

What industry uses mixed-culture fermentation?

- Pharmaceuticals

What industry uses mixed-culture fermentation?

- Pharmaceuticals
- Wine

What industry uses mixed-culture fermentation?

- Pharmaceuticals
- Wine
- Beer

What industry uses mixed-culture fermentation?

- Pharmaceuticals
- Wine
- Beer
- Wastewater treatment

What industry uses mixed-culture fermentation?

- ☐ Pharmaceuticals
- ☐ Wine
- ☐ Beer
- ☒ Wastewater treatment

Would you employ a mono-culture to treat wastewater?

- ☐ Yes
- ☐ No

Would you employ a mono-culture to treat wastewater?

☐ Yes

☒ No

Should farmers employ mono-cropping?

- ☐ Yes
- ☐ No

Should farmers employ mono-cropping?

☐ Yes

☒ No

Should you invest your life savings in one stock?

- ☐ Yes
- ☐ No

Should you invest your life savings in one stock?

☐ Yes

☒ No

Conclusion

Diversity is good

Routes to Fuel

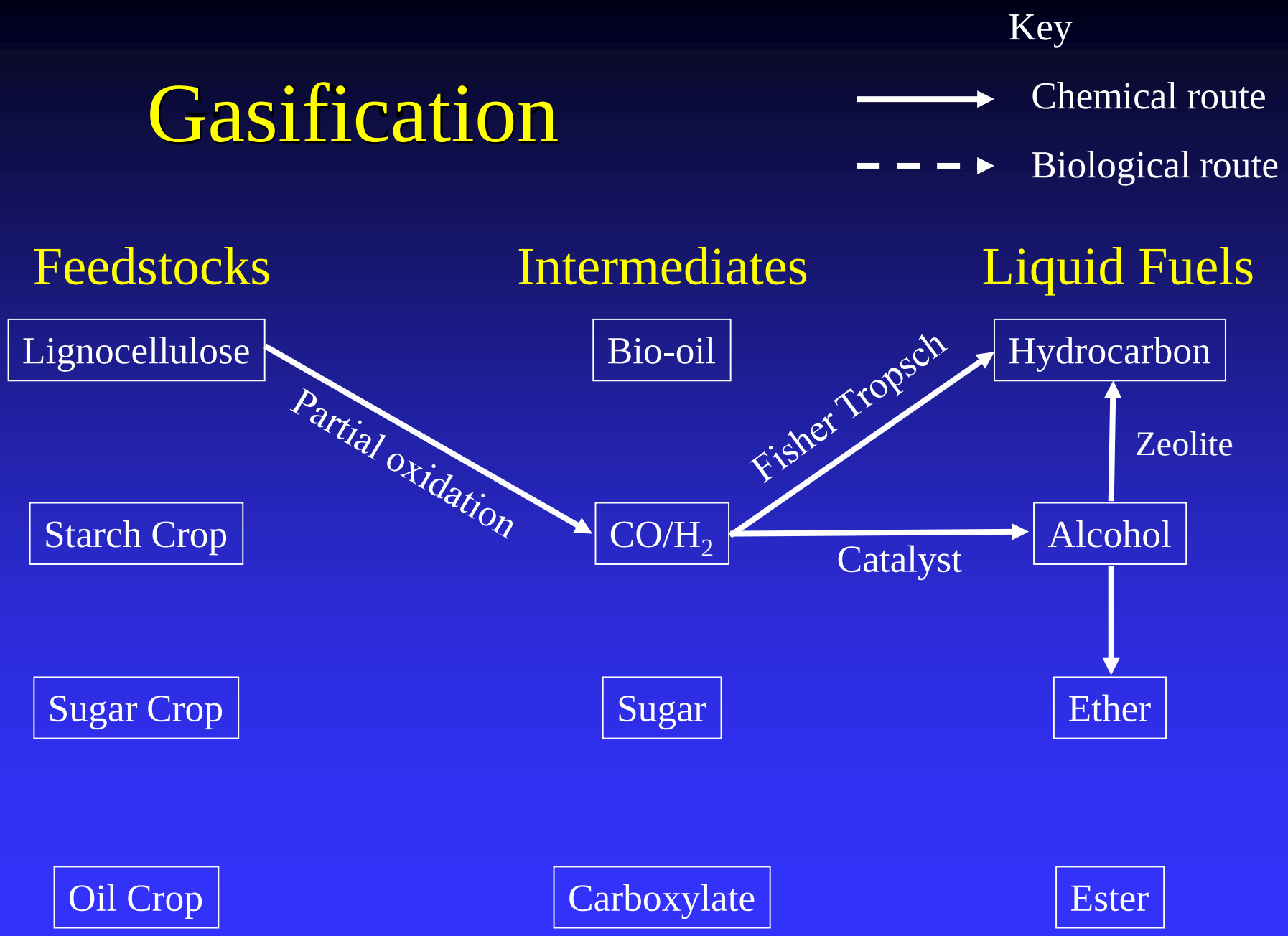
Key

Chemical route

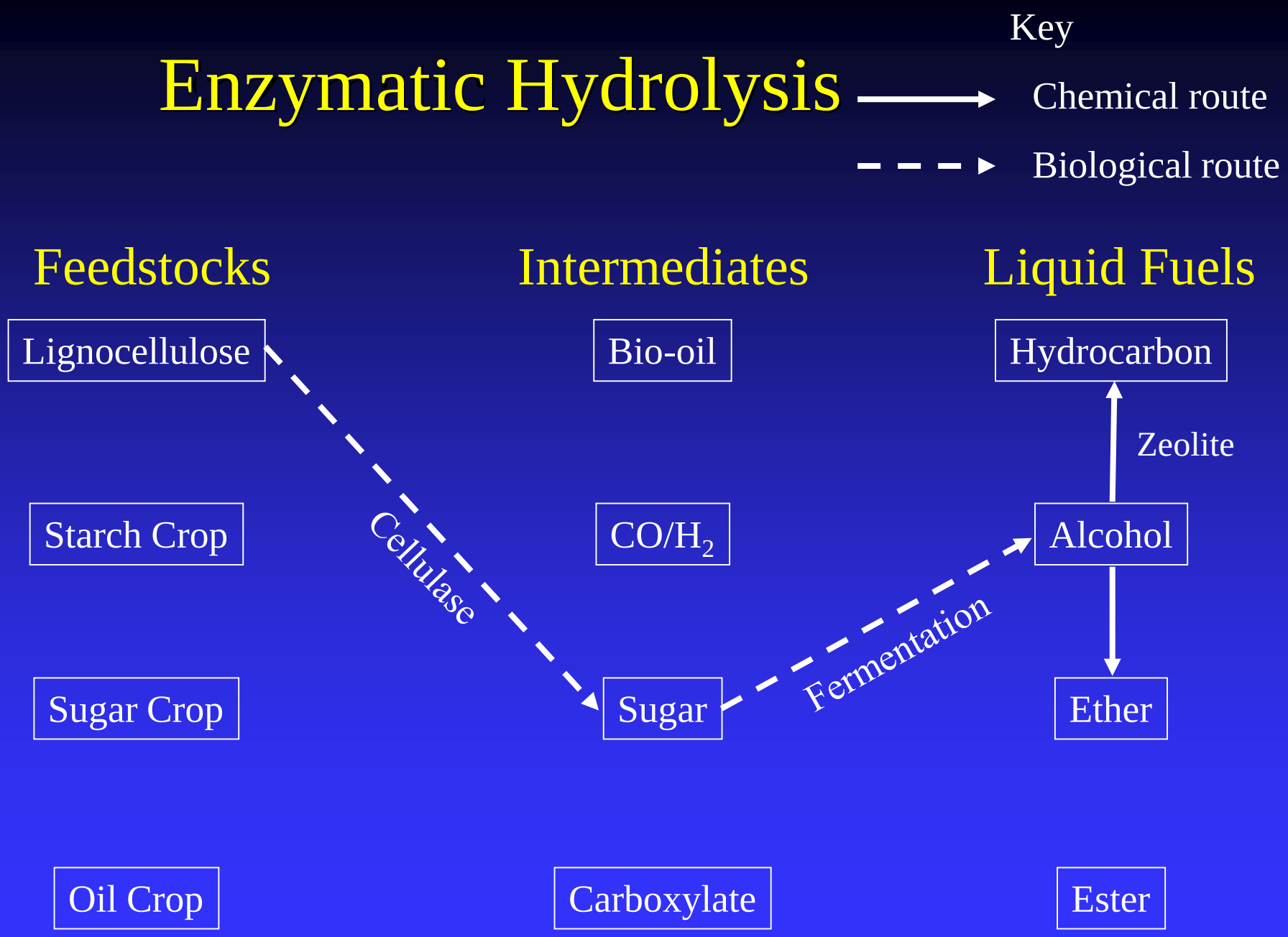
Biological route

Feedstocks	Intermediates	Liquid Fuels
Lignocellulose	Bio-oil	Hydrocarbon
Starch Crop	CO/H_2	Alcohol
Sugar Crop	Sugar	Ether
Oil Crop	Carboxylate	Ester

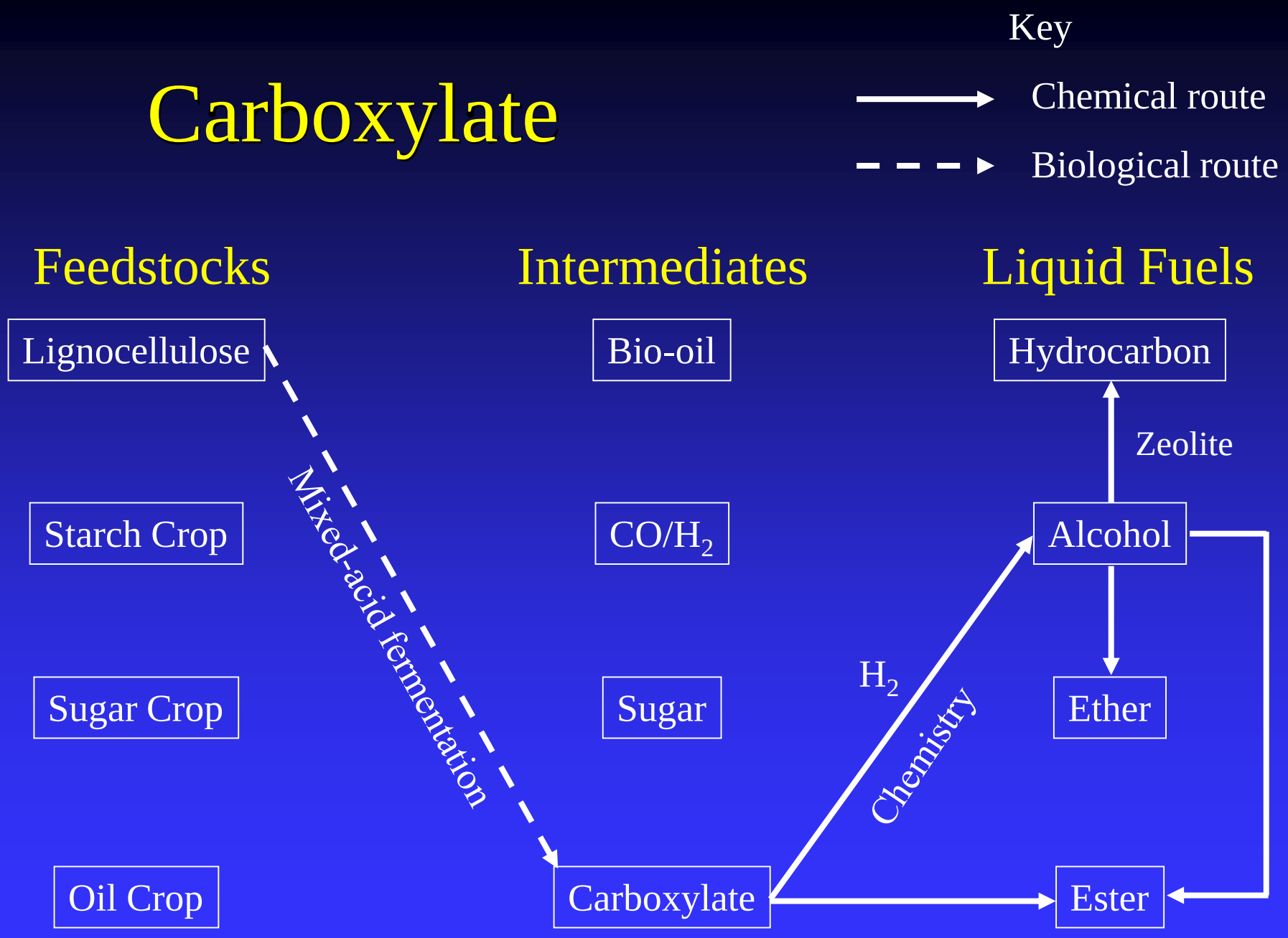
Gasification



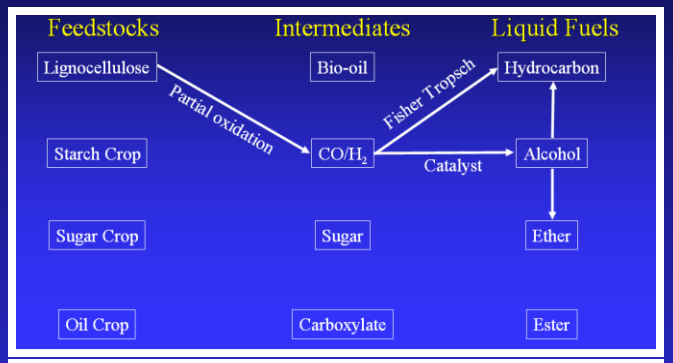
Enzymatic Hydrolysis



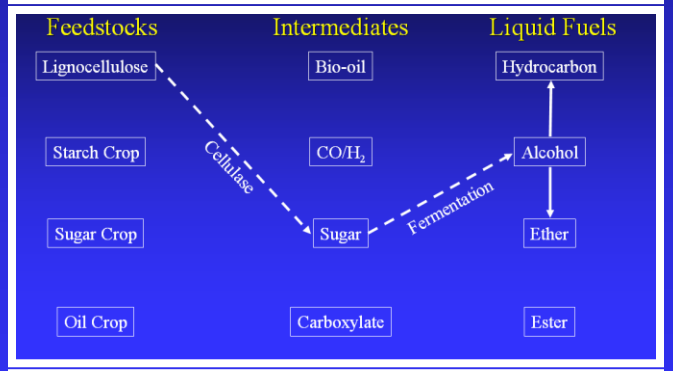
Carboxylate



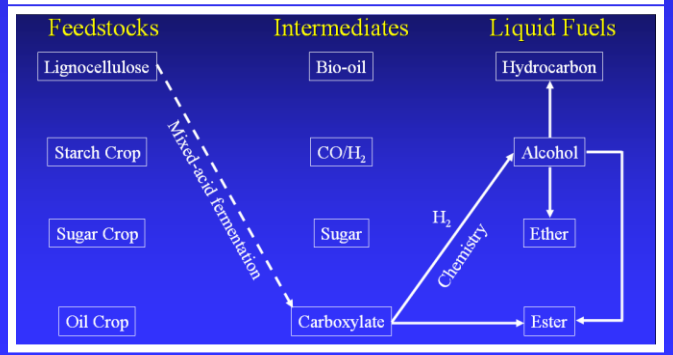
Compare three routes



Thermochemical Platform



Sugar Platform



Carboxylate Platform

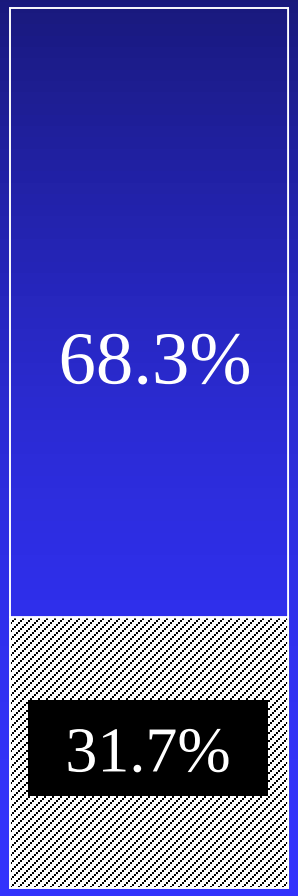
Compare on an equal basis

Polysaccharides

($C_6H_{10}O_5$)

Lignin

($CH_{1.12}O_{0.377}$)

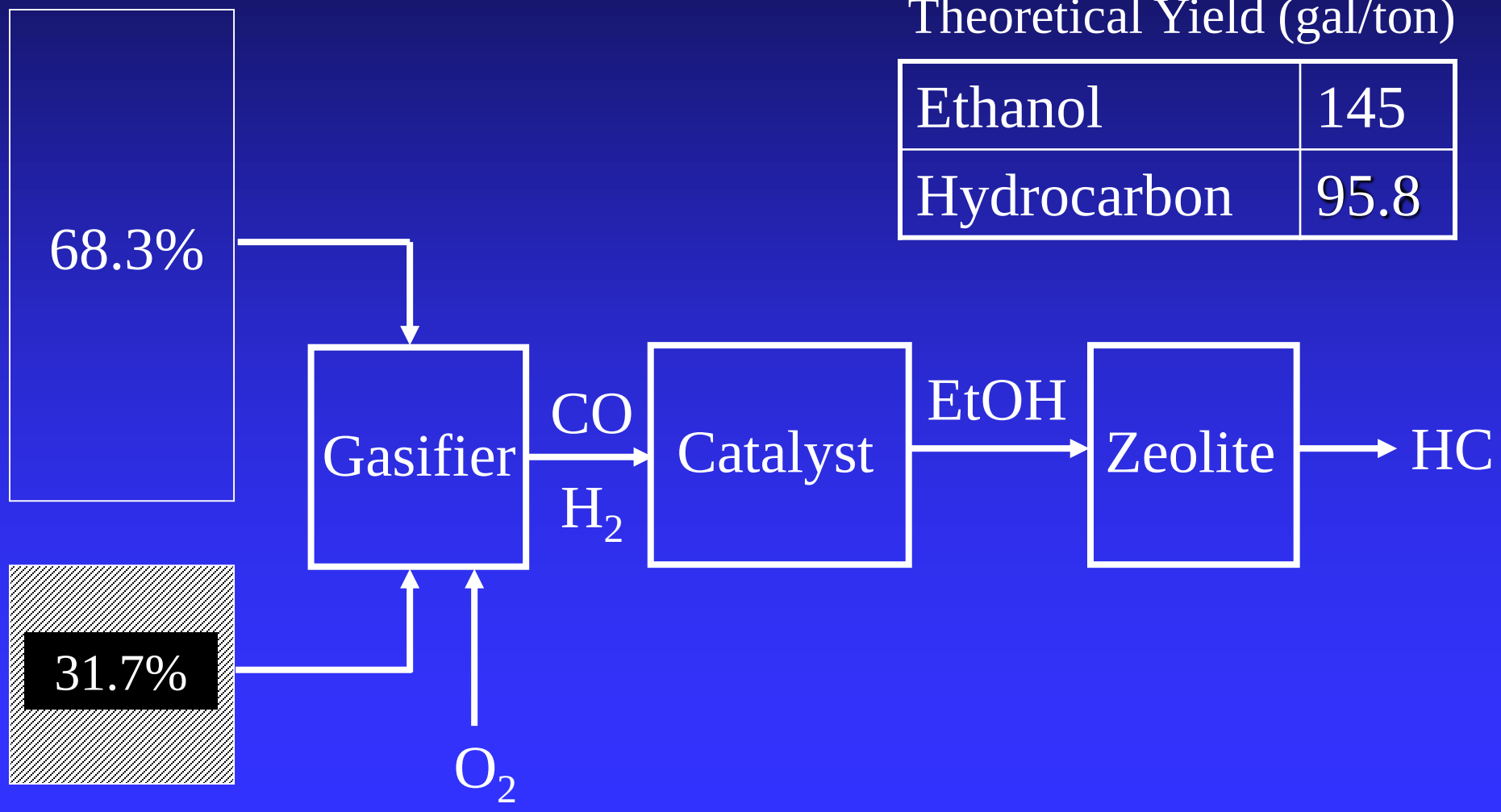


"Balanced Biomass"

Thermochemical Platform

Theoretical Yield (gal/ton)

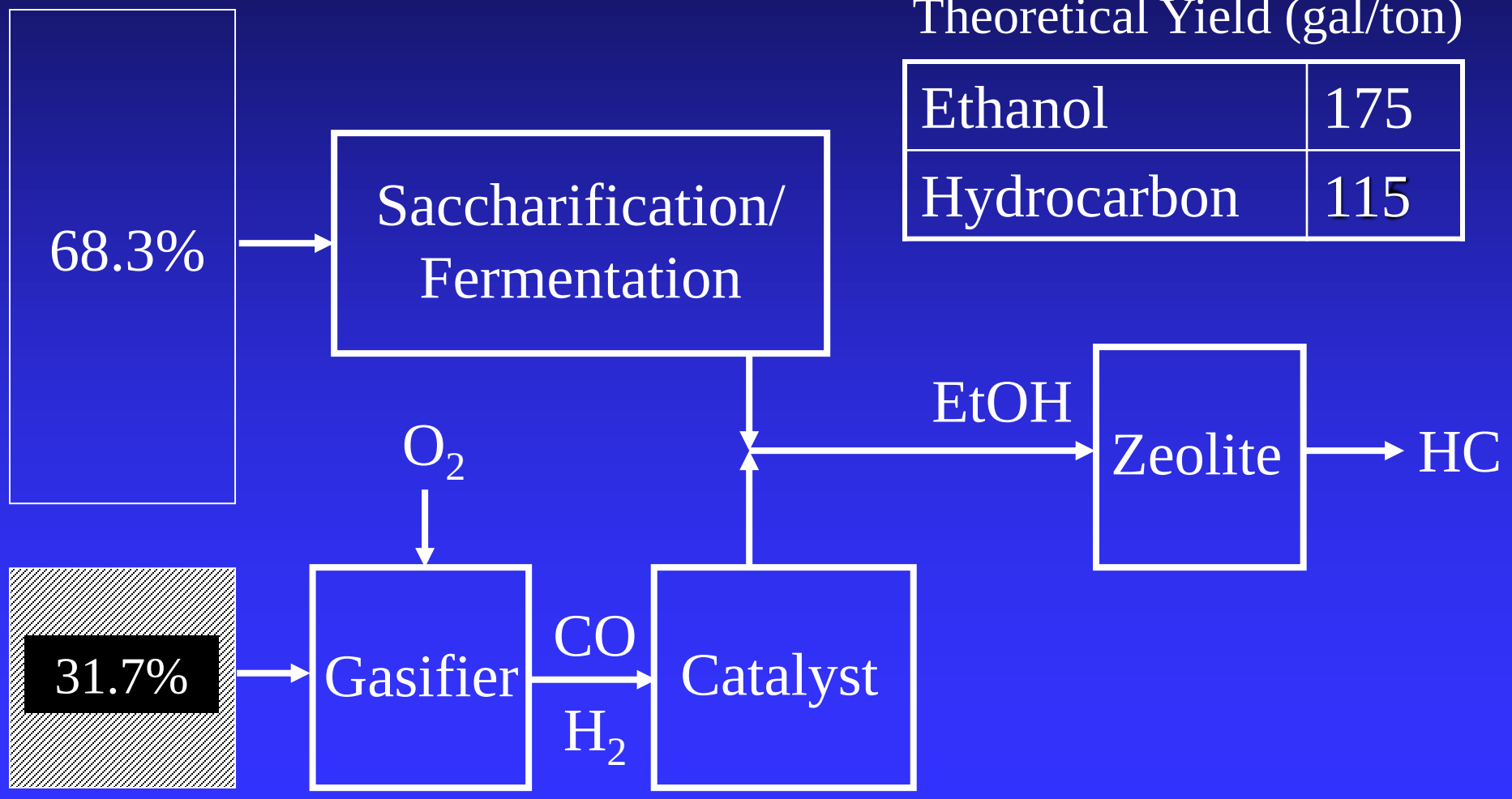
Ethanol	145
Hydrocarbon	95.8



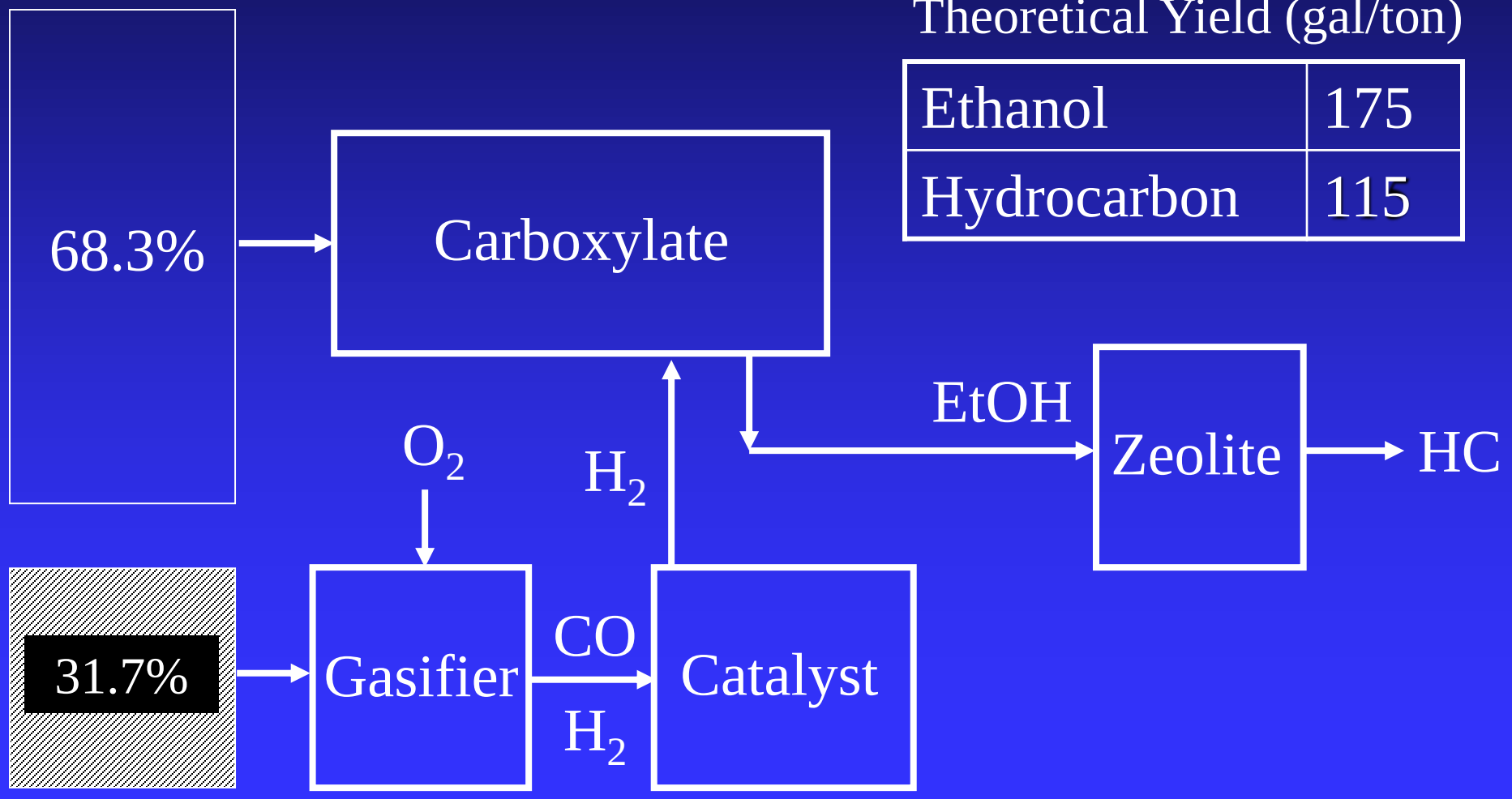
Sugar Platform

Theoretical Yield (gal/ton)

Ethanol	175
Hydrocarbon	115



Carboxylate Platform



Theoretical Yield (gal/ton)

Ethanol	175
Hydrocarbon	115

Summary

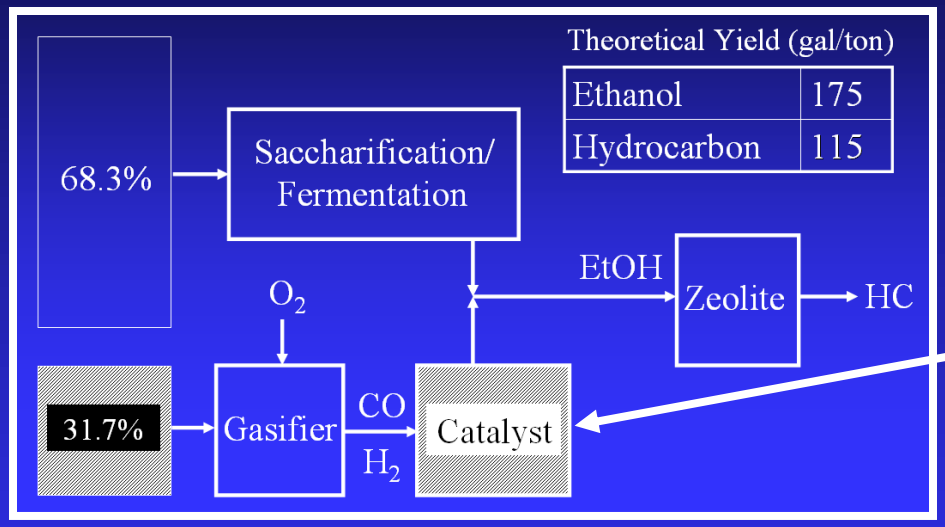
	Theoretical Yield (gal/ton)	
	Ethanol	Hydrocarbon
Thermochemical	145	95.8
Sugar	175	115
Carboxylate	175	115

Summary

	Theoretical Yield (gal/ton)	
	Ethanol	Hydrocarbon
Thermochemical	145	95.8
Sugar	175	115
Carboxylate	175	115

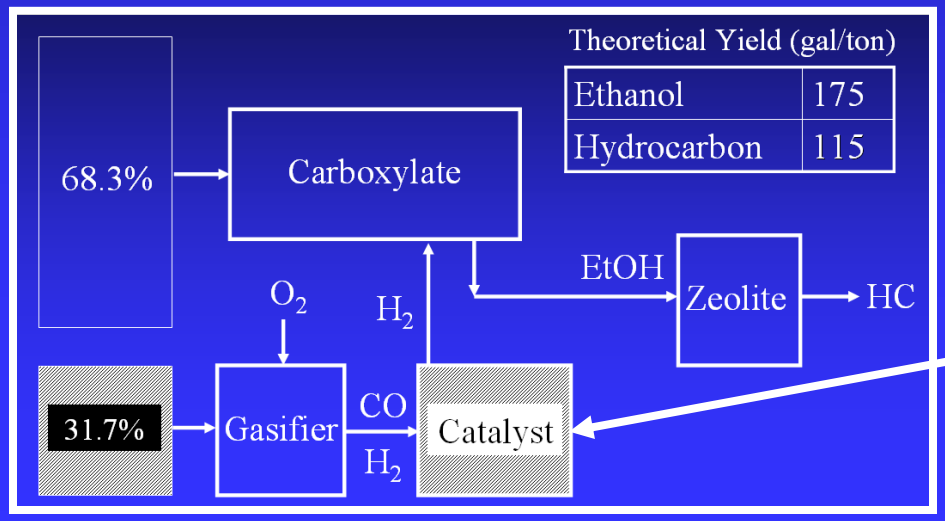
Partial oxidation of polysaccharides → Lower yield

Sugar vs Carboxylate Platforms



Sugar Platform

Alcohol catalyst – difficult



Carboxylate Platform

Shift catalyst – easy

Comparison of Three Platforms

	High Yield	Easy Catalyst
Thermochemical		
Sugar	×	
Carboxylate	×	×

Comparison of Three Platforms

	High Yield	Easy Catalyst
Thermochemical		
Sugar	×	
Carboxylate	×	×

Worth further investigation

Carboxylate Platform: Chemicals and Fuels from Biomass

Mark Holtzapple
Department of Chemical Engineering
Texas A&M University
College Station, TX

Commercial Name

MixAlco Process:

Chemicals and Fuels from Biomass

Mark Holtzapple
Department of Chemical Engineering
Texas A&M University
College Station, TX

Outline

- Carboxylate platform (MixAlco)
  - Primary alcohol route
  - Secondary alcohol route
  - Kolbe electrolysis
- Economics
- History
- Conclusions
- Future improvements

MixAlco Advantages

- Can use wet feedstocks
- Wide range of feedstocks
- Energy efficient
- No genetically modified organisms
- Many products
- Distributed processing

MixAlco Advantages

- Can use wet feedstocks
- Wide range of feedstocks
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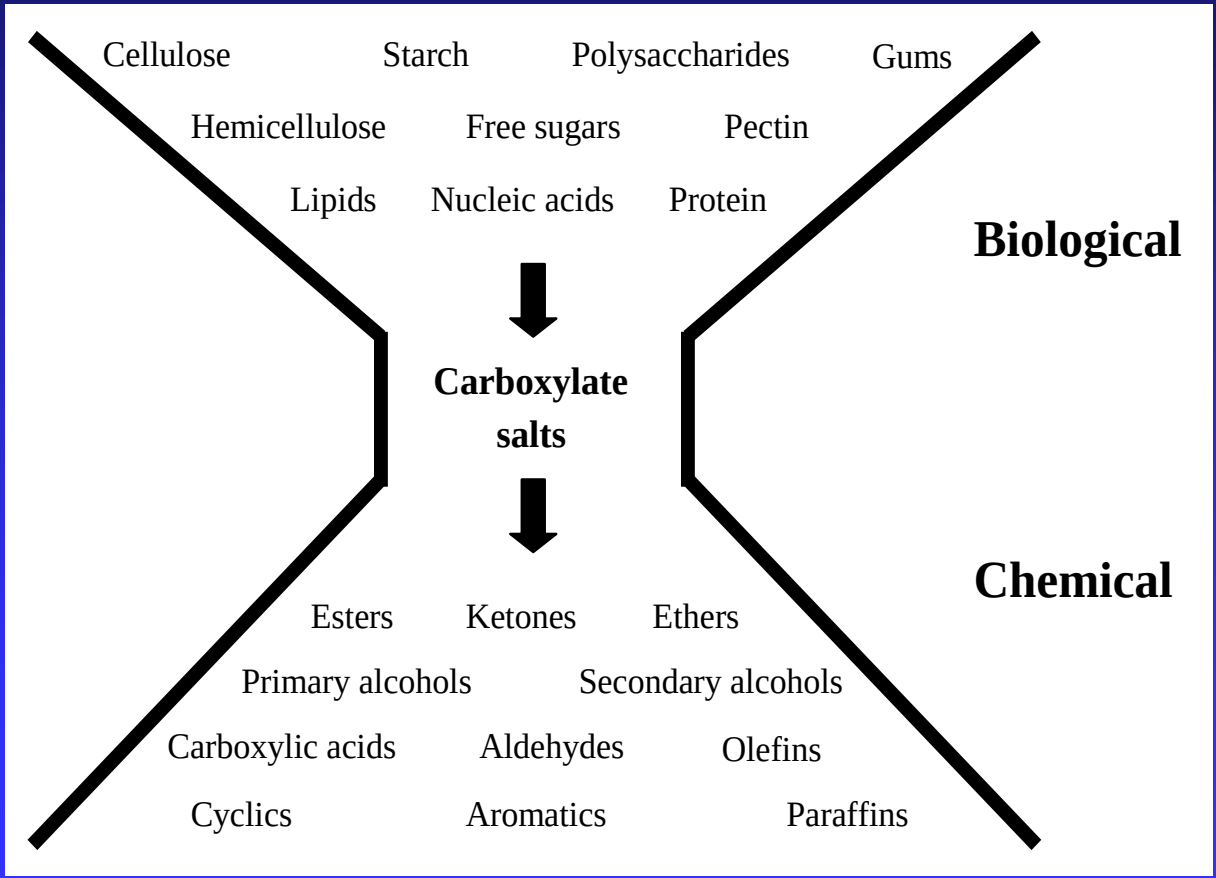
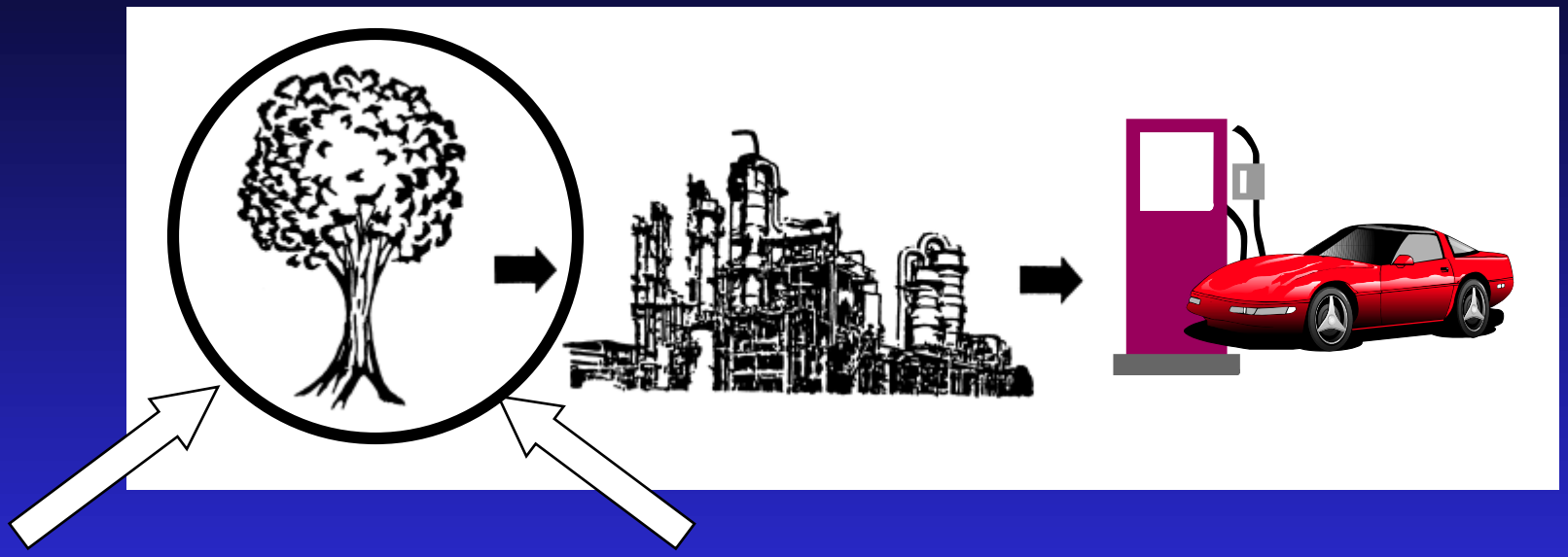
- Distributed processing

The Big Mouth



MixAlco Feedstocks



- trees
- grass
- agricultural residues
- energy crops

- animal manure
- sewage sludge
- municipal solid waste
- algae “bodies”

MixAlco Advantages

- Can use wet feedstocks
- Wide range of feedstocks
- Energy efficient
- No genetically modified organisms
- Many products
- Distributed processing

Dewatering Energetics

Ethanol Distillation (5% to 99.9%)

$$3\ \frac{\text{kg steam}}{\text{L ethanol}} = 8.4\ \frac{\text{MJ heat}}{\text{kg ethanol}} = \boxed{28.5\%}\ \text{of the combustion heat}$$

Source: B.L. Maiorella, Ethanol, *Comprehensive Biotechnology*, Vol. 3, Pergamon Press (1985).

MixAlco: Carboxylate Salt Vapor-Compression Dewatering (5% to 100%)

$$\frac{15.7\ \text{MJ heat}}{1000\ \text{kg water}} \times \frac{95\ \text{kg water}}{5\ \text{kg acid}} = \frac{0.3\ \text{MJ}}{\text{kg acid}} = \boxed{1.7\%}\ \text{of the combustion heat}$$

Work = 5.5 kWh/thous gal = 1.45 kWh/m^3

Assume heat to work is 33% efficient

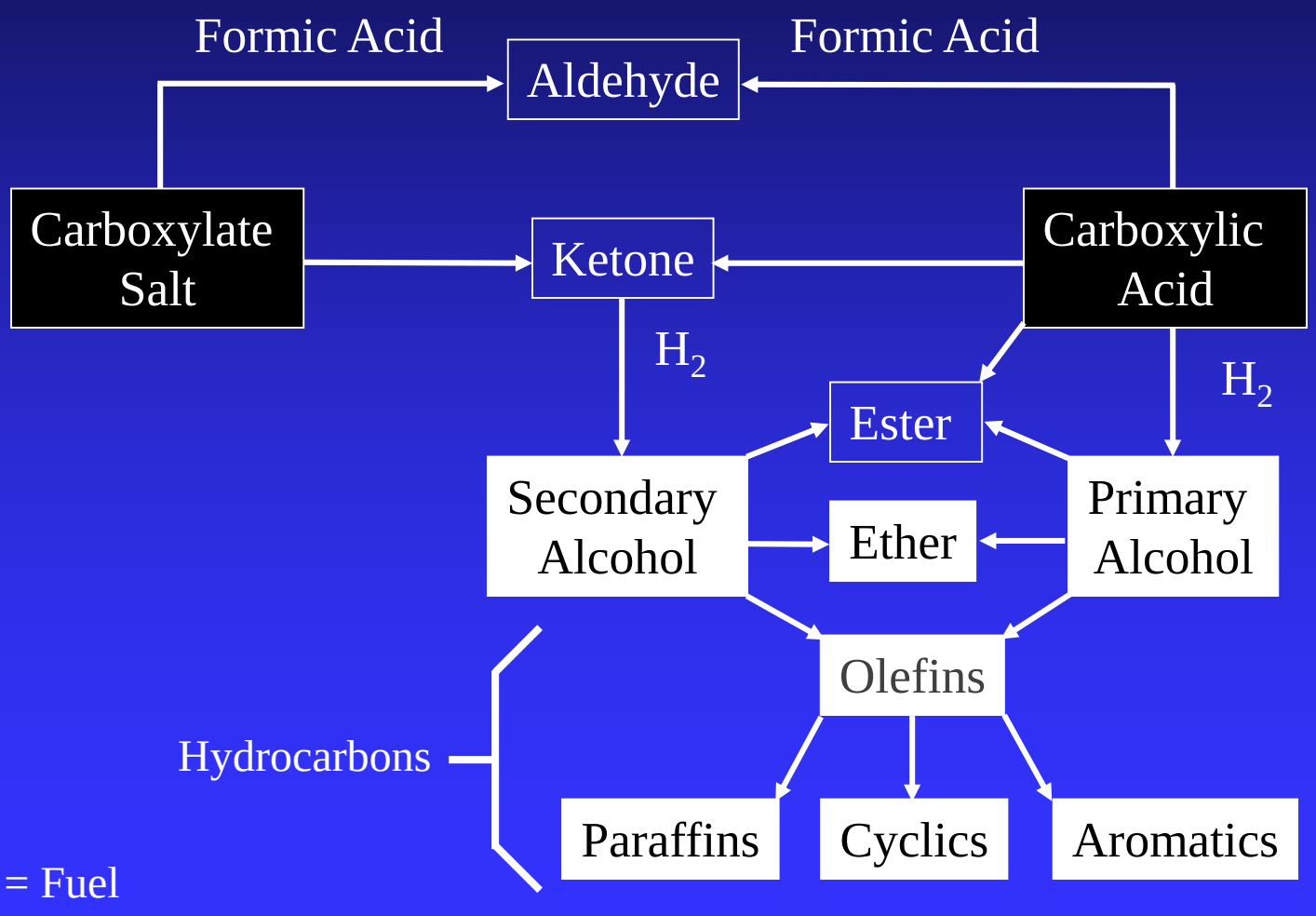
MixAlco Advantages

- Can use wet feedstocks
- Wide range of feedstocks
- Energy efficient
- No genetically modified organisms
- Many products
- Distributed processing

MixAlco Advantages

- Can use wet feedstocks
- Wide range of feedstocks
- Energy efficient
- No genetically modified organisms
- Many products
- Distributed processing

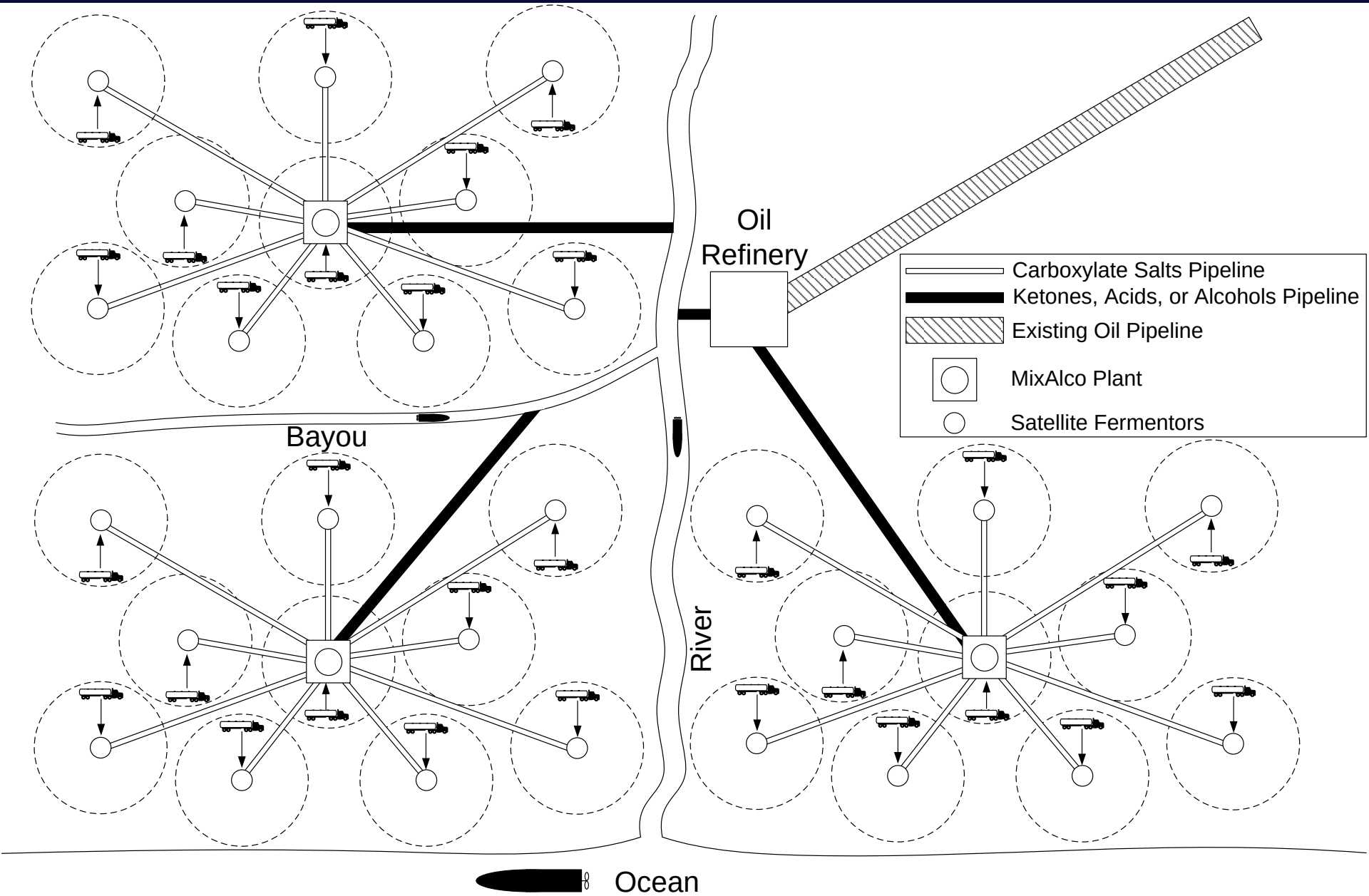
Chemistry


Formic Acid
Aldehyde
Formic Acid
Carboxylate Salt
Ketone
Carboxylic Acid
H_2
H_2
Ester
Secondary Alcohol
Ether
Primary Alcohol
Olefins
Hydrocarbons
Paraffins
Cyclics
Aromatics
= Fuel

MixAlco Advantages

- Can use wet feedstocks
- Wide range of feedstocks
- Energy efficient
- No genetically modified organisms
- Many products
- Distributed processing

MixAlco Logistics (Example)



MixAlco Advantages

No Enzyme Addition

MixAlco Advantages

No

Sterility

MixAlco Advantages

Mixed Culture

No

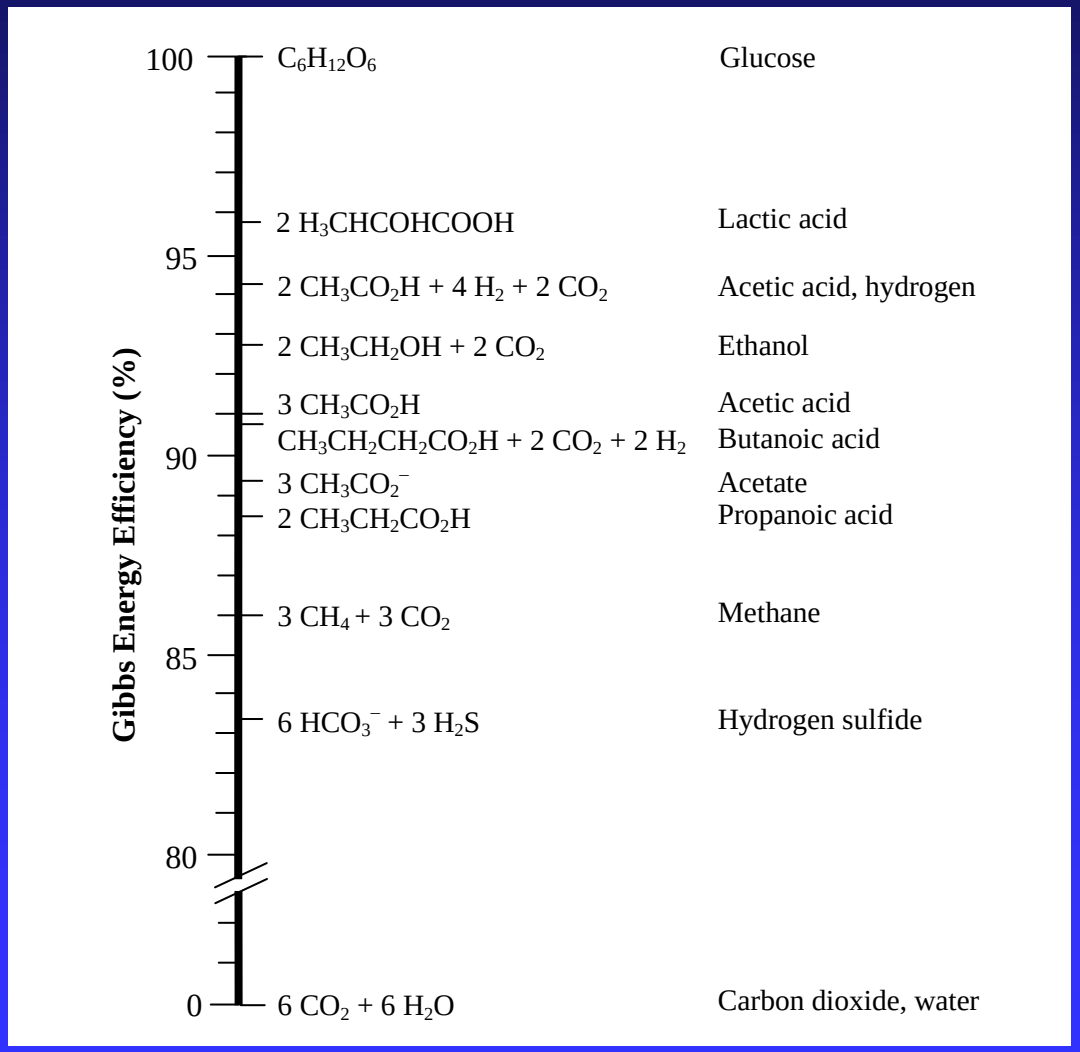
Sterility

Environments where organic acids naturally form

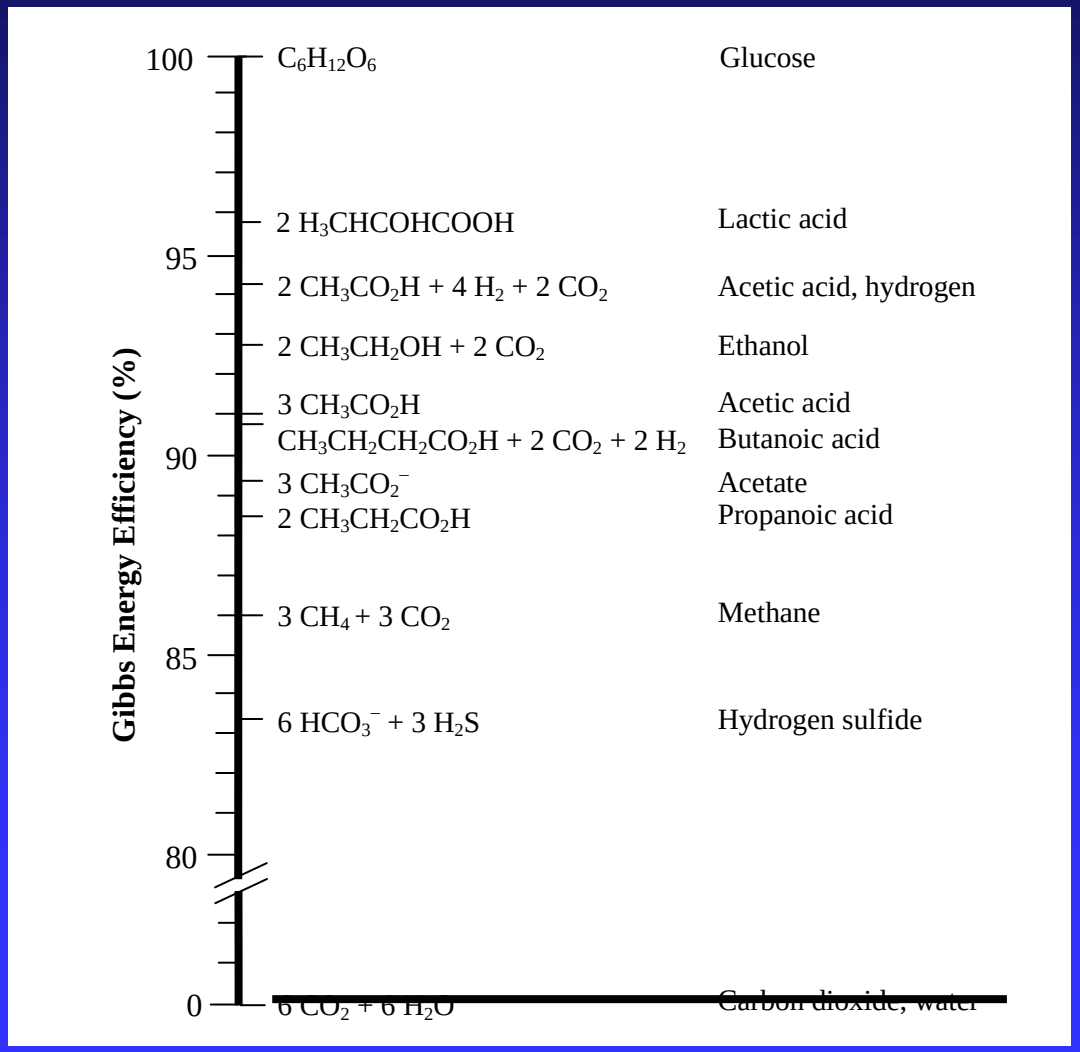
- animal rumen
  - cattle
  - sheep
  - deer
- anaerobic sewage digestors
- swamps
- termite guts

Why are organic acids favored?

Why are organic acids favored?

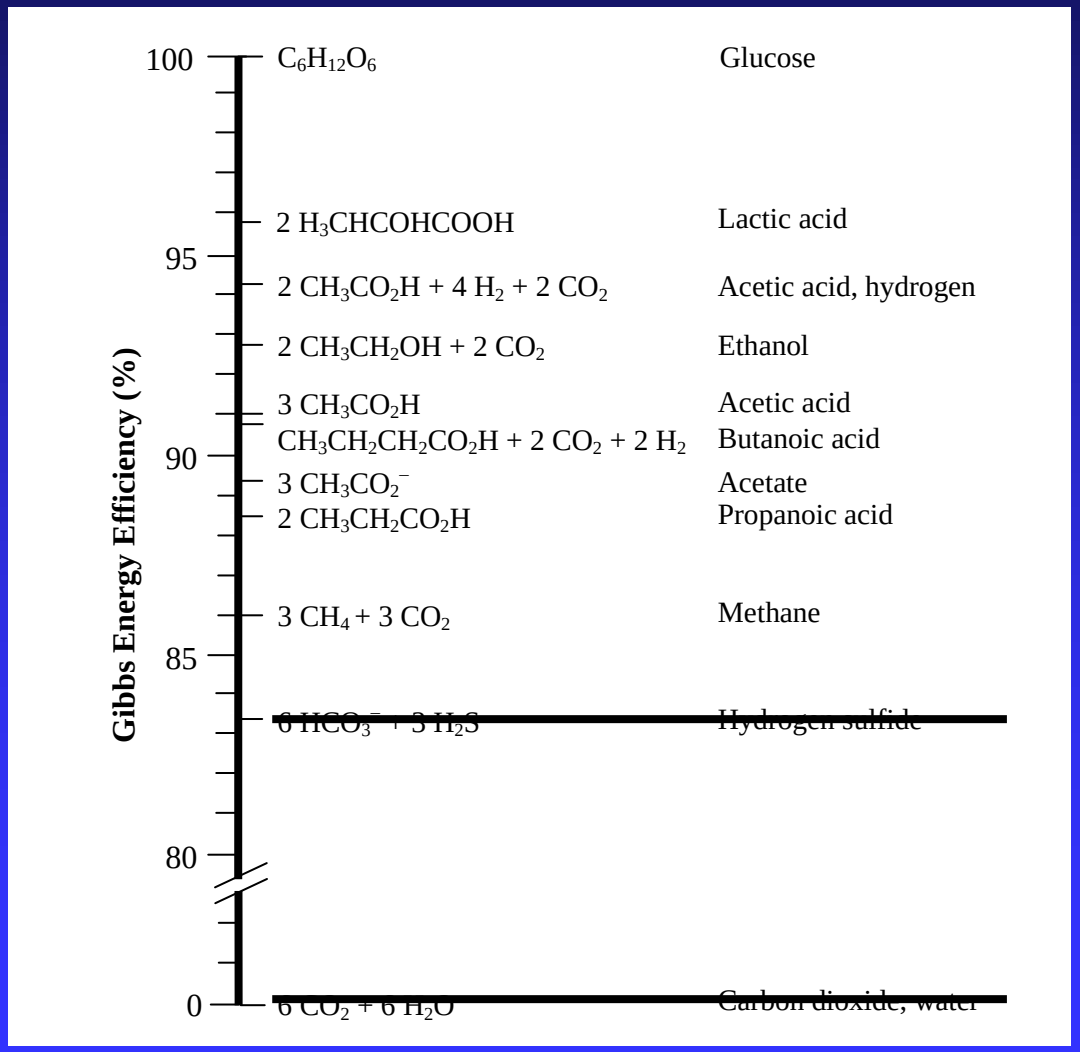


Why are organic acids favored?



Do not add oxygen

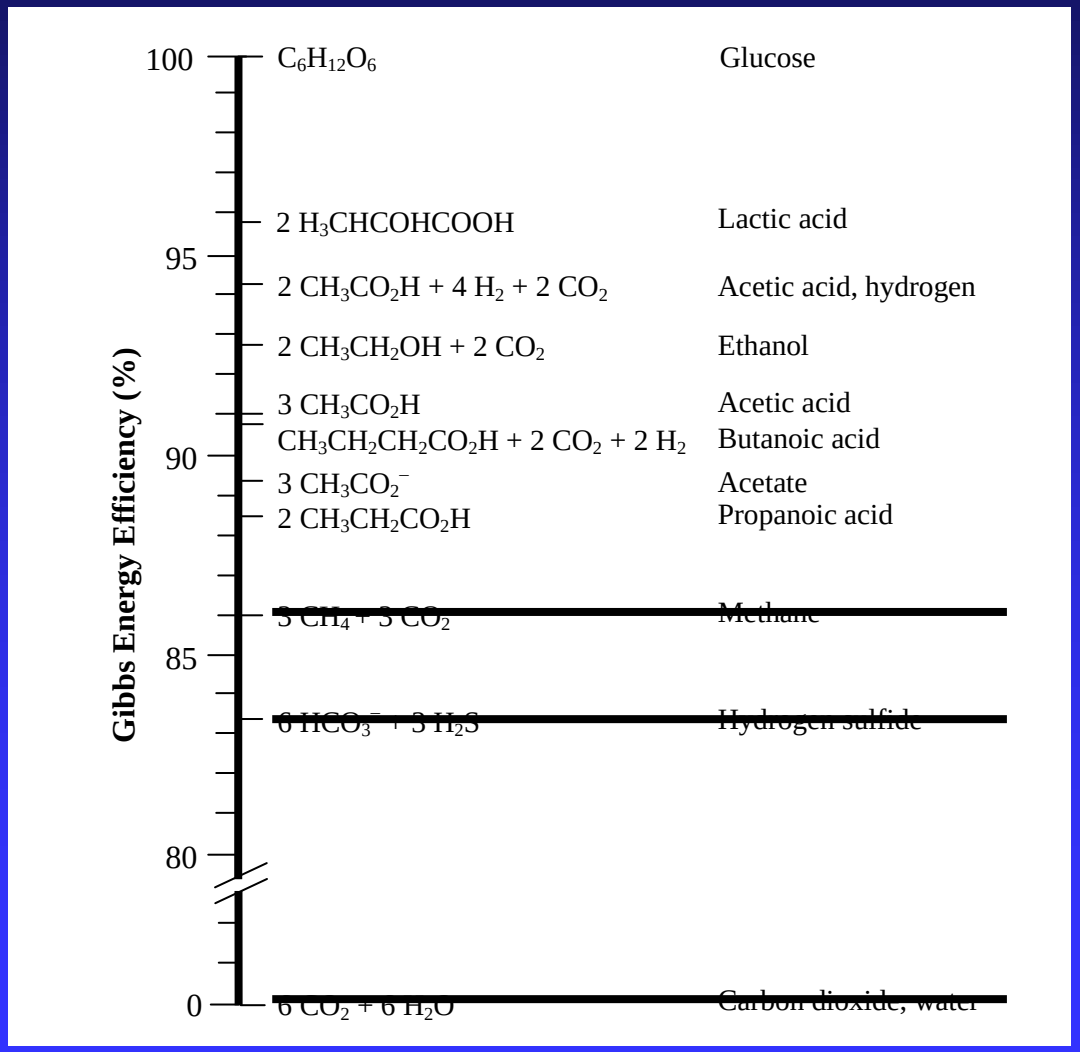
Why are organic acids favored?



Do not add sulfate

Do not add oxygen

Why are organic acids favored?

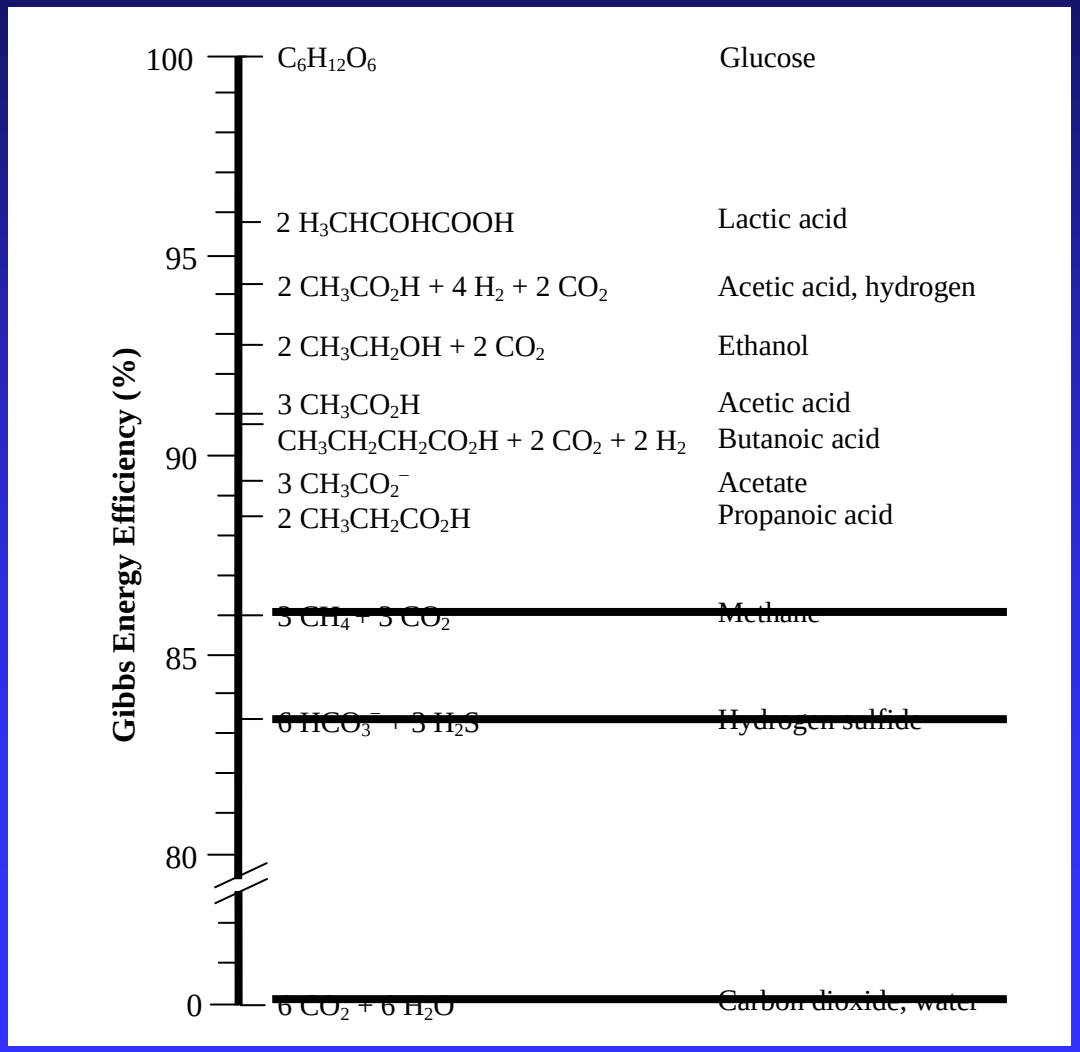


Add methanogen inhibitors

Do not add sulfate

Do not add oxygen

Why are organic acids favored?



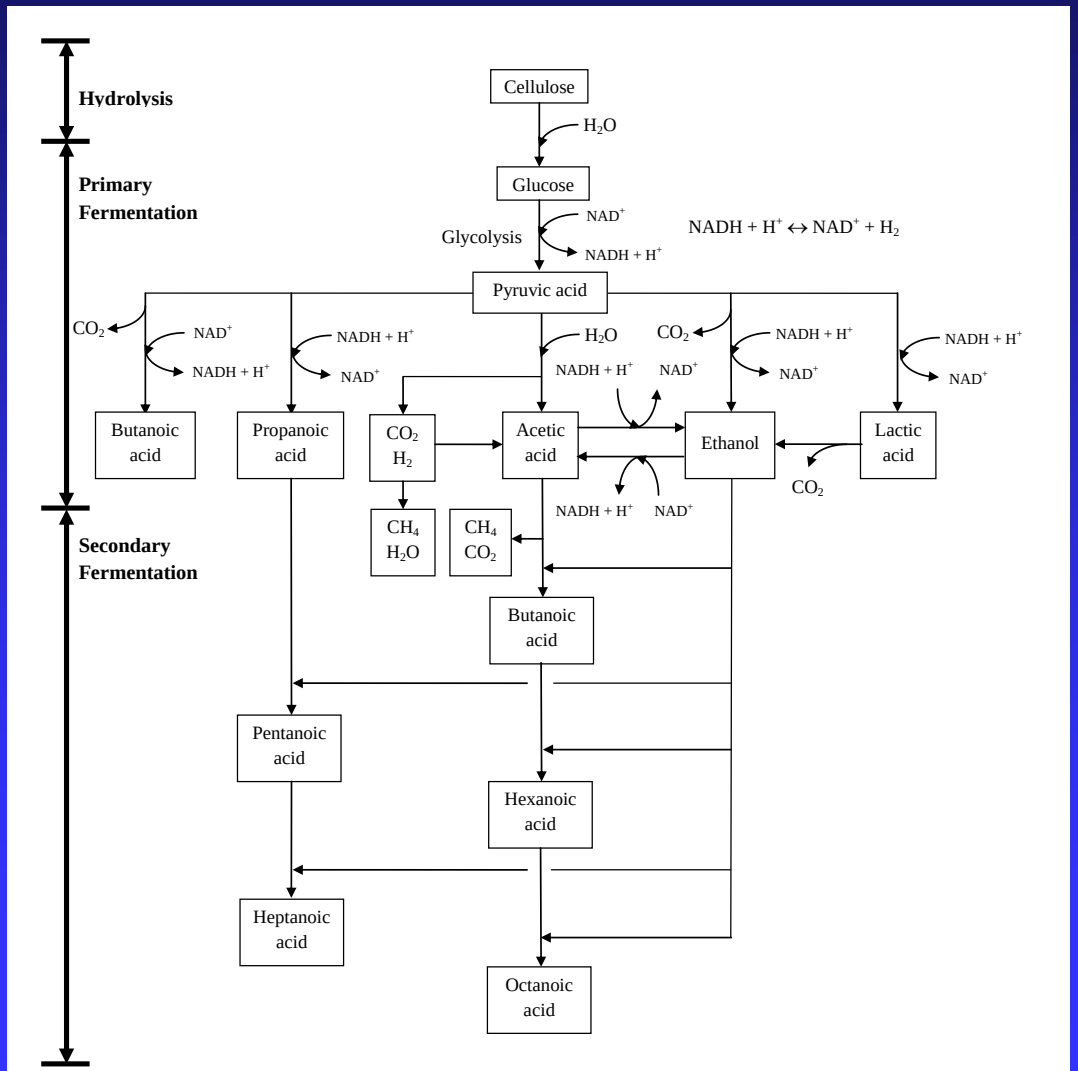
} Low energy state

Add methanogen inhibitors

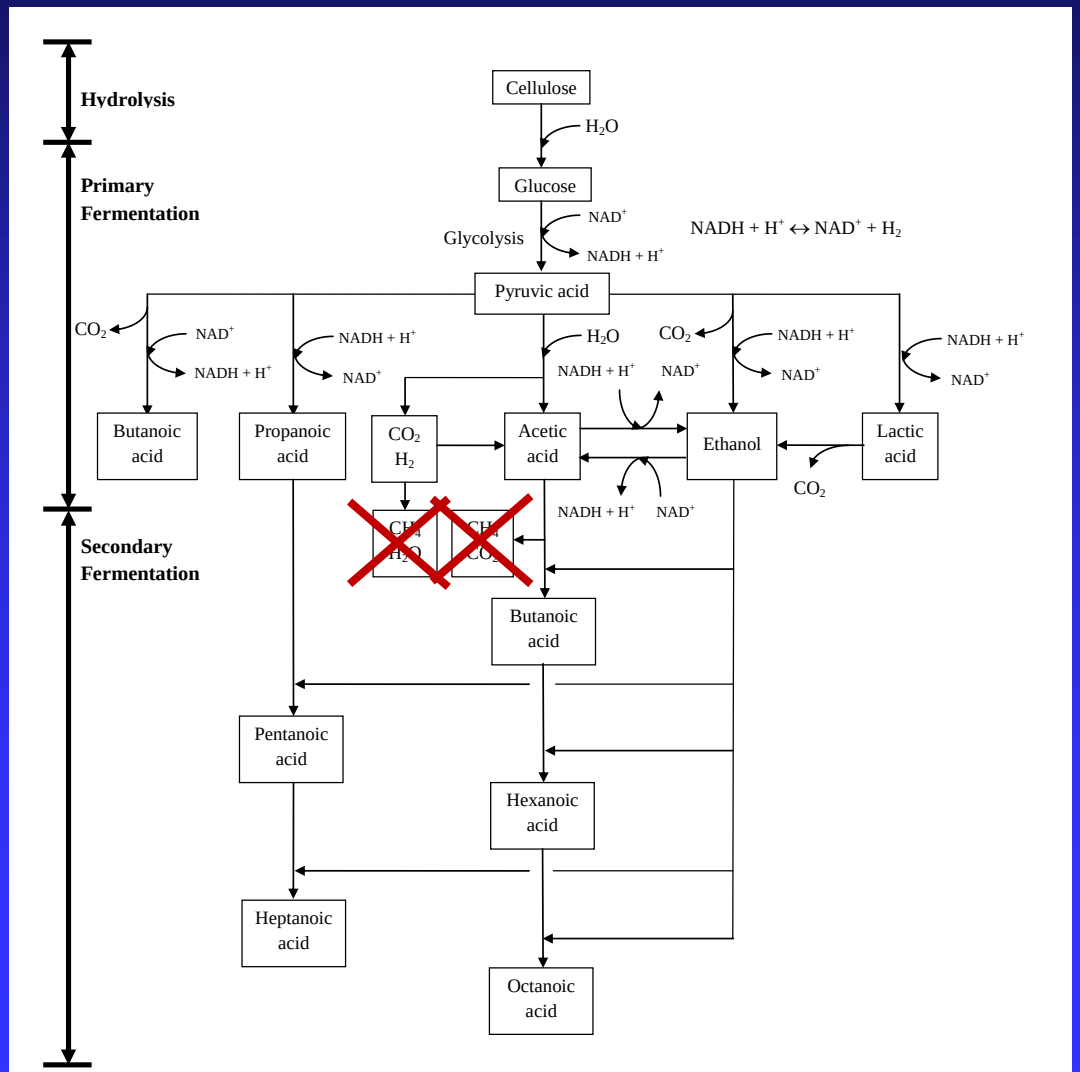
Do not add sulfate

Do not add oxygen

Metabolic Pathways

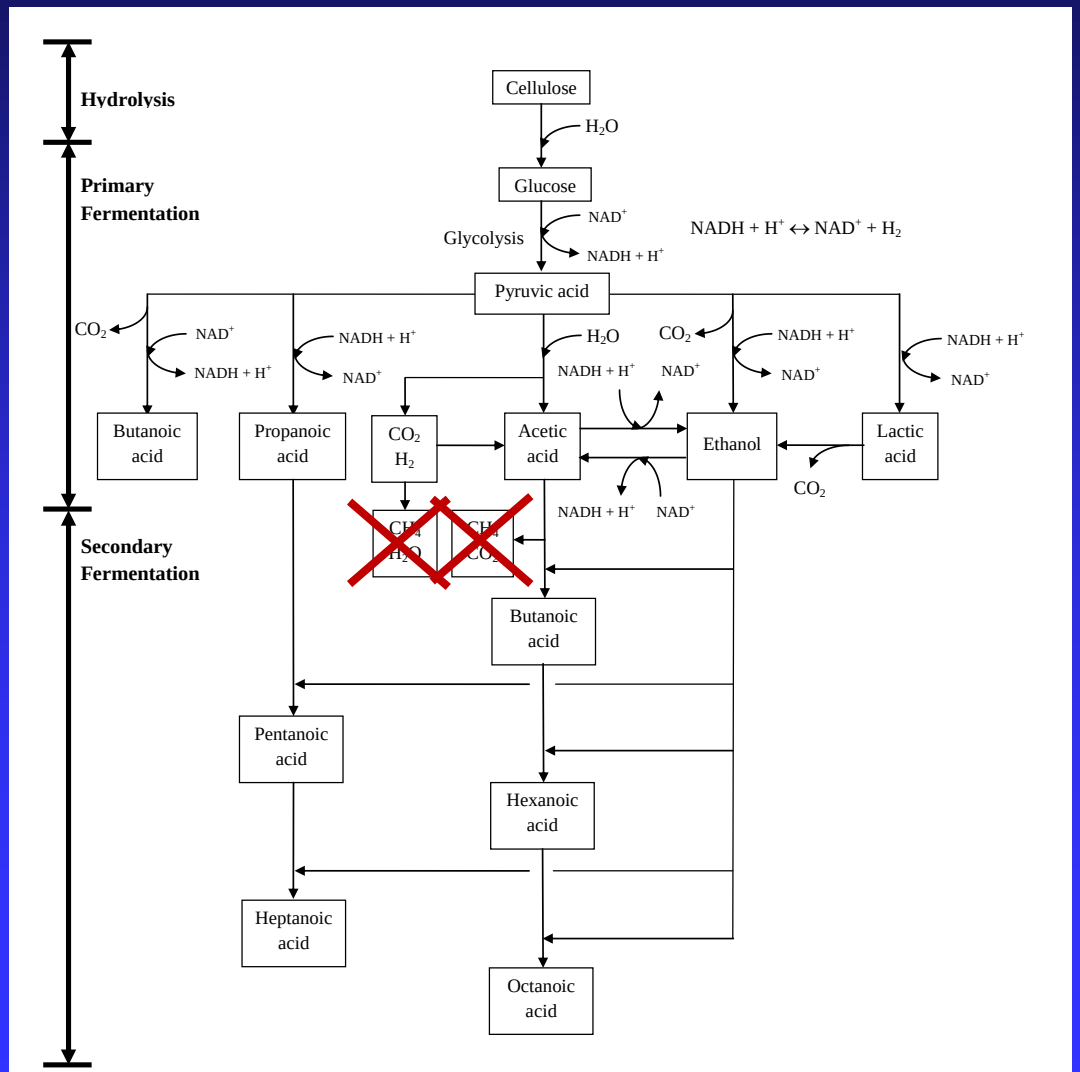


Metabolic Pathways



Iodoform

Metabolic Pathways



Chain Elongation

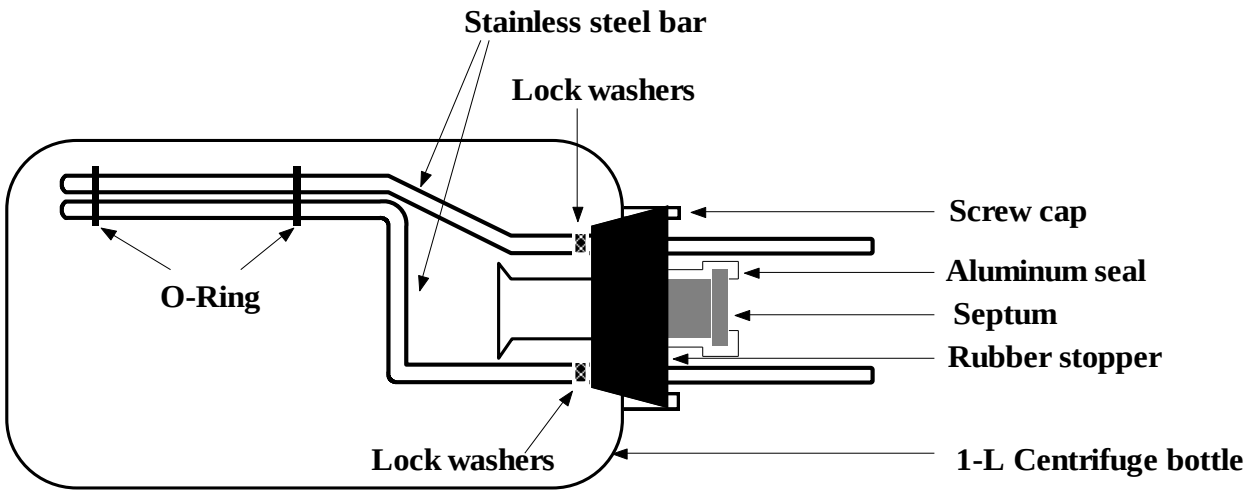
Typical Product Spectrum at Different Culture Temperatures

	40°C	55°C
C2 – Acetic	41 wt %	80 wt %
C3 – Propionic	15 wt %	4 wt %
C4 – Butyric	21 wt %	15 wt %
C5 – Valeric	8 wt %	<1 wt %
C6 – Caproic	12 wt %	<1 wt %
C7 – Heptanoic	3 wt %	<1 wt %
	100 wt %	100 wt %

Fermentors

- **Submerged**
- Pile

Laboratory Fermentors



Design of Rotary Fermentors

Laboratory Incubator

Fermentation Stability

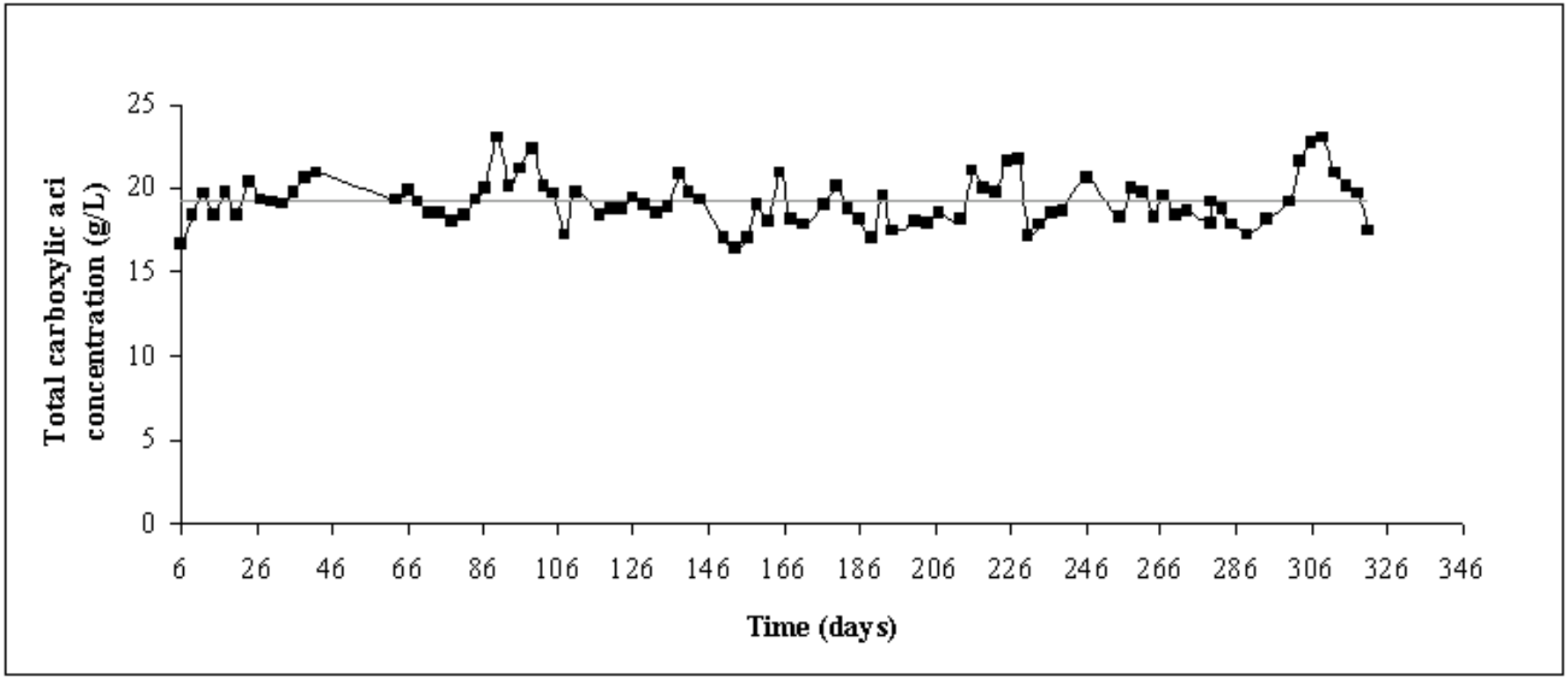
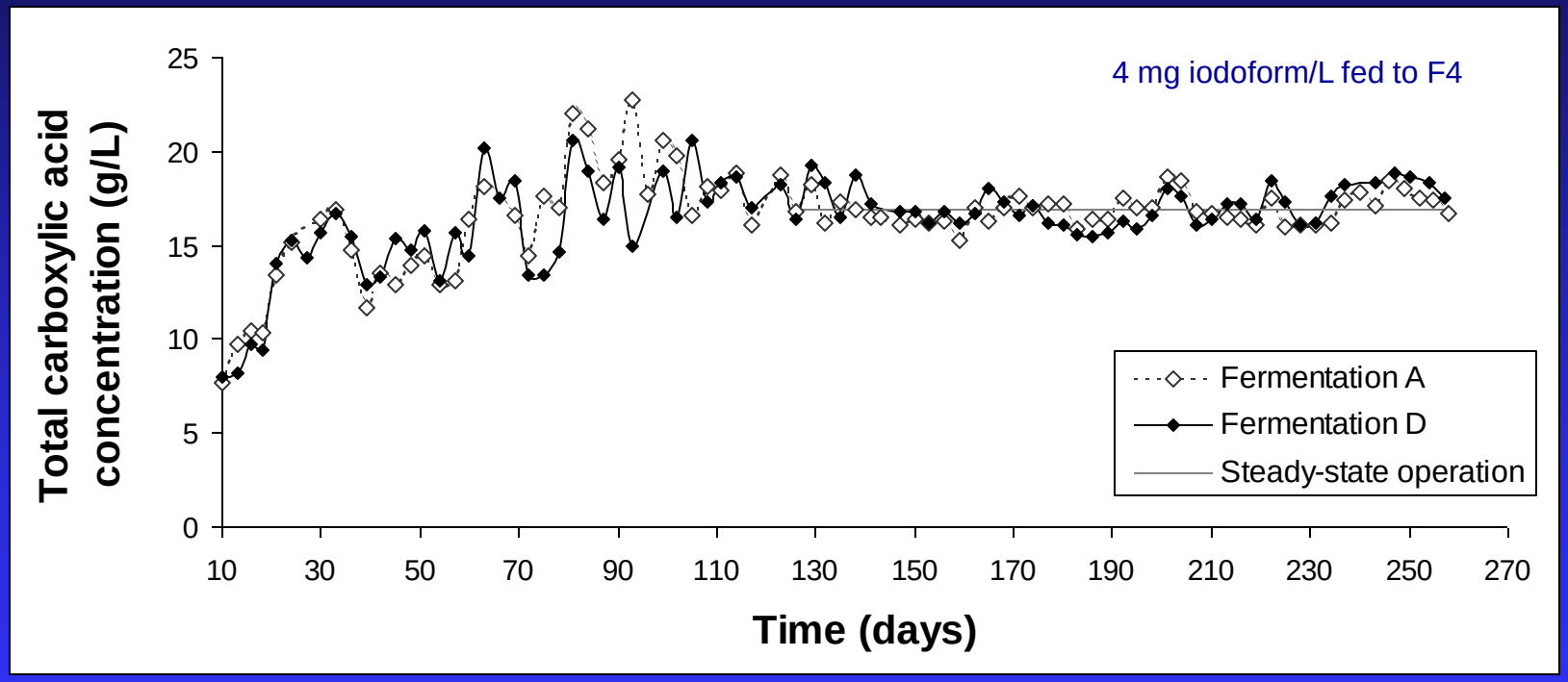


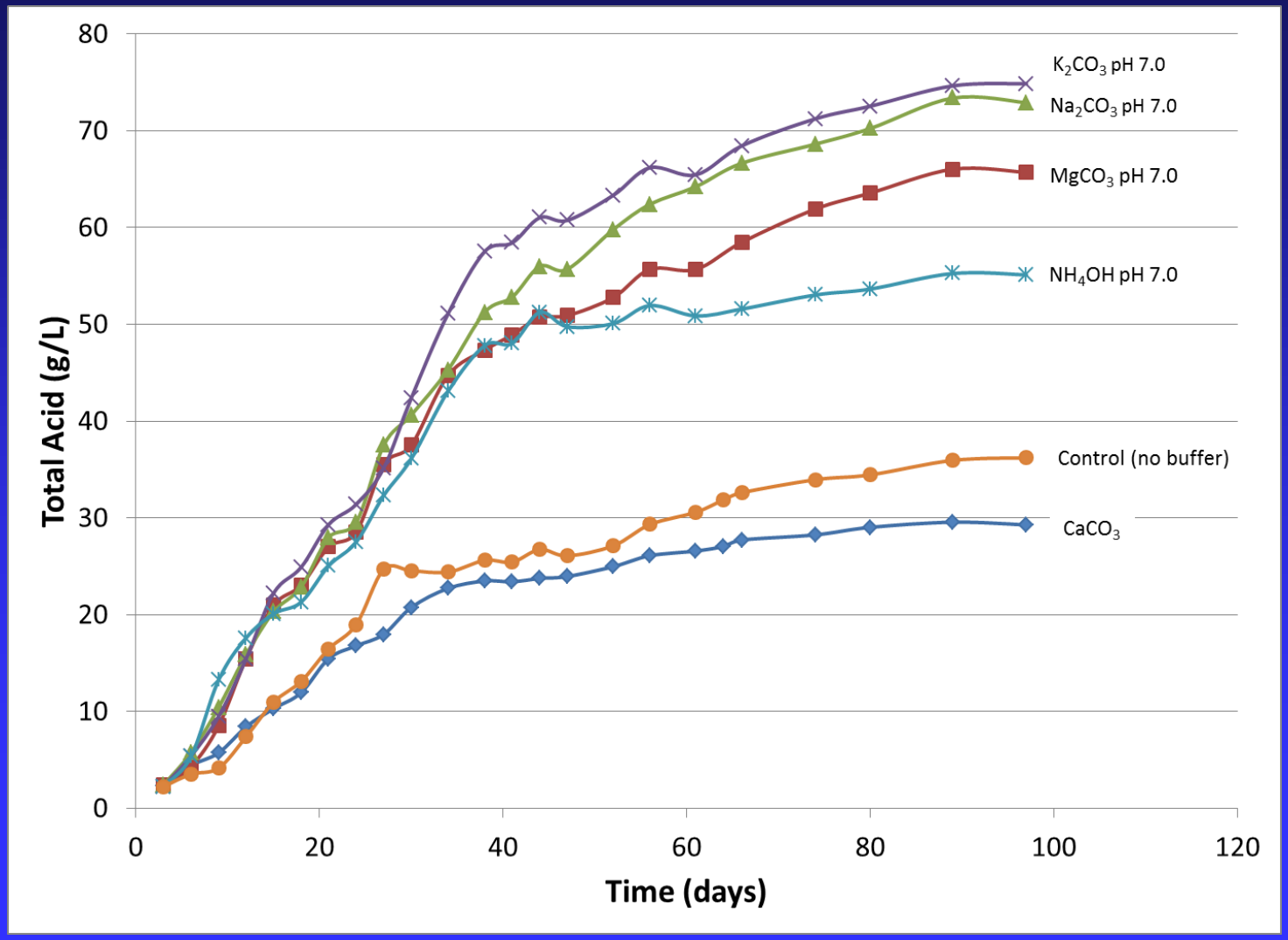
Figure VII-1: Total carboxylic acid concentration for 80% paper/20%biosludge fermentation (Terrestrial inocula, LRT= 24 days and VSLR=1.9 g/(L·d)).

Reproducibility



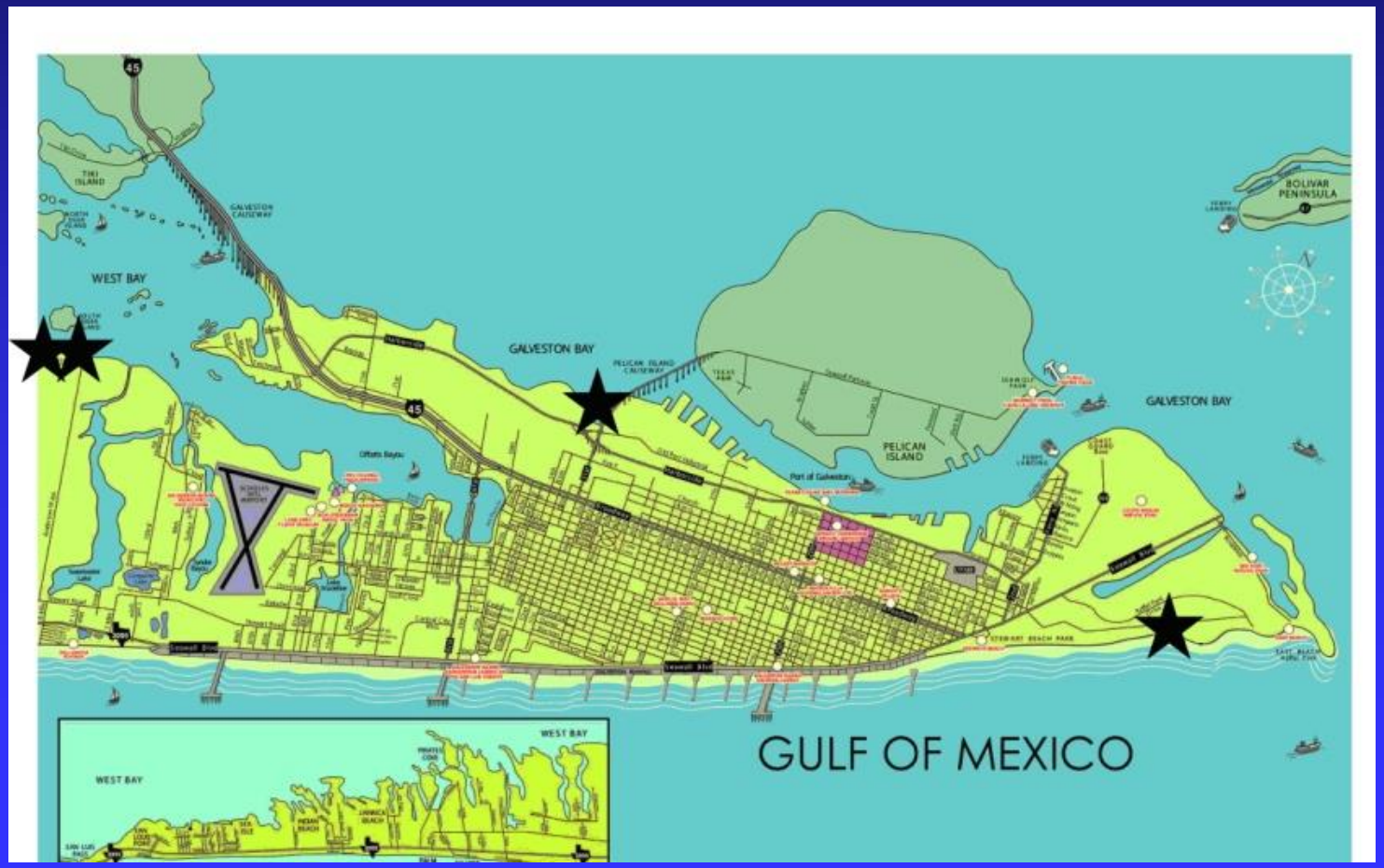
Terrestrial inocula
LRT = 20.7 days
VSLR = 3.1 g/(L·d))

Buffers


80
70
60
50
40
30
20
10
0
Total Acid (g/L)
0
20
40
60
80
100
120
Time (days)
K_2CO_3 pH 7.0
Na_2CO_3 pH 7.0
$MgCO_3$ pH 7.0
NH_4OH pH 7.0
Control (no buffer)
$CaCO_3$

Marine Inoculum Source

Galveston Island, TX



16s Ribosomal Typing of Galveston Marine Culture

Microorganisms
(Ammonium bicarbonate buffer, 55°C)

Biosafety	Microorganism	Percent of Culture
1	*Thermobrachium celere*	67
1	*Clostridium xylanovorans*	13
1	*Bacillus thermocloaceae*	7
1	*Parasporobacterium paucivorans*	7
1	*Caloramator fervidus*	7

1 – Safe
5 – Hazardous

Microorganism Identification:
Terry Gentry, Soil and Crop Sciences, Texas A&M University

Microorganisms
(Calcium carbonate buffer, 55°C)

Biosafety	Microorganism	Percent of Culture
1	*Clostridium stercorarium subsp. leptospartum*	14
1	*Clostridium thermocellum*	13
1	*Ureibacillus suwonensis*	11
1	*Coprothermobacter proteolyticus*	10
1	*Clostridium jejuense*	6

1 – Safe
5 – Hazardous

Microorganism Identification:
Terry Gentry, Soil and Crop Sciences, Texas A&M University

Microorganisms
(Calcium carbonate buffer, 55°C)

Biosafety	Microorganism	Percent of Culture
1	*Clostridium stercorarium subsp. leptospartum*	6
1	*Clostridium orbiscindens*	3
1	*Clostridium sporosphaeroides*	3
1	*Clostridium stercorarium subsp. leptospartum*	3
1	*Clostridium ultunense*	3

1 – Safe
5 – Hazardous

Microorganism Identification:
Terry Gentry, Soil and Crop Sciences, Texas A&M University

Microorganisms
(Calcium carbonate buffer, 55°C)

Biosafety	Microorganism	Percent of Culture
1	*Clostridium straminisolvens*	2
1	*Clostridium xylanovorans*	2
1	*Desulfotomaculum carboxydivorans*	2
1	*Parasporobacterium paucivorans*	2
1	*Carboxydothermus hydrogenoformans*	1

1 – Safe
5 – Hazardous

Microorganism Identification:
Terry Gentry, Soil and Crop Sciences, Texas A&M University

Microorganisms
(Calcium carbonate buffer, 55°C)

Biosafety	Microorganism	Percent of Culture
1	*Clostridium alkalicellum*	1
1	*Clostridium cellulosi*	1
1	*Clostridium jejuense*	1
1	*Clostridium sp. JC3*	1
1	*Clostridium stercorarium subsp. leptospartum*	1

1 – Safe
5 – Hazardous

Microorganism Identification:
Terry Gentry, Soil and Crop Sciences, Texas A&M University

Microorganisms
(Calcium carbonate buffer, 55°C)

Biosafety	Microorganism	Percent of Culture
1	*Clostridium thermocellum*	1
1	*Clostridium thermosuccinogenes*	1
1	*Clostridium xylanovorans*	1
1	*Desulfitobacterium dichloroeliminans*	1
1	*Gracilibacter thermotolerans*	1

1 – Safe
5 – Hazardous

Microorganism Identification:
Terry Gentry, Soil and Crop Sciences, Texas A&M University

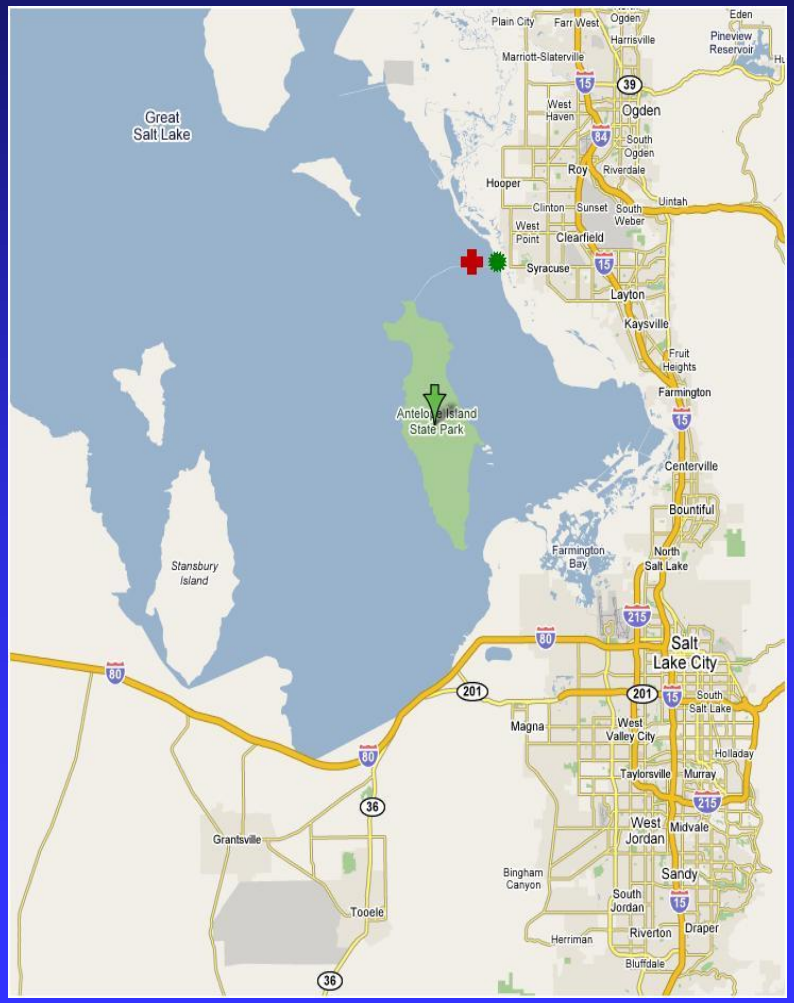
Microorganisms
(Calcium carbonate buffer, 55°C)

Biosafety	Microorganism	Percent of Culture
1	*Mahella australiensis*	1
1	*Thermoanaerobacterium aotearoense*	1
1	*thermophilic anaerobic bacterium K1L1*	1

1 – Safe
5 – Hazardous

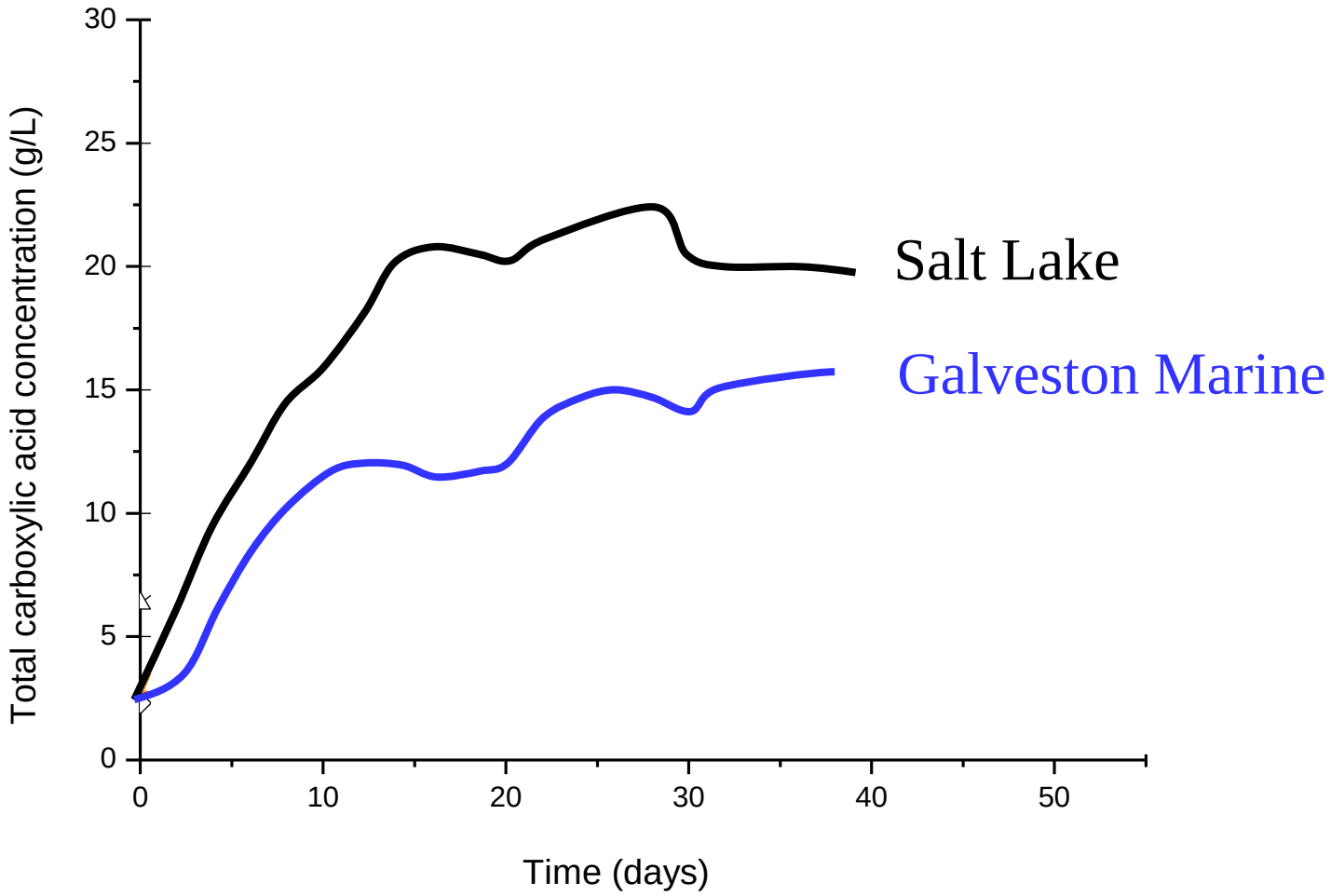
Microorganism Identification:
Terry Gentry, Soil and Crop Sciences, Texas A&M University

Great Salt Lake Inoculum Source



Great Salt Lake, UT

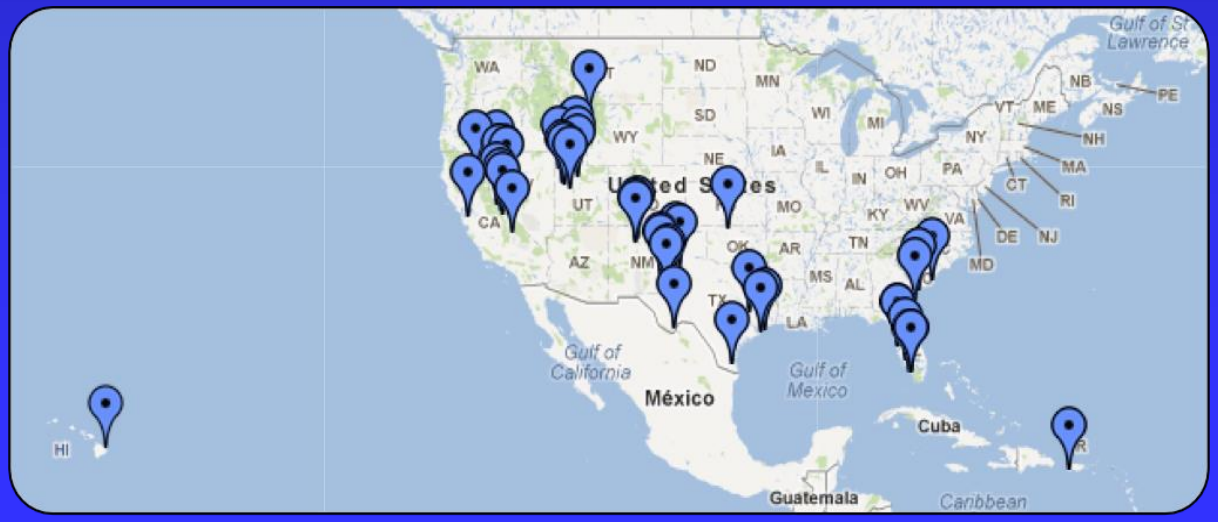
Marine vs Salt Lake



Bioprospecting

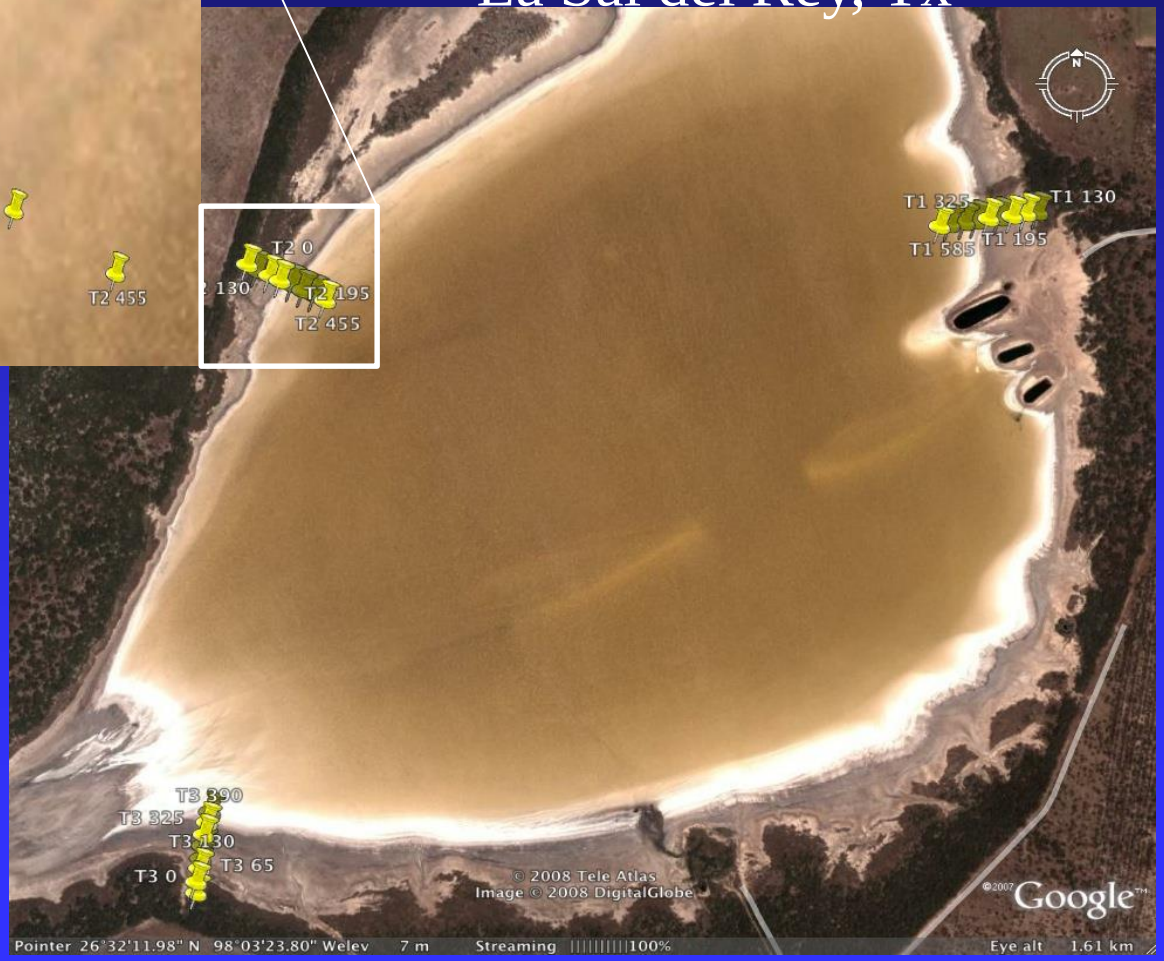
Sites of Interest

- High salt content
- Thermal lakes/springs



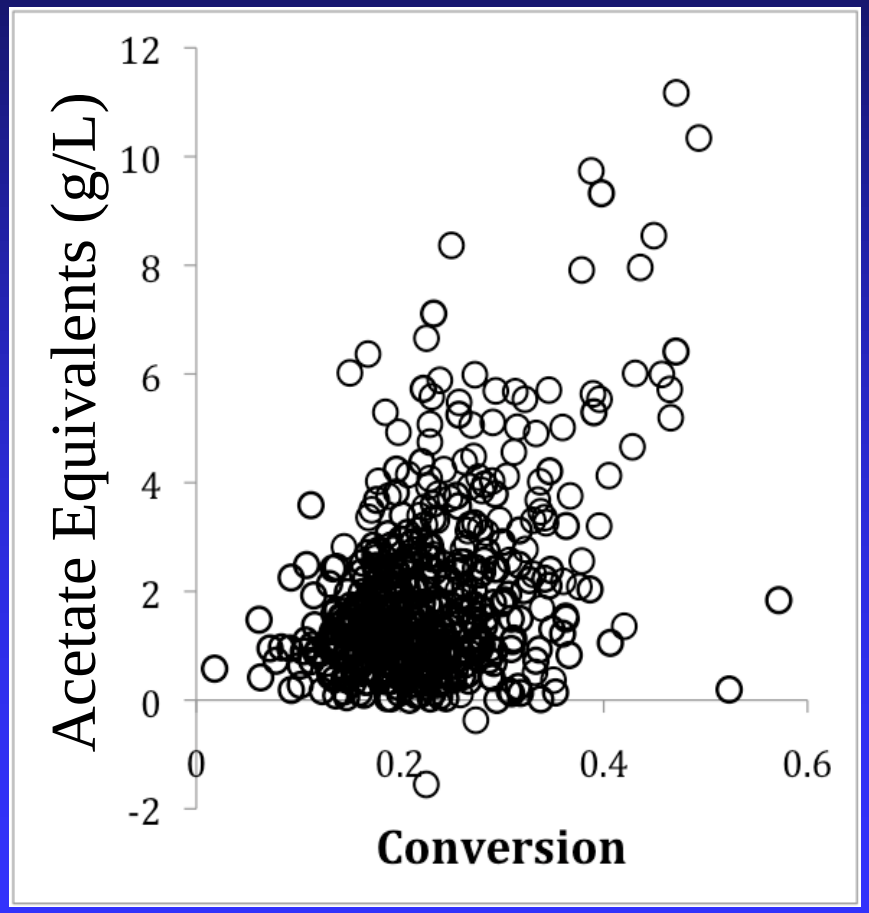
Site Sampling

La Sal del Rey, Tx

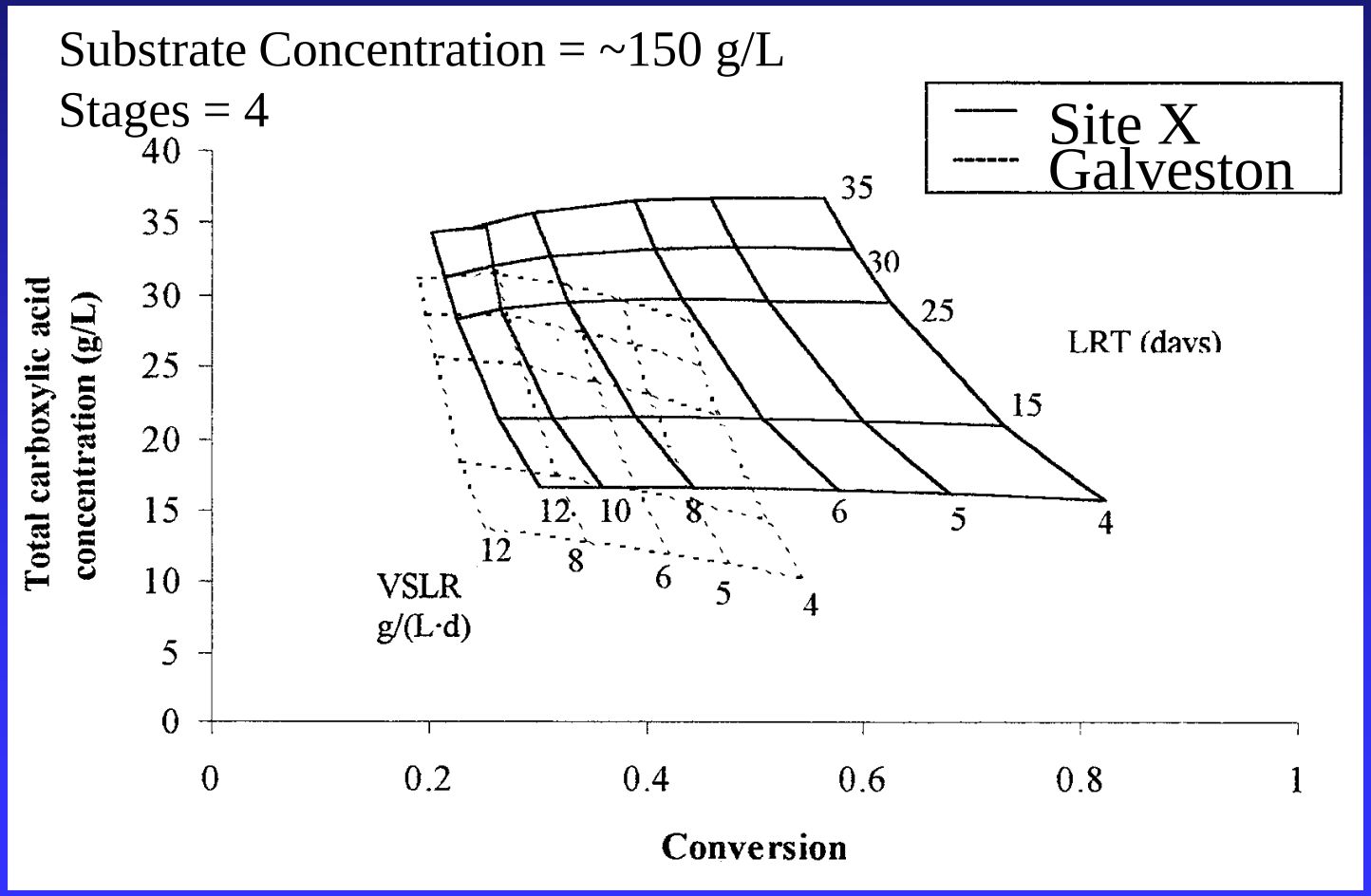


Bioprospecting Results

Heather Wilkinson
Plant Pathology
Texas A&M



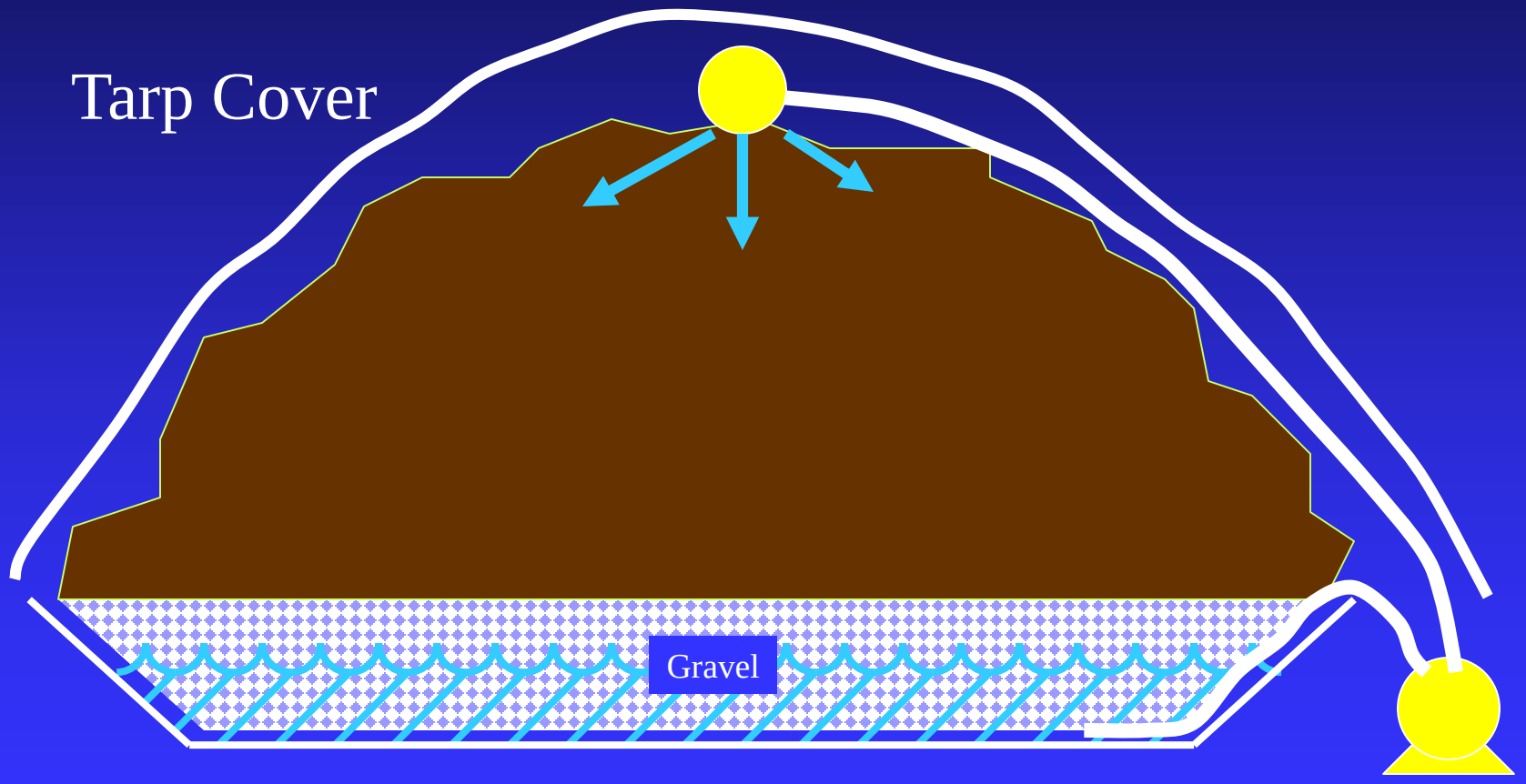
Compare CPDM maps



Fermentors

- Submerged
- **Pile**

Tarp-Covered Pile Fermentor

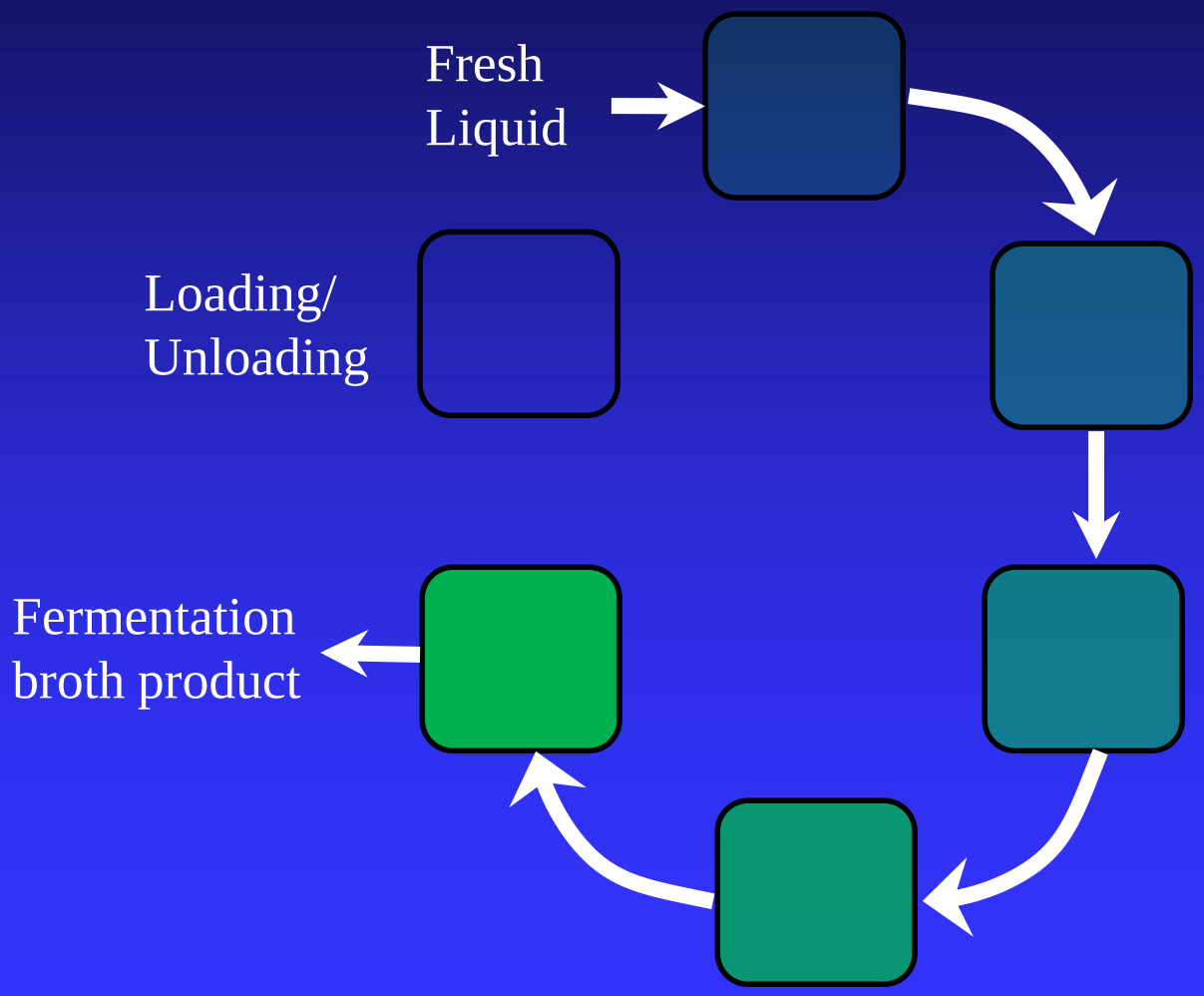


Round Robin System

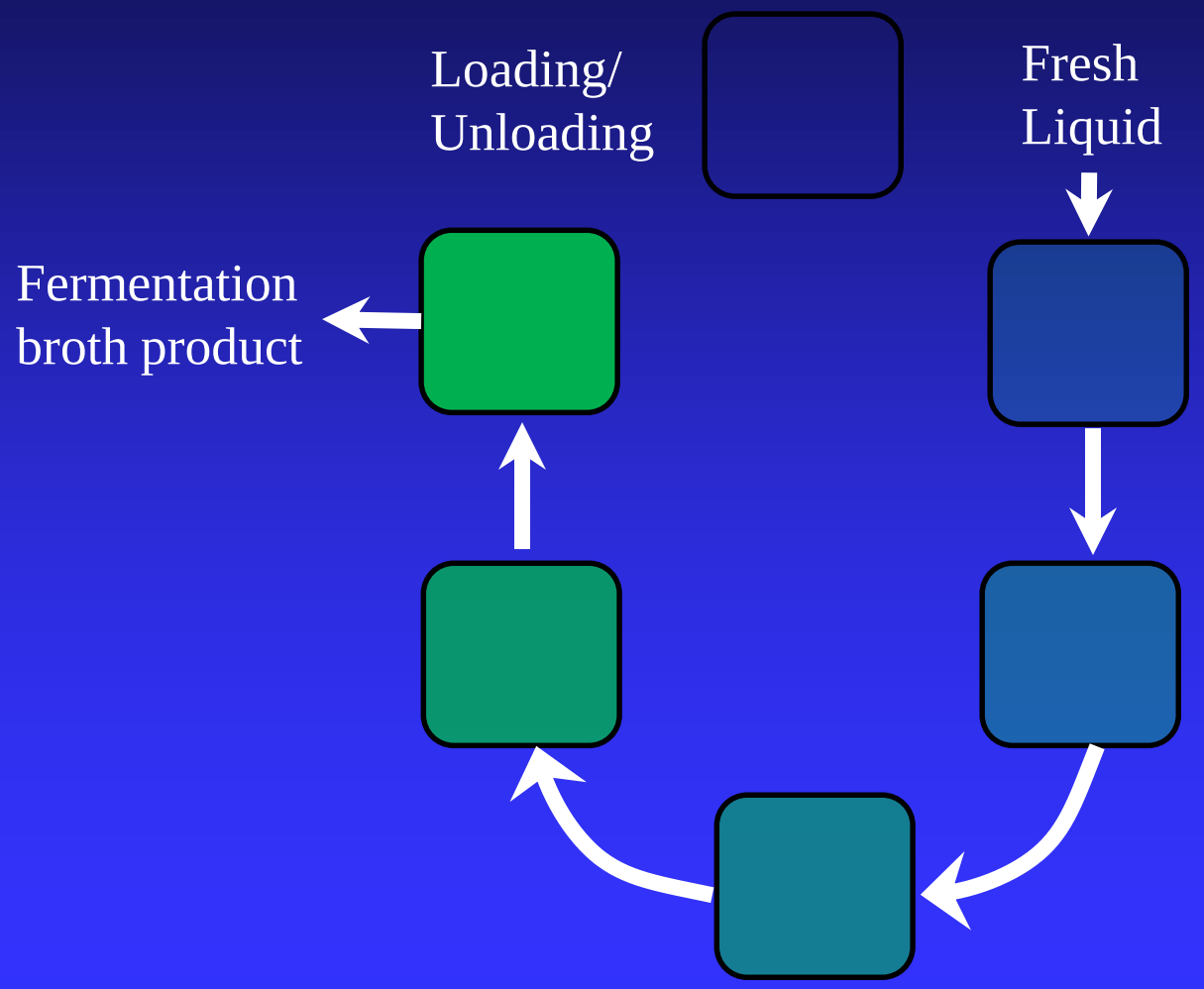
Advantages:

- Minimal solids handling
- Low capital
- Complete conversion
- High product concentration

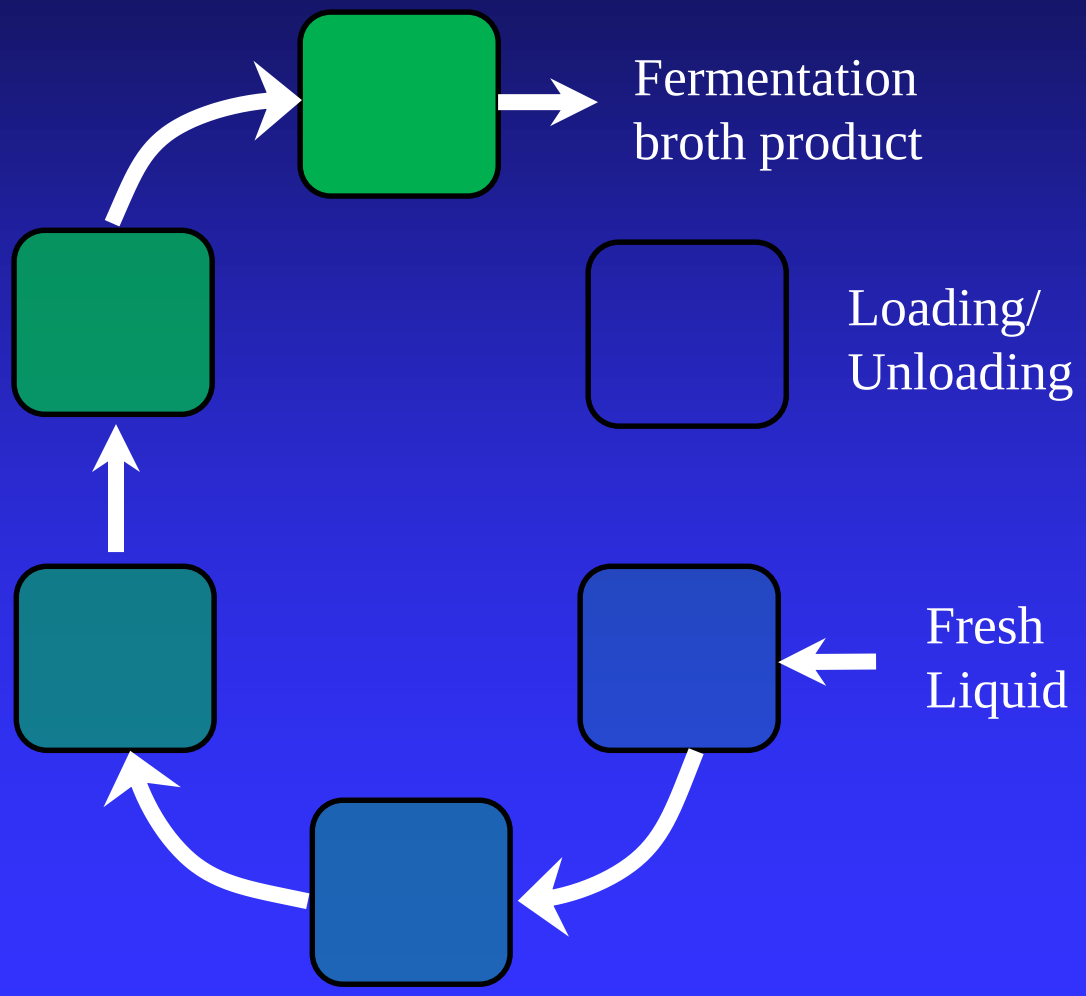
Round Robin System



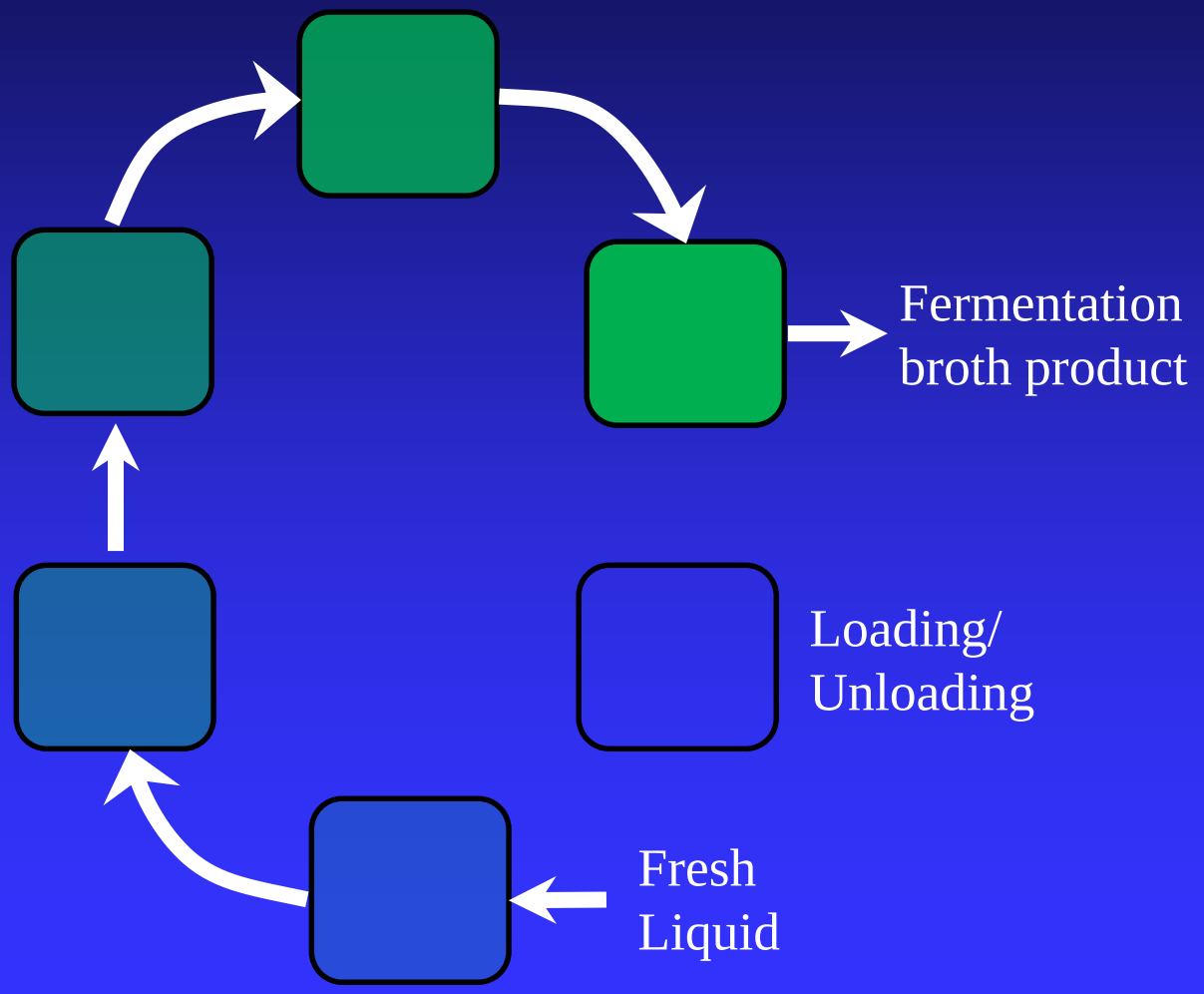
Round Robin System



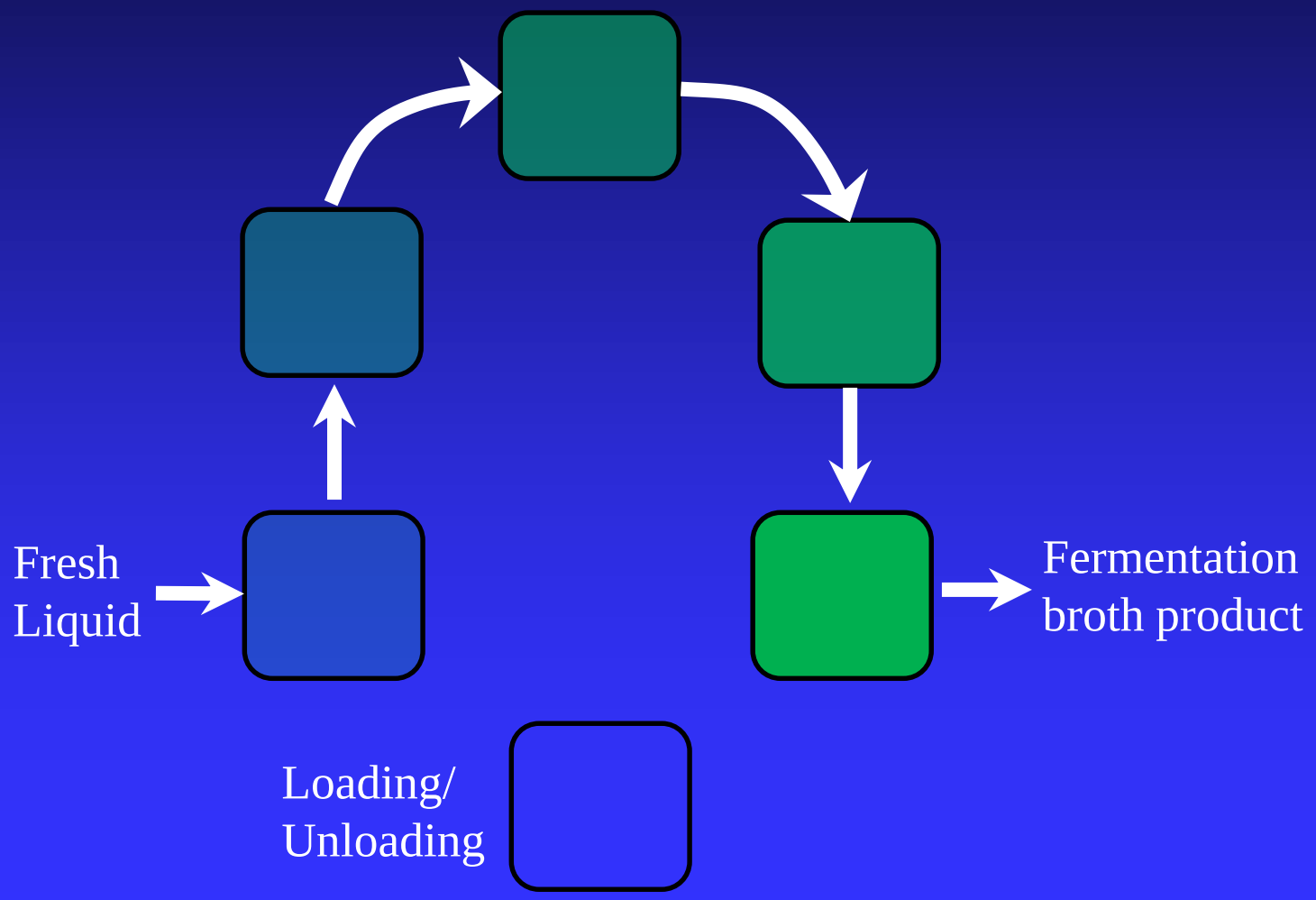
Round Robin System



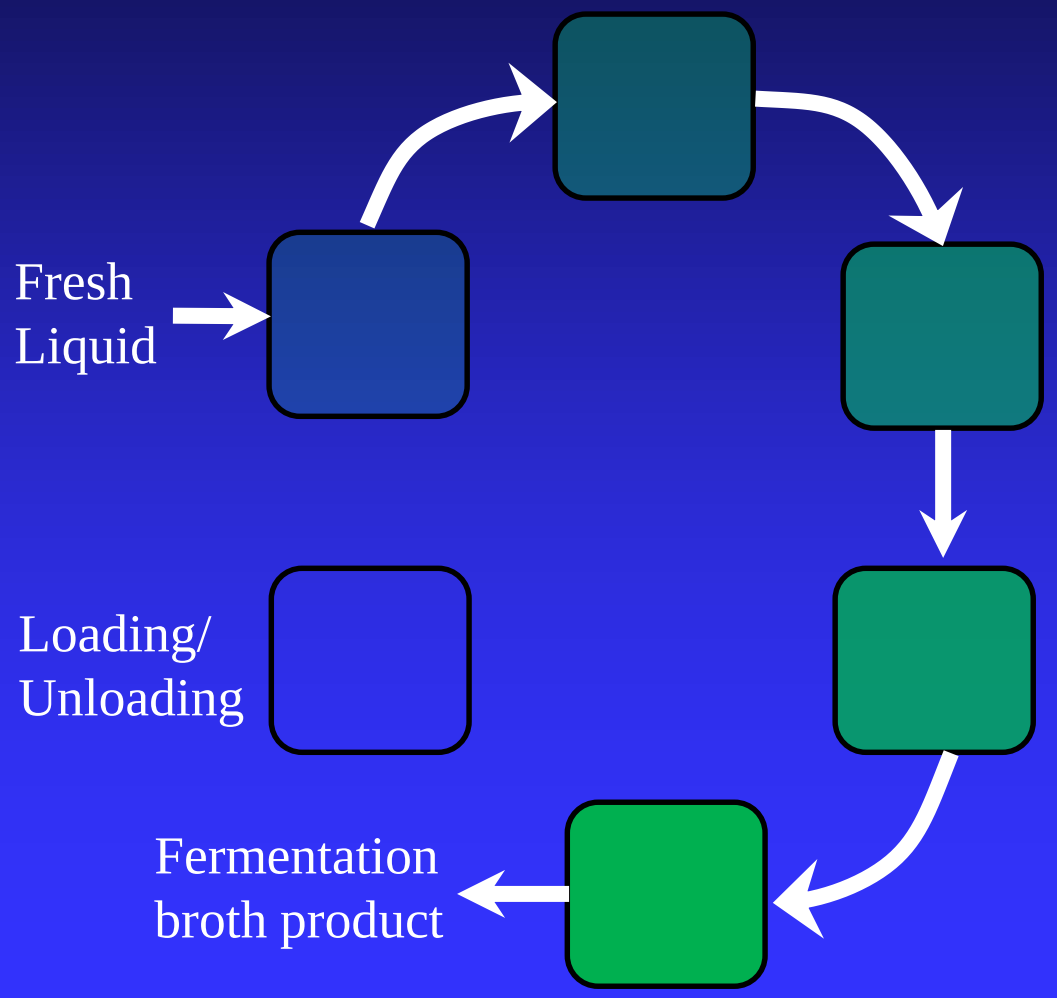
Round Robin System



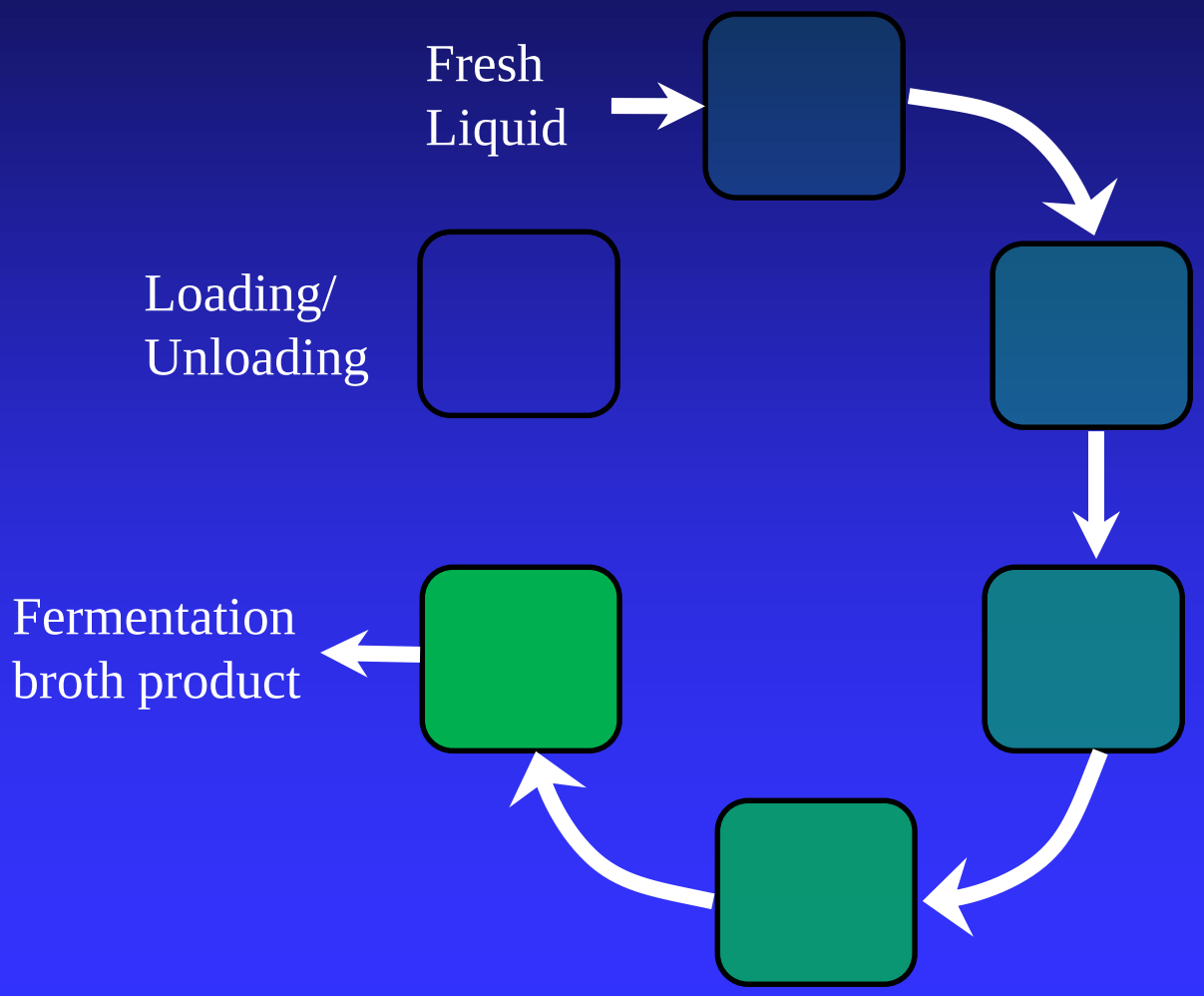
Round Robin System



Round Robin System



Round Robin System



Outline

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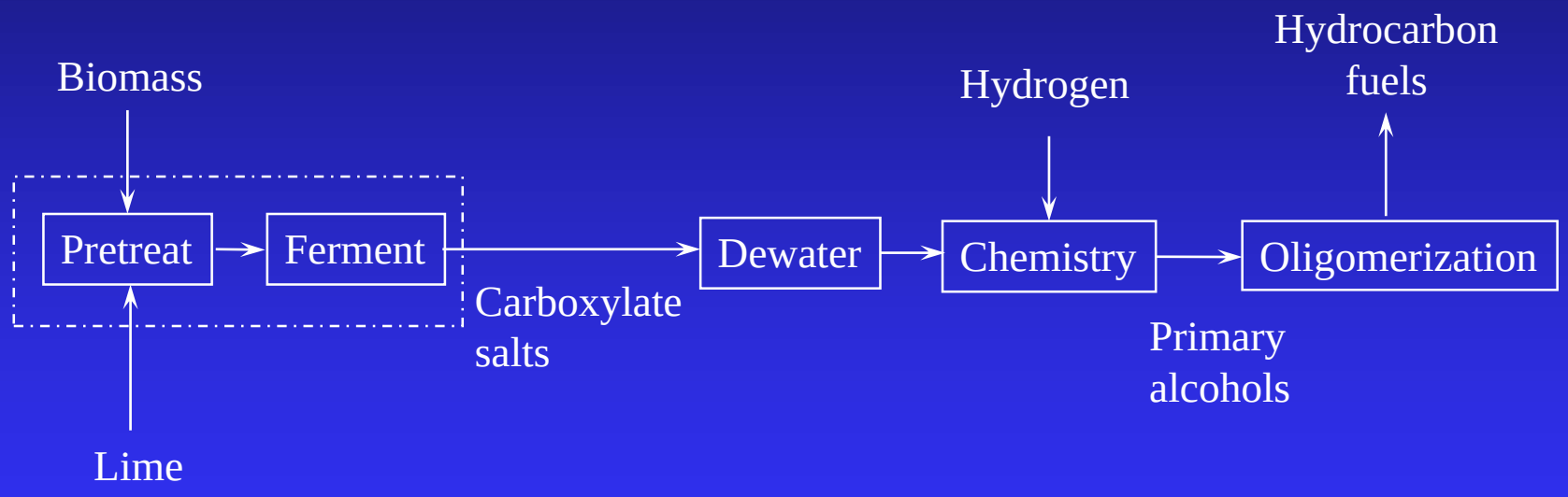
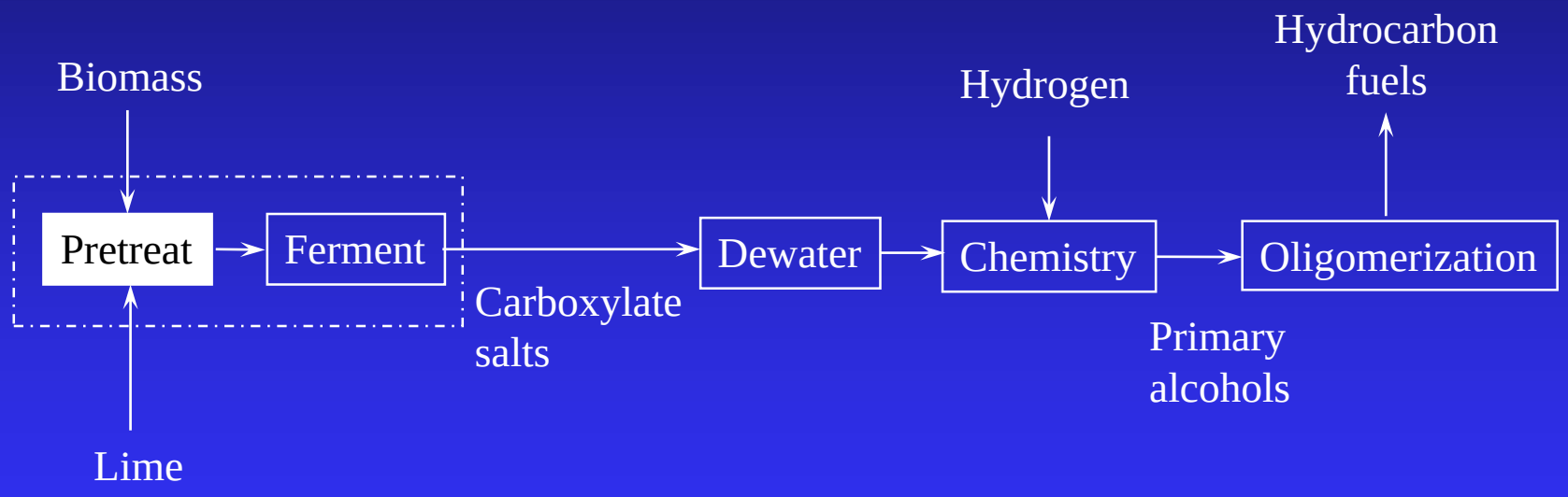
  - Primary alcohol route
  - Secondary alcohol route
  - Kolbe electrolysis
- Economics
- History
- Conclusions
- Future improvements

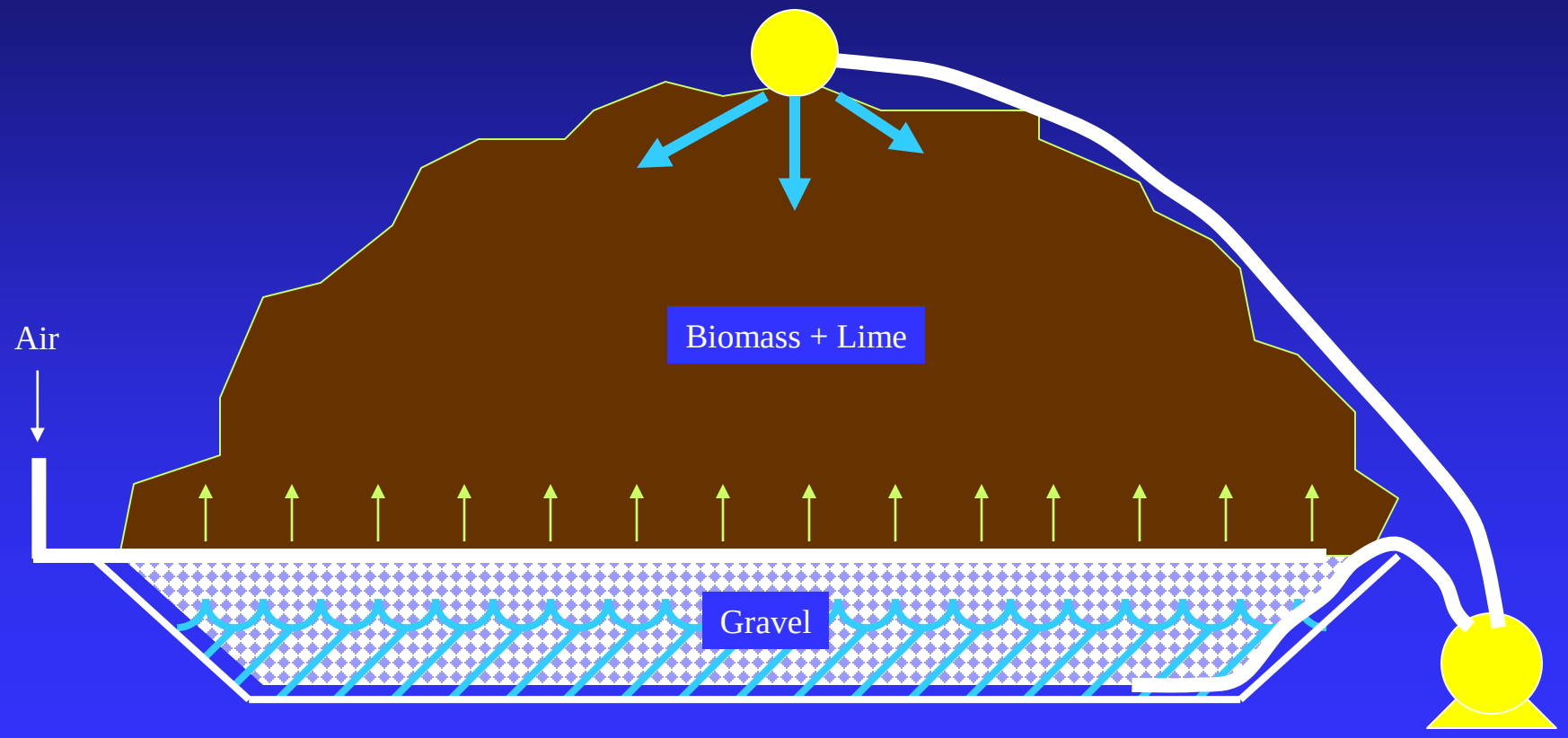
MixAlco Process



MixAlco Process



Oxidative Lime Preatment (OLP)

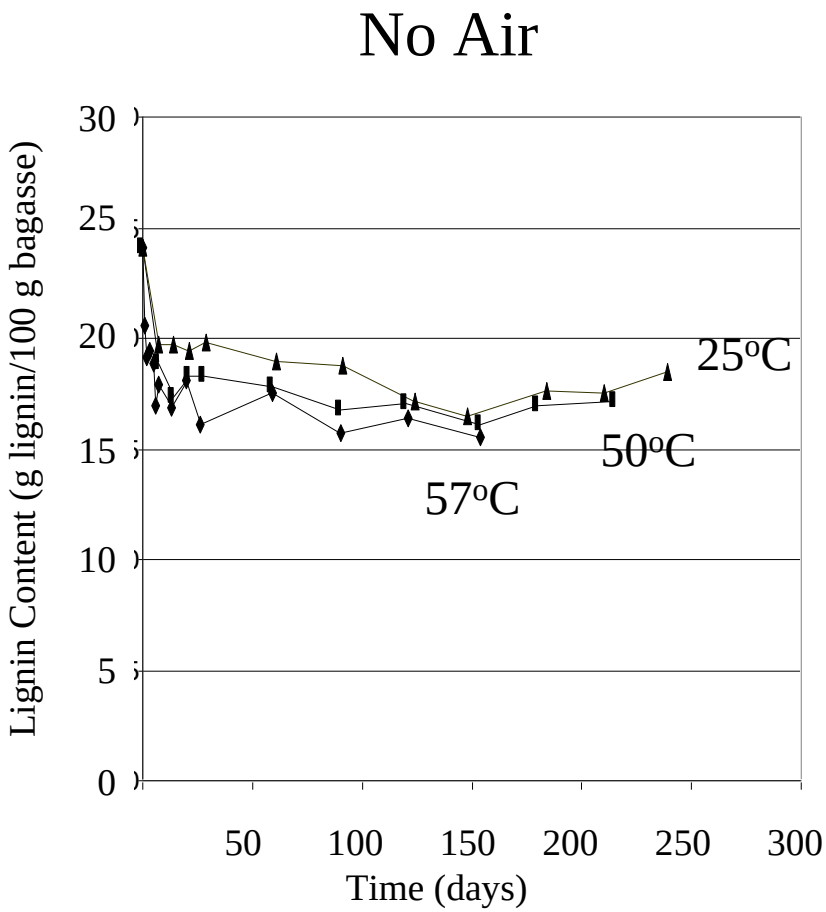


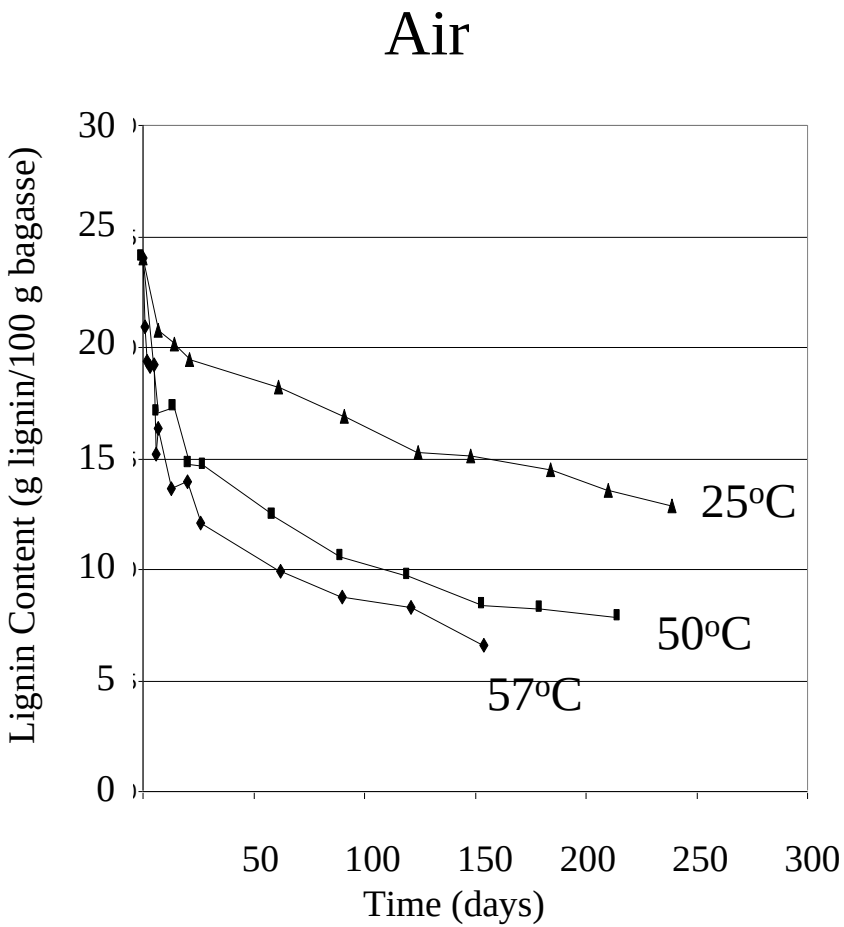
Building the Pile

~100 ft

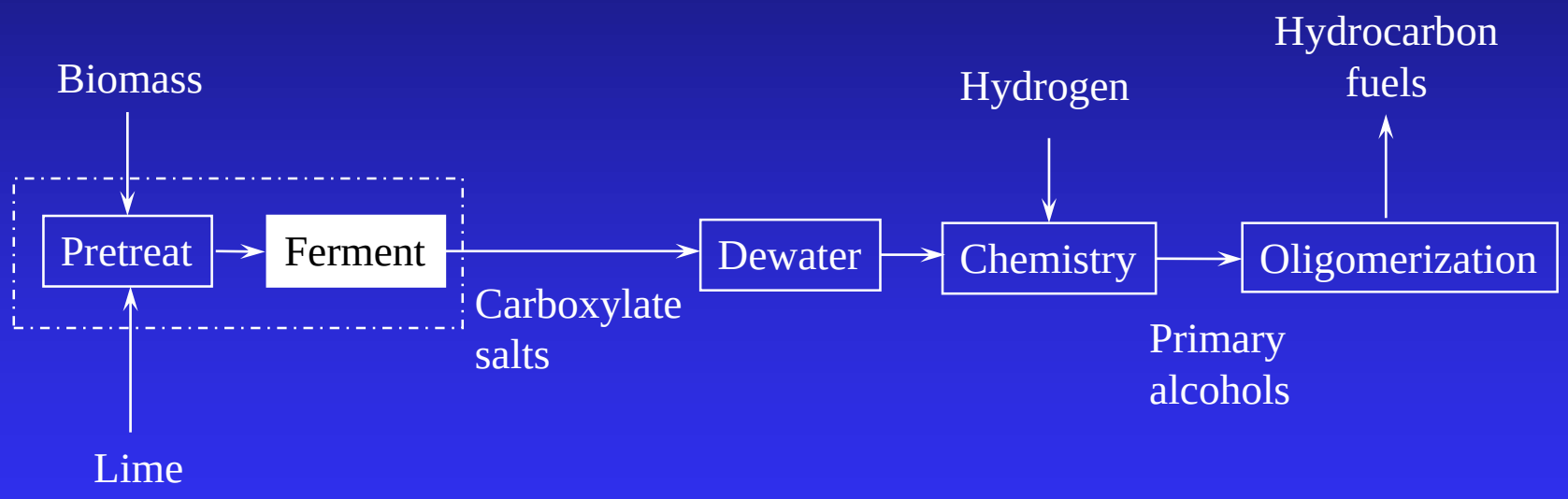
Building the Pile

Lignin Removal

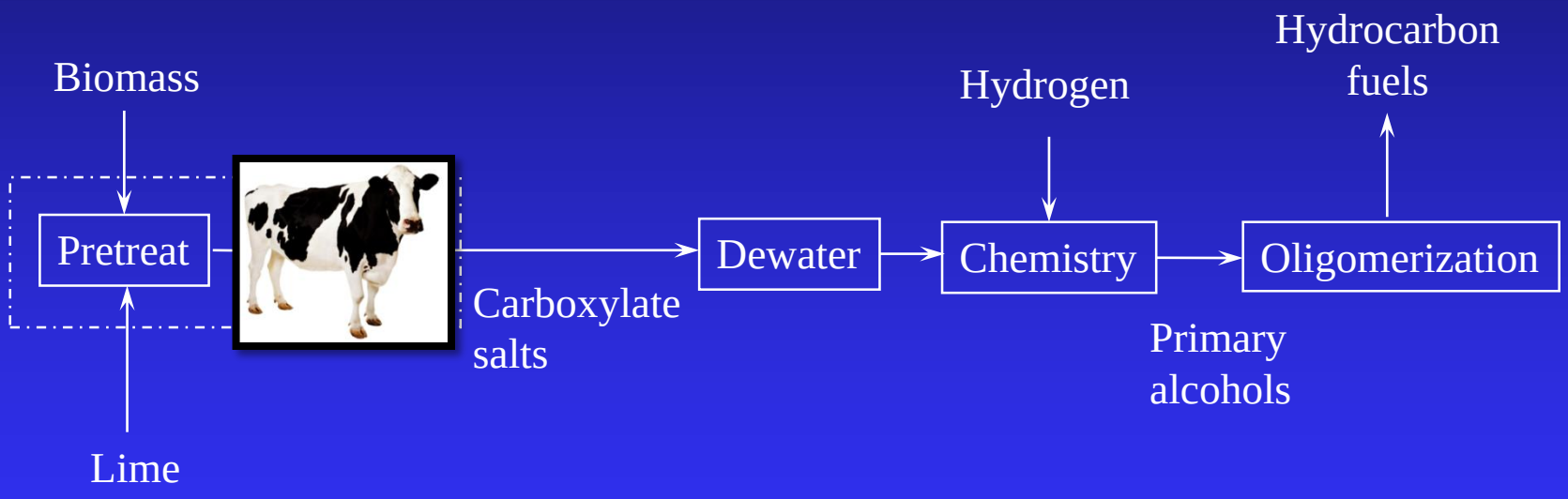




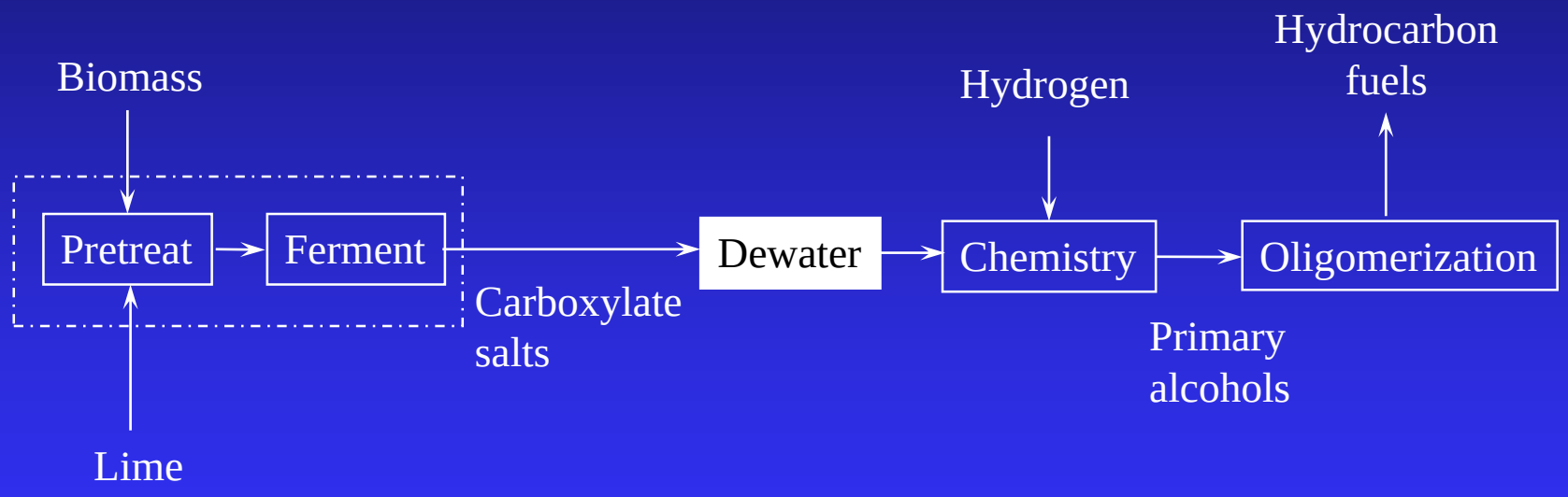
MixAlco Process



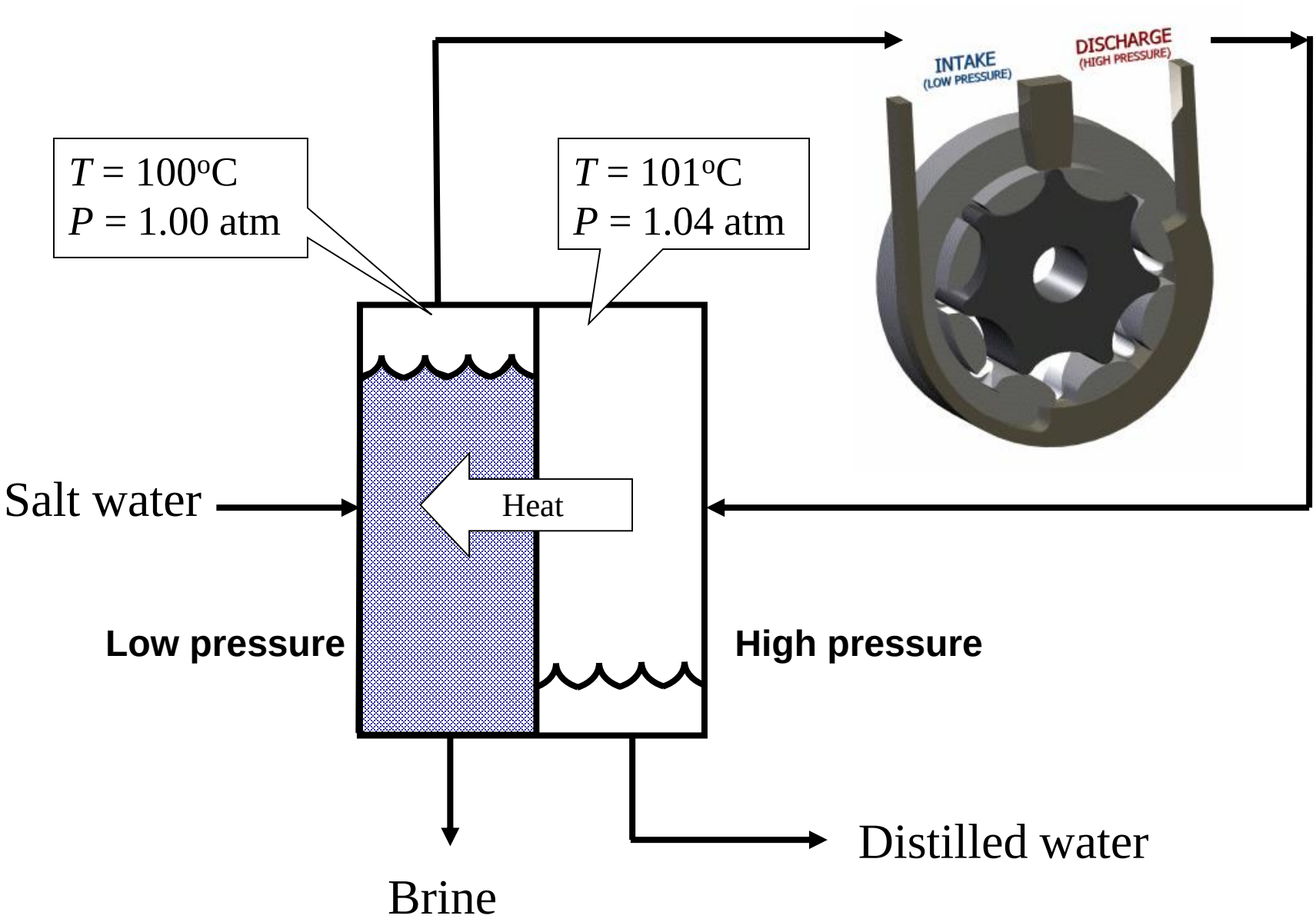
MixAlco Process



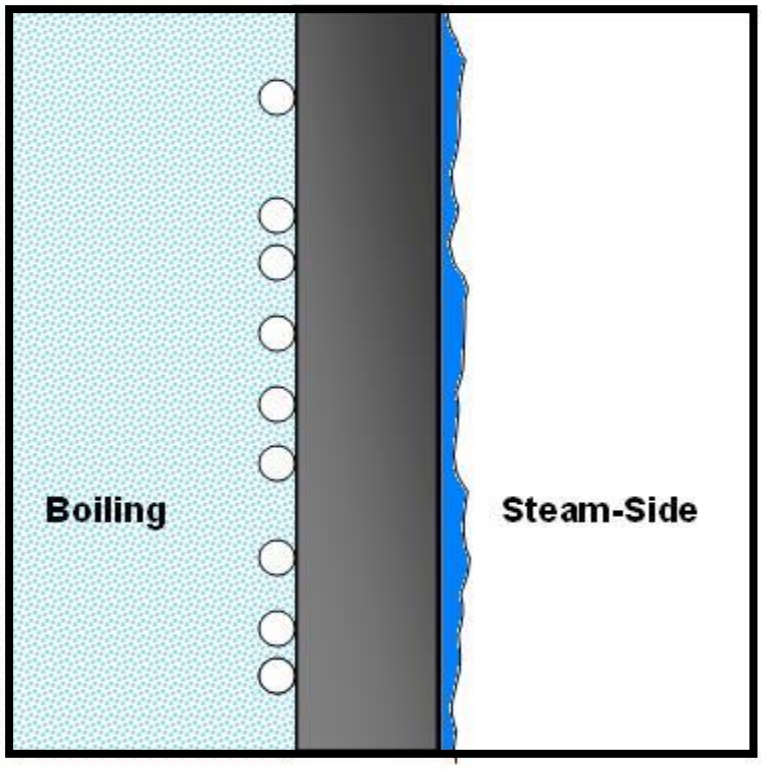
MixAlco Process



Vapor-Compression Desalination

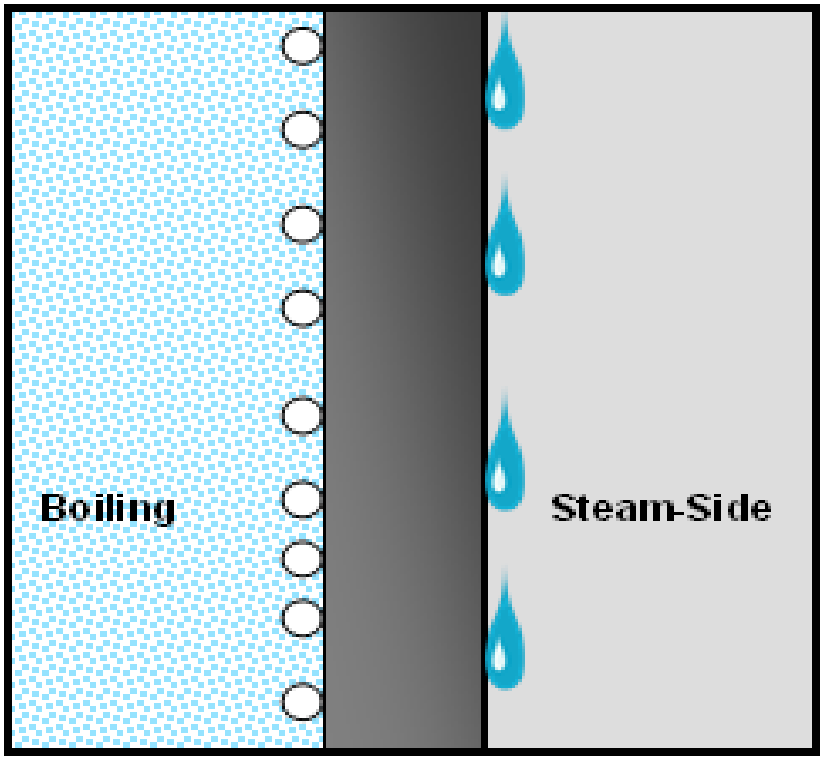


Filmwise Condensation



~2,000 Btu/(h·ft^2·^{o}F)

Dropwise Condensation



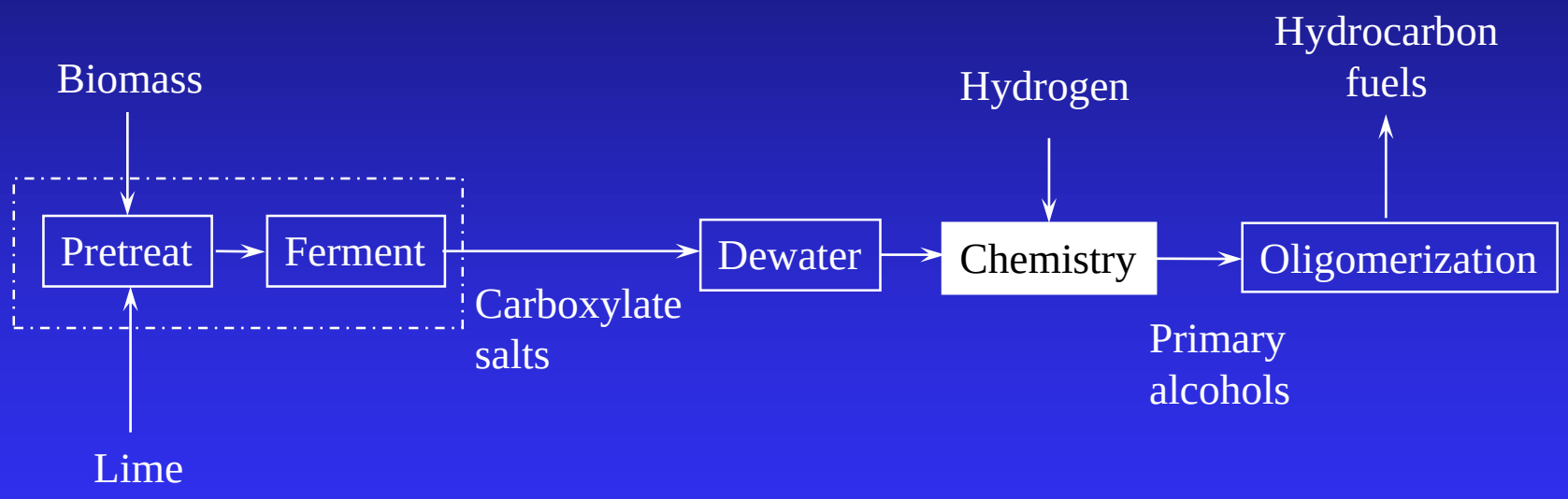
~42,000 Btu/(h·ft^2·^{o}F)

Laredo Desalination Project

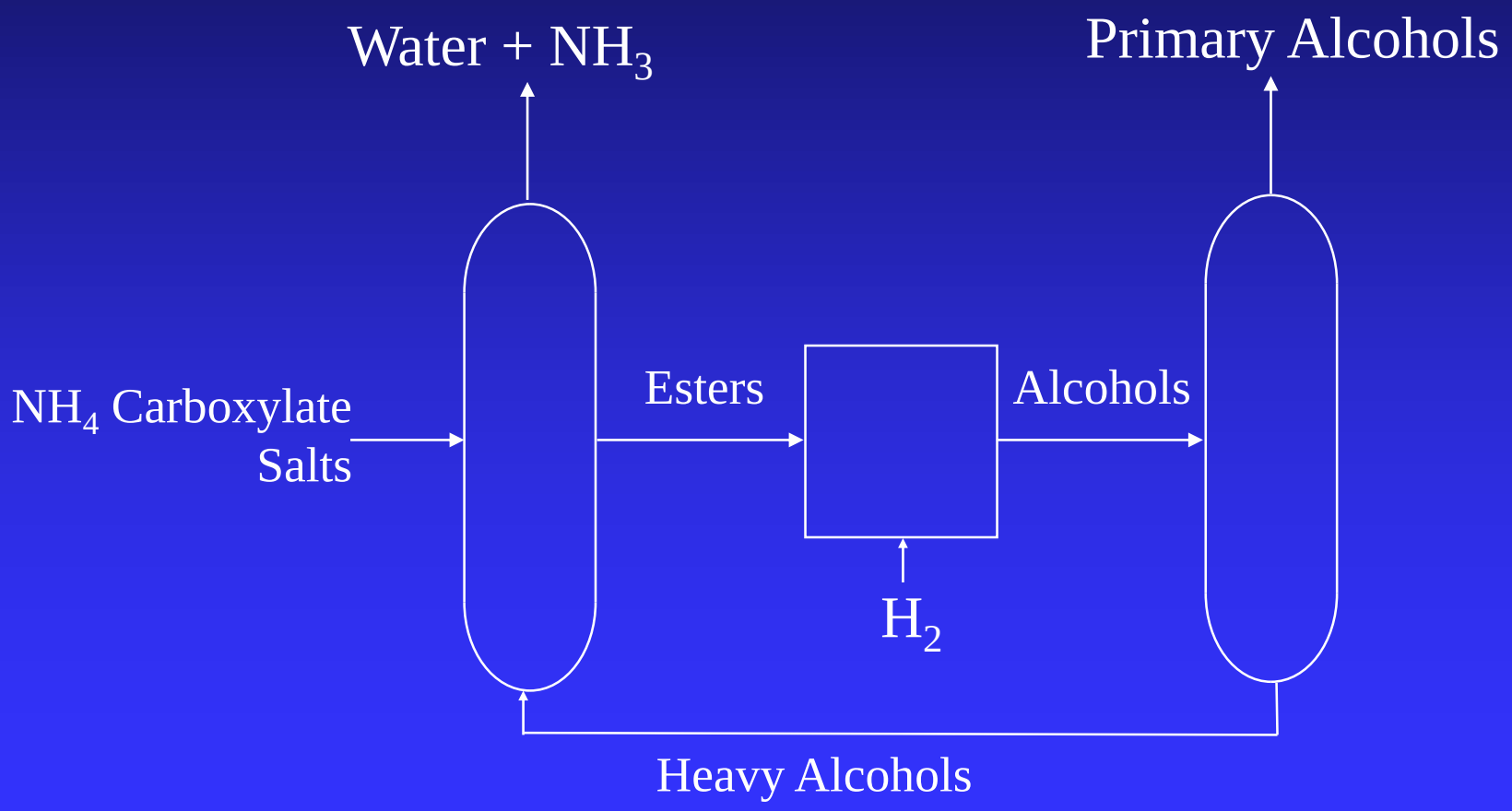
Laredo Desalination Project

STAR ROTOR
Corporation

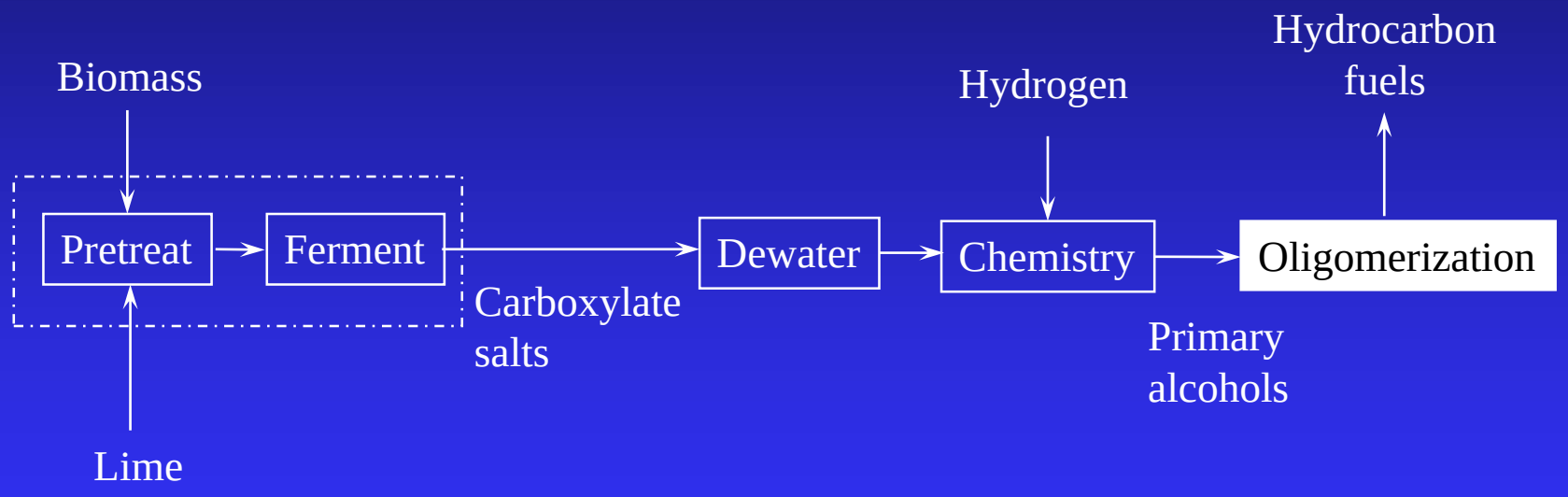
MixAlco Process



Chemistry


Water + NH_3
Primary Alcohols
NH_4 Carboxylate Salts
Esters
Alcohols
H_2
Heavy Alcohols

MixAlco Process



Oligomerizaton Chemistry

Dehydrate

$$H_3C\overset{\overset{H}{|}}{\underset{\underset{H}{|}}{C}}OH \rightarrow H_2C{=}CH_2 + H_2O$$

Ethanol Ethylene

Trimerize

$$H_2C{=}CH_2 + H_2C{=}CH_2 + H_2C{=}CH_2 \rightarrow H_3CCH_2CH_2HC{=}CHCH_3$$

Ethylene Ethylene Ethylene Hexene

Saturate

$$H_3CCH_2CH_2\ HC{=}CHCH_3 + H_2 \rightarrow H_3CCH_2CH_2CH_2CH_2CH_3$$

Hexene Hexane

Outline

- Carboxylate platform (MixAlco)
  - Primary alcohol route
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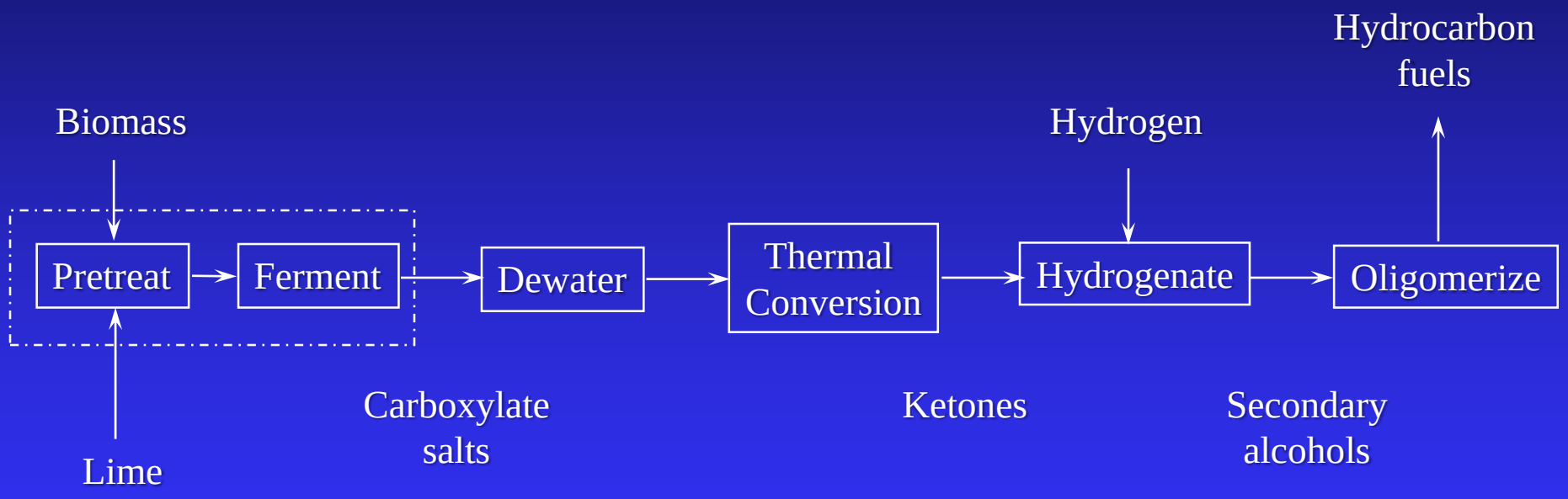
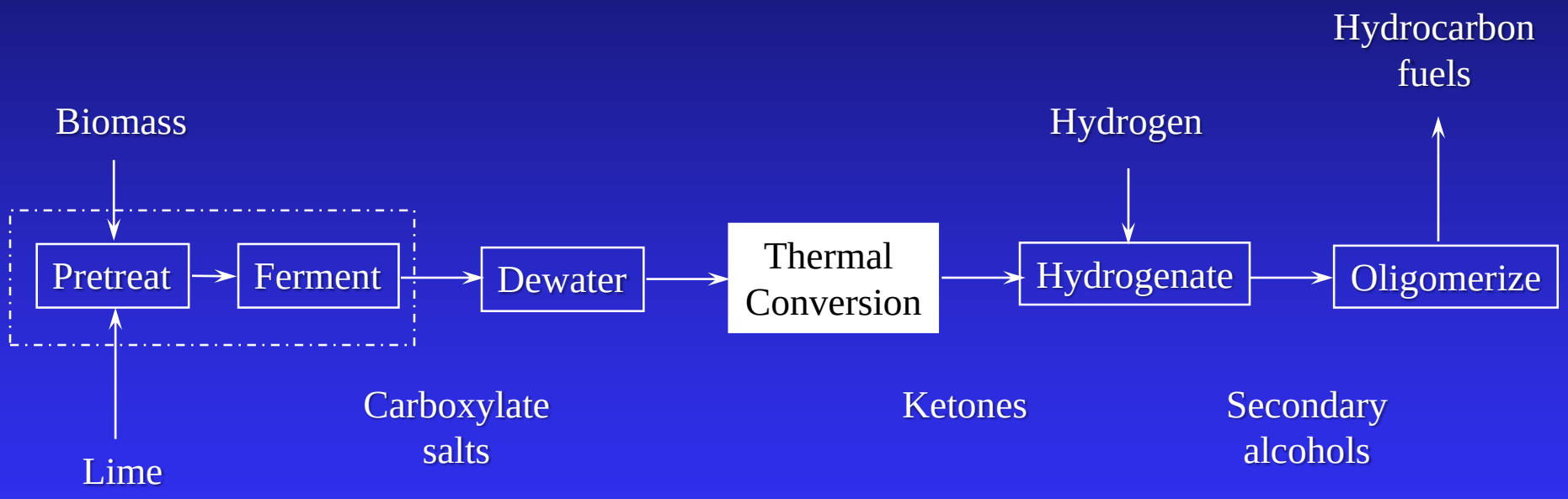
MixAlco Process



MixAlco Process



Thermal Conversion Stoichiometry

$$H_3C\overset{\overset{\displaystyle O}{\|}}{C}OCaO\overset{\overset{\displaystyle O}{\|}}{C}CH_3 \rightarrow H_3C\overset{\overset{\displaystyle O}{\|}}{C}CH_3 + CaCO_3$$

Calcium Acetate Acetone

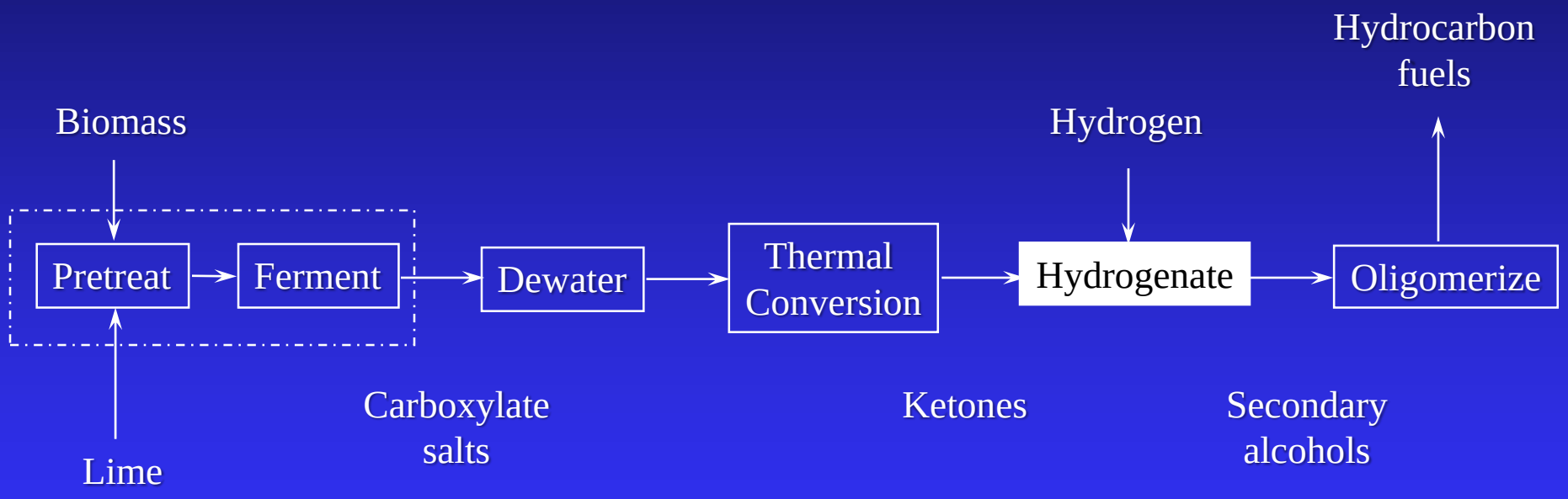
$$H_3CCH_2\overset{\overset{\displaystyle O}{\|}}{C}OCaO\overset{\overset{\displaystyle O}{\|}}{C}CH_2CH_3 \rightarrow H_3CCH_2\overset{\overset{\displaystyle O}{\|}}{C}CH_2CH_3 + CaCO_3$$

Calcium Propionate Diethyl Ketone

$$H_3CCH_2CH_2\overset{\overset{\displaystyle O}{\|}}{C}OCaO\overset{\overset{\displaystyle O}{\|}}{C}CH_2CH_2CH_3 \rightarrow H_3CCH_2CH_2\overset{\overset{\displaystyle O}{\|}}{C}CH_2CH_2CH_3 + CaCO_3$$

Calcium Butyrate Dipropyl Ketone

MixAlco Process



Ketone Hydrogenation Stoichiometry

$$H_3C\overset{\overset{\displaystyle O}{\|}}{C}CH_3 + H_2 \rightarrow H_3C\underset{\underset{\displaystyle H}{|}}{\overset{\overset{\displaystyle OH}{|}}{C}}CH_3$$

Acetone Isopropanol

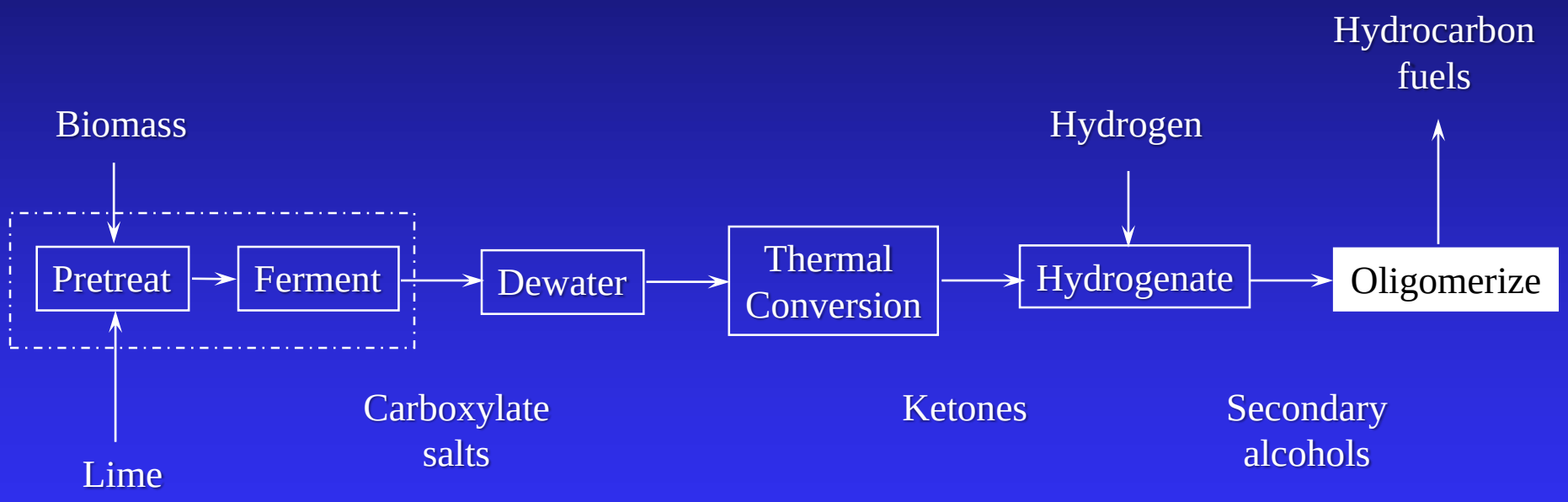
$$H_3C\overset{\overset{\displaystyle O}{\|}}{C}CH_2CH_3 + H_2 \rightarrow H_3C\underset{\underset{\displaystyle H}{|}}{\overset{\overset{\displaystyle OH}{|}}{C}}CH_2CH_3$$

Methyl Ethyl Ketone 2-Butanol

$$H_3CCH_2\overset{\overset{\displaystyle O}{\|}}{C}CH_2CH_3 + H_2 \rightarrow H_3CCH_2\underset{\underset{\displaystyle H}{|}}{\overset{\overset{\displaystyle OH}{|}}{C}}CH_2CH_3$$

Diethyl Ketone 3-Pentanol

MixAlco Process



Oligomerizaton Chemistry

Dehydrate

$$H_3C\overset{\overset{\displaystyle OH}{|}}{\underset{\underset{\displaystyle H}{|}}{C}}CH_3 \rightarrow H_3CHC{=}CH_2 + H_2O$$

Isopropanol Propylene

Dimerize

$$H_3CHC{=}CH_2 + H_3CHC{=}CH_2 \rightarrow H_3CCH_2CH_2\ HC{=}CHCH_3$$

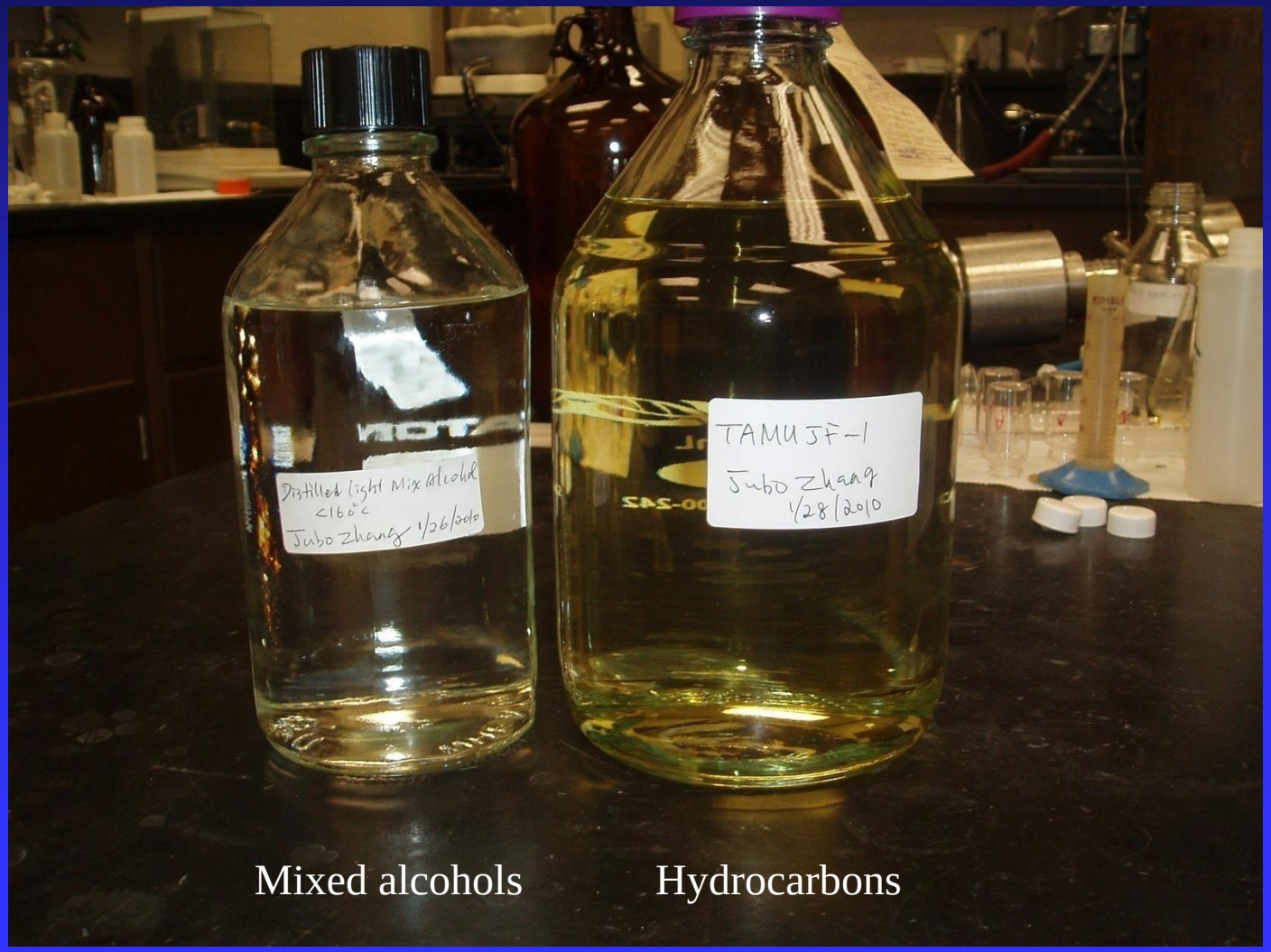
Propylene Propylene Hexene

Saturate

$$H_3CCH_2CH_2\ HC{=}CHCH_3 + H_2 \rightarrow H_3CCH_2CH_2CH_2CH_2CH_3$$

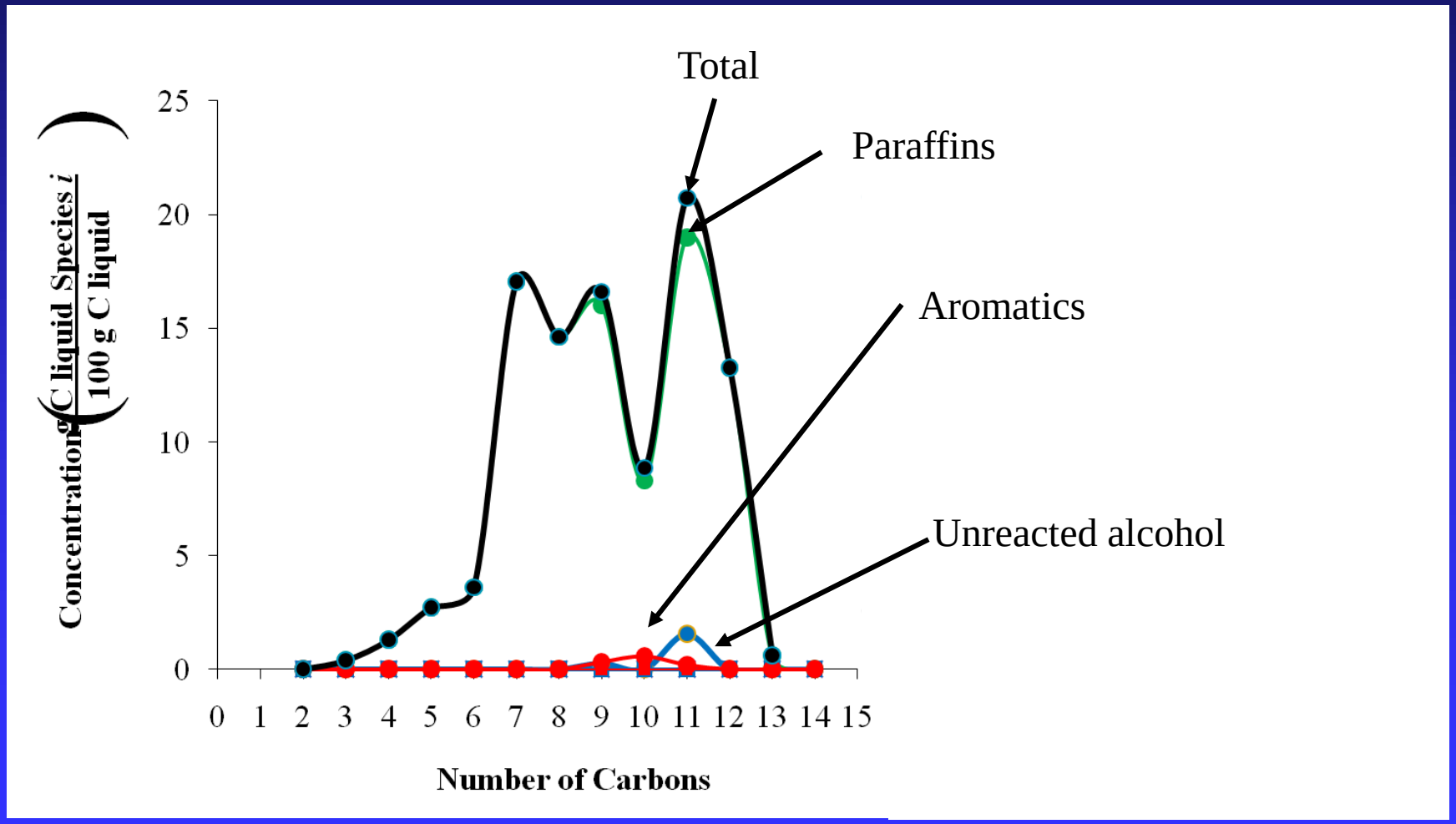
Hexene Hexane

Mixed Alcohols to Hydrocarbons

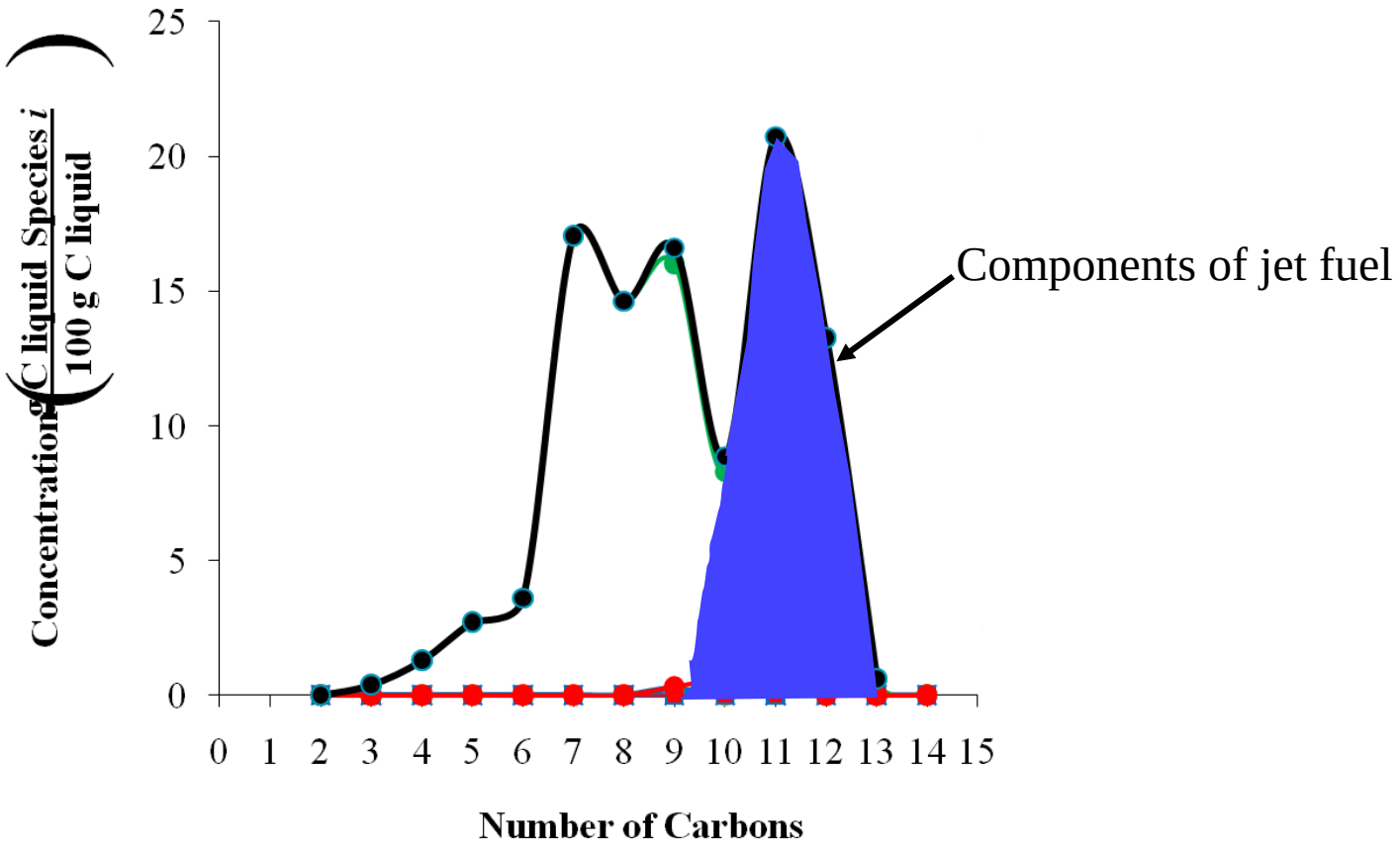


Mixed alcohols Hydrocarbons

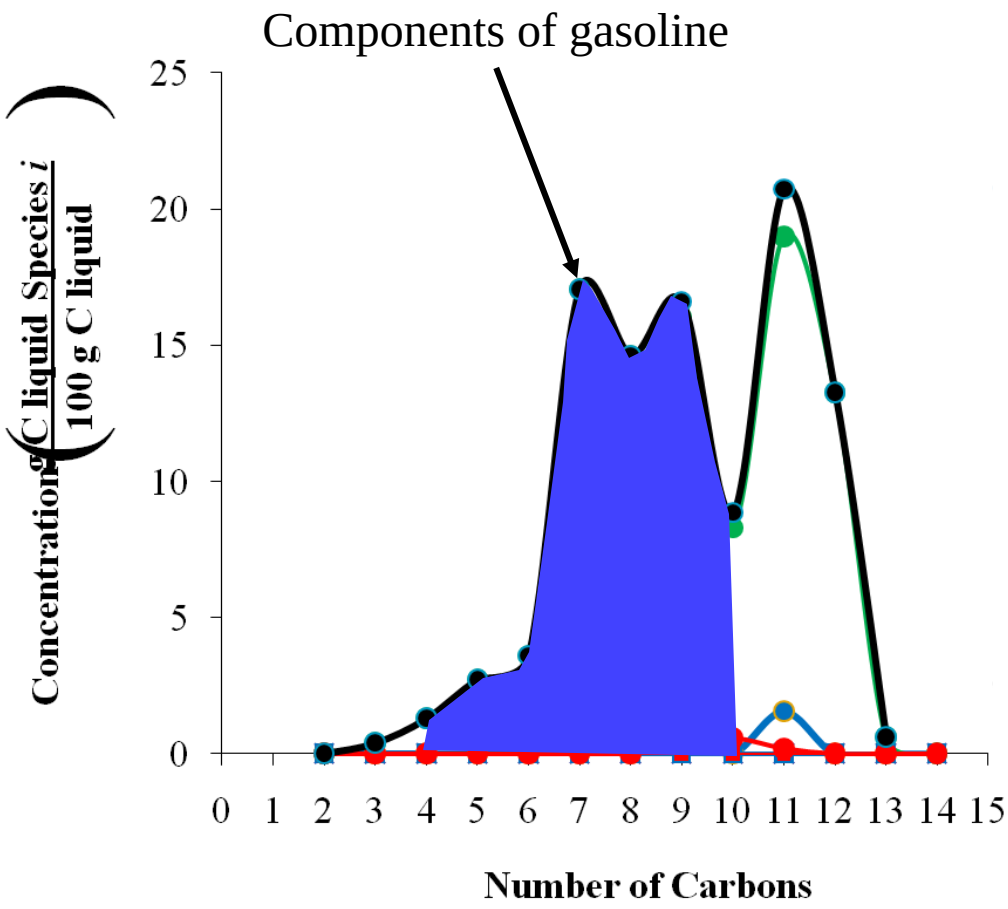
Mixed Alcohols to Hydrocarbons



Mixed Alcohols to Hydrocarbons



Mixed Alcohols to Hydrocarbons



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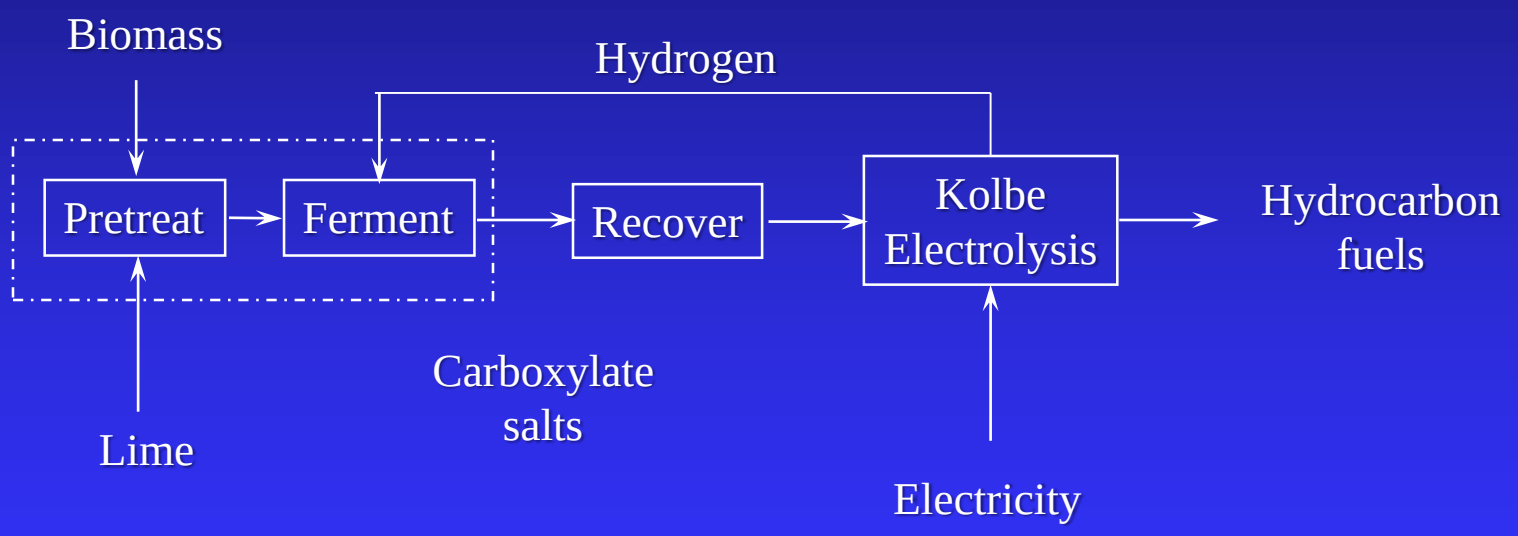

$$RCOOH + HOOCR' \rightarrow R\text{–}R' + 2\ CO_2 + H_2$$

Outline

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Comparison of three routes

Primary Alcohol

$RH_2C–COOH + 2\ H_2 \rightarrow RH_2C–CH_2OH + H_2O$

$RH_2C–C\ H_2OH \rightarrow RHC= CH_2 + H_2O$

$RHC= CH_2 + H_2 \rightarrow RH_2C–CH_3$

$RH_2C–COOH + 3\ H_2 \rightarrow RH_2C–CH_3 + 2\ H_2O$

Secondary Alcohol

$RCOOH + HOOCCH_2R' \rightarrow RCOCH_2R' + CO_2 + H_2O$

$RCOCH_2R' + H_2 \rightarrow RHCOHCH_2R'$

$RHCOHCH_2R' \rightarrow RC= CH_2R' + H_2O$

$RC= CH_2R' + H_2 \rightarrow RCH_2–CH_2R'$

$RCOOH + HOOCCH_2R' + 2\ H_2 \rightarrow RCH_2–CH_2R' + CO_2 + 2\ H_2O$

Kolbe

$RCOOH + HOOCR' \rightarrow R–R' + 2\ CO_2 + H_2$

Comparison of three routes

Primary Alcohol

$RH_2C–COOH + 2\ H_2 \rightarrow RH_2C–CH_2OH + H_2O$

$RH_2C–C\ H_2OH \rightarrow RHC= CH_2 + H_2O$

$RHC= CH_2 + H_2 \rightarrow RH_2C–CH_3$

$RH_2C–COOH + 3\ H_2 \rightarrow RH_2C–CH_3 + 2\ H_2O$

Secondary Alcohol

$RCOOH + HOOCCH_2R' \rightarrow RCOCH_2R' + CO_2 + H_2O$

$RCOCH_2R' + H_2 \rightarrow RHCOHCH_2R'$

$RHCOHCH_2R' \rightarrow RC= CH_2R' + H_2O$

$RC= CH_2R' + H_2 \rightarrow RCH_2–CH_2R'$

$RCOOH + HOOCCH_2R' + 2\ H_2 \rightarrow RCH_2–CH_2R' + CO_2 + 2\ H_2O$

Kolbe

$RCOOH + HOOCR' \rightarrow R–R' + 2\ CO_2 + H_2$

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$RC= CH_2R' + H_2 \rightarrow RCH_2–CH_2R'$

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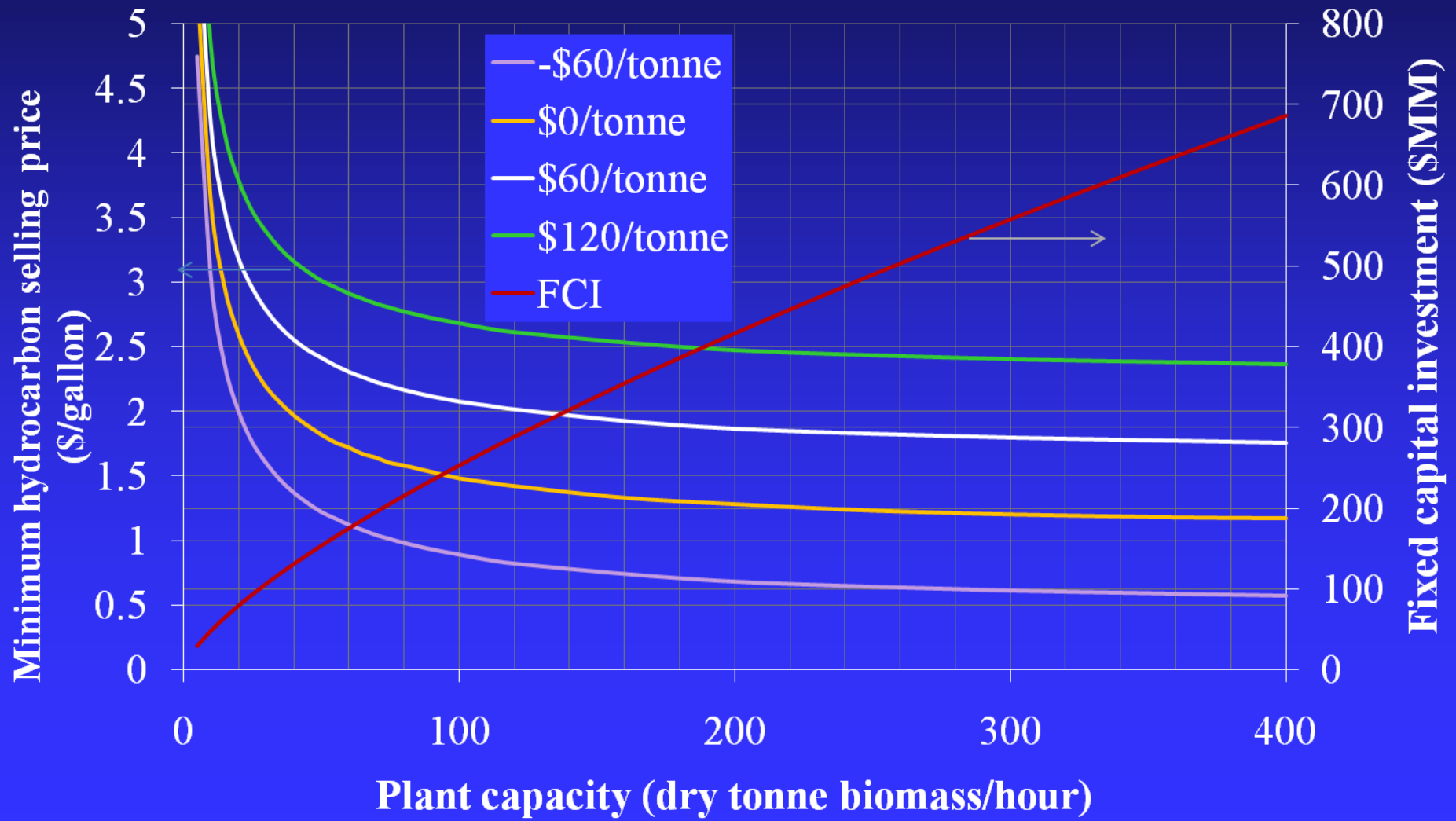
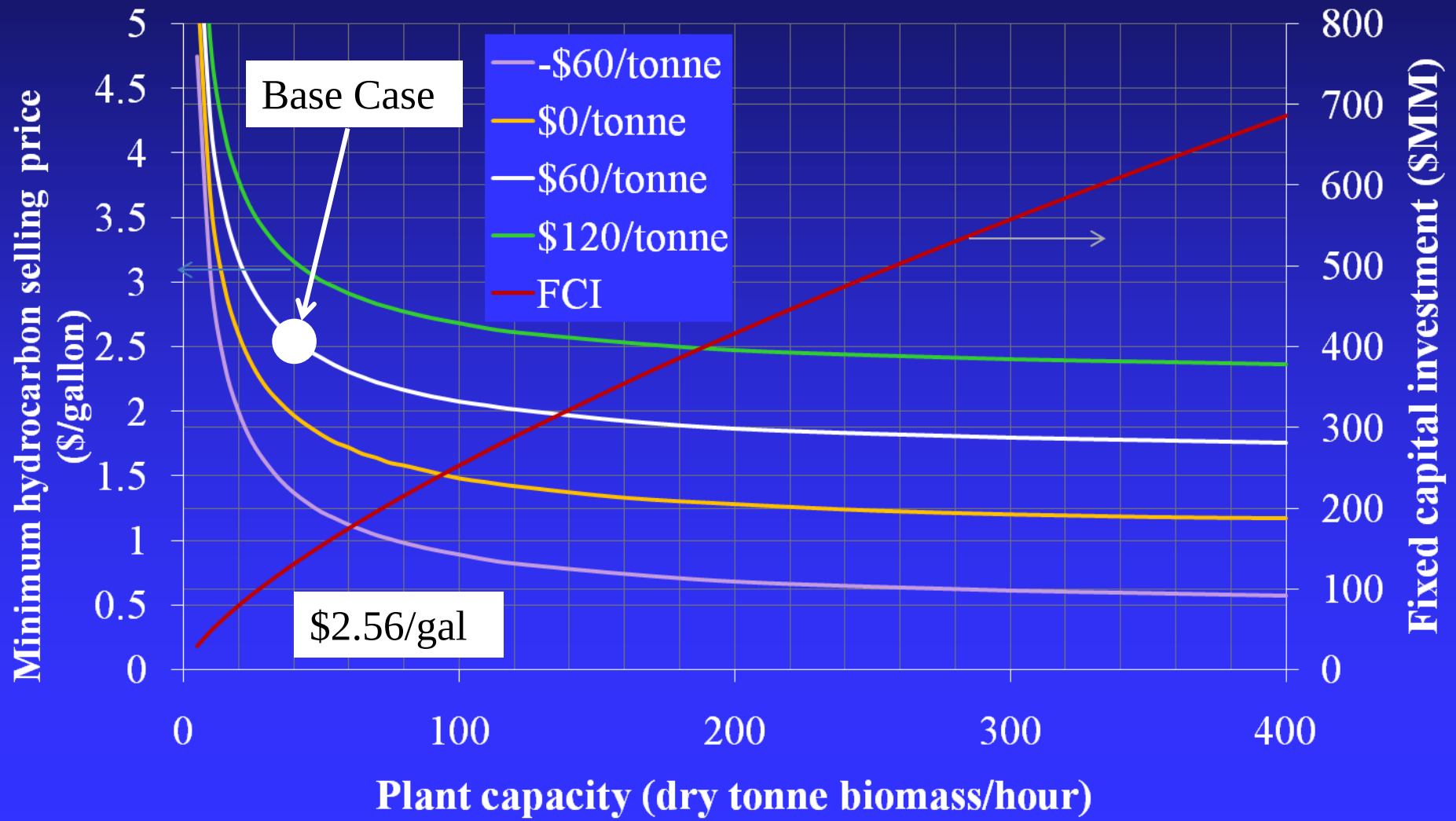
Sensitivity to Plant Capacity

Hydrogen from gasified residue



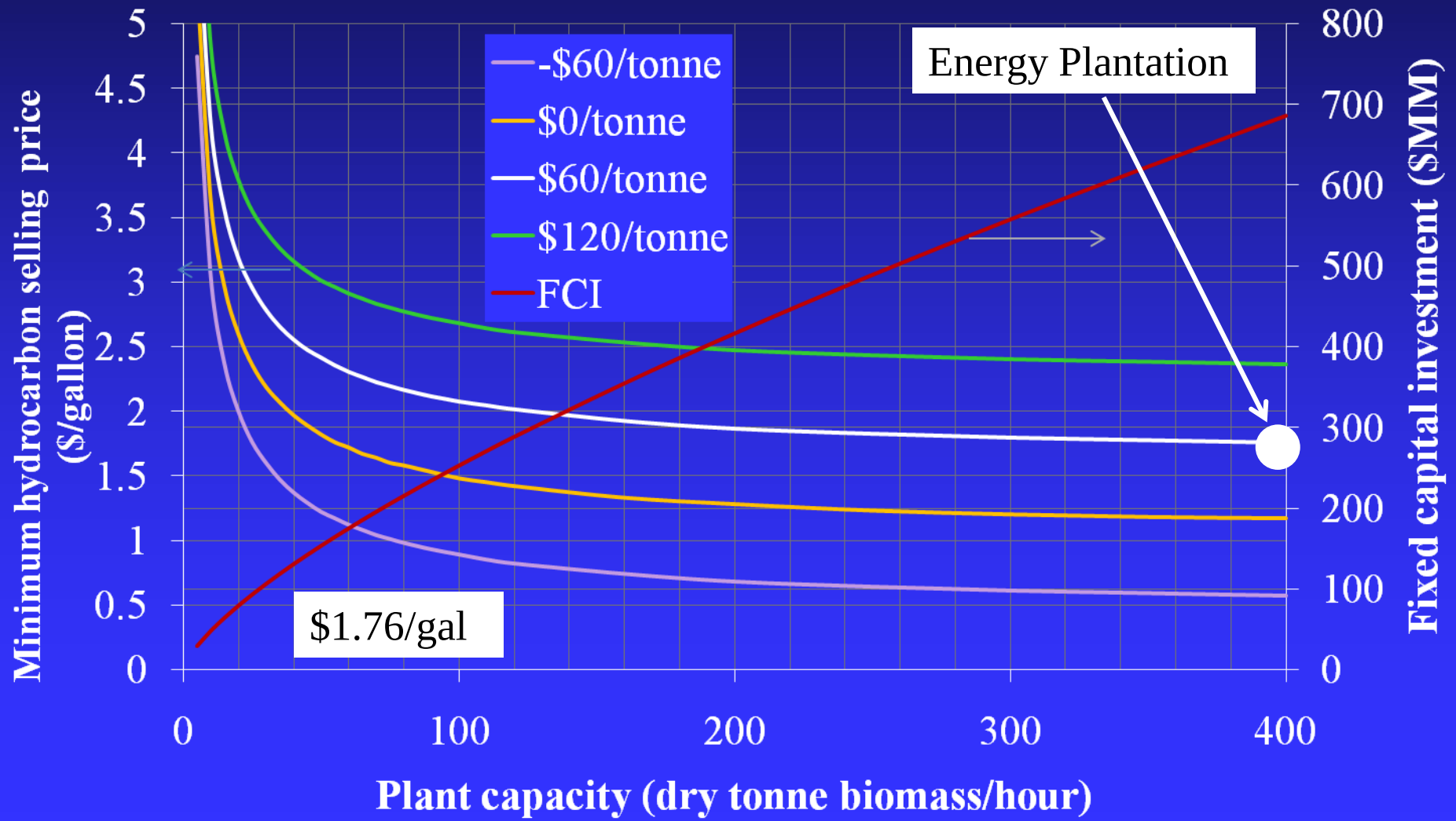
Sensitivity to Plant Capacity

Hydrogen from gasified residue



Sensitivity to Plant Capacity

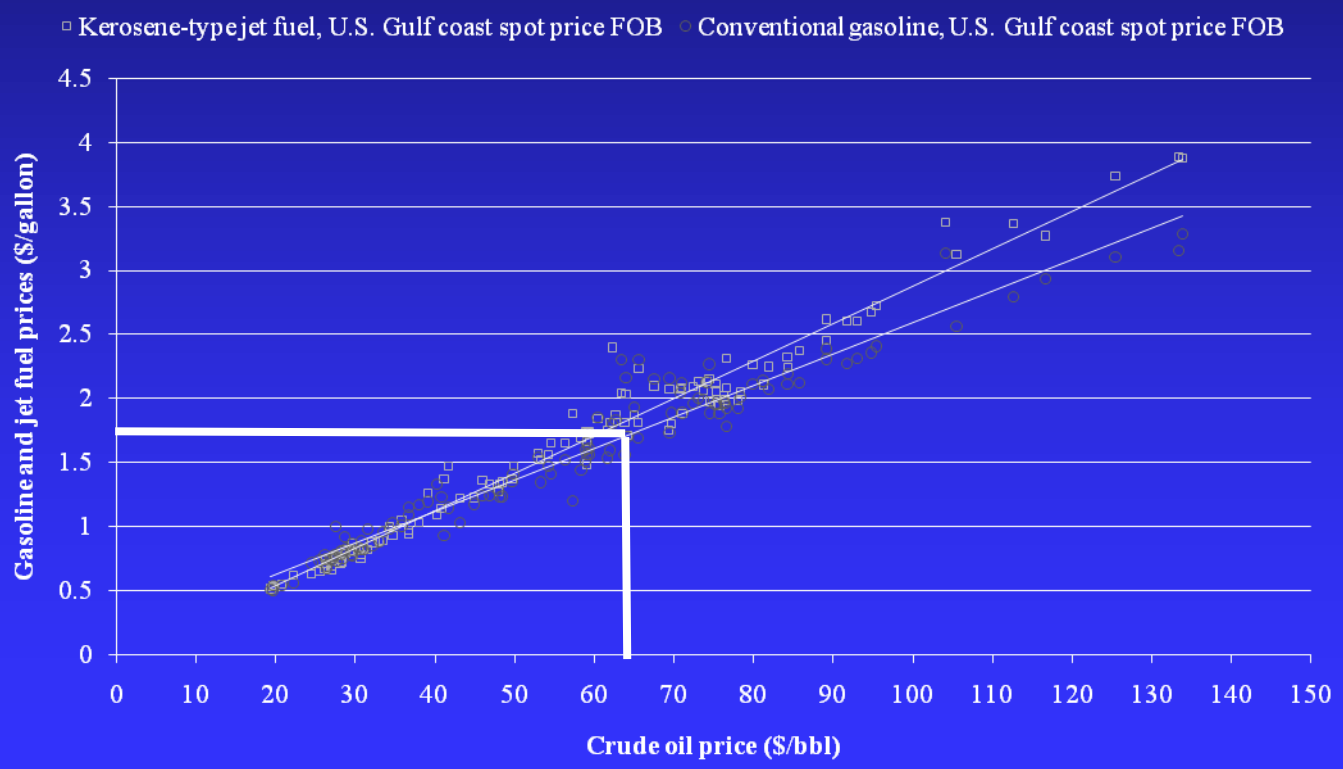
Hydrogen from gasified residue



Gasoline and jet fuel prices vs. crude oil price

At 400 tonne/h, minimum hydrocarbon selling price = $1.76/gal

Without subsidies, competes when crude oil = $63/bbl



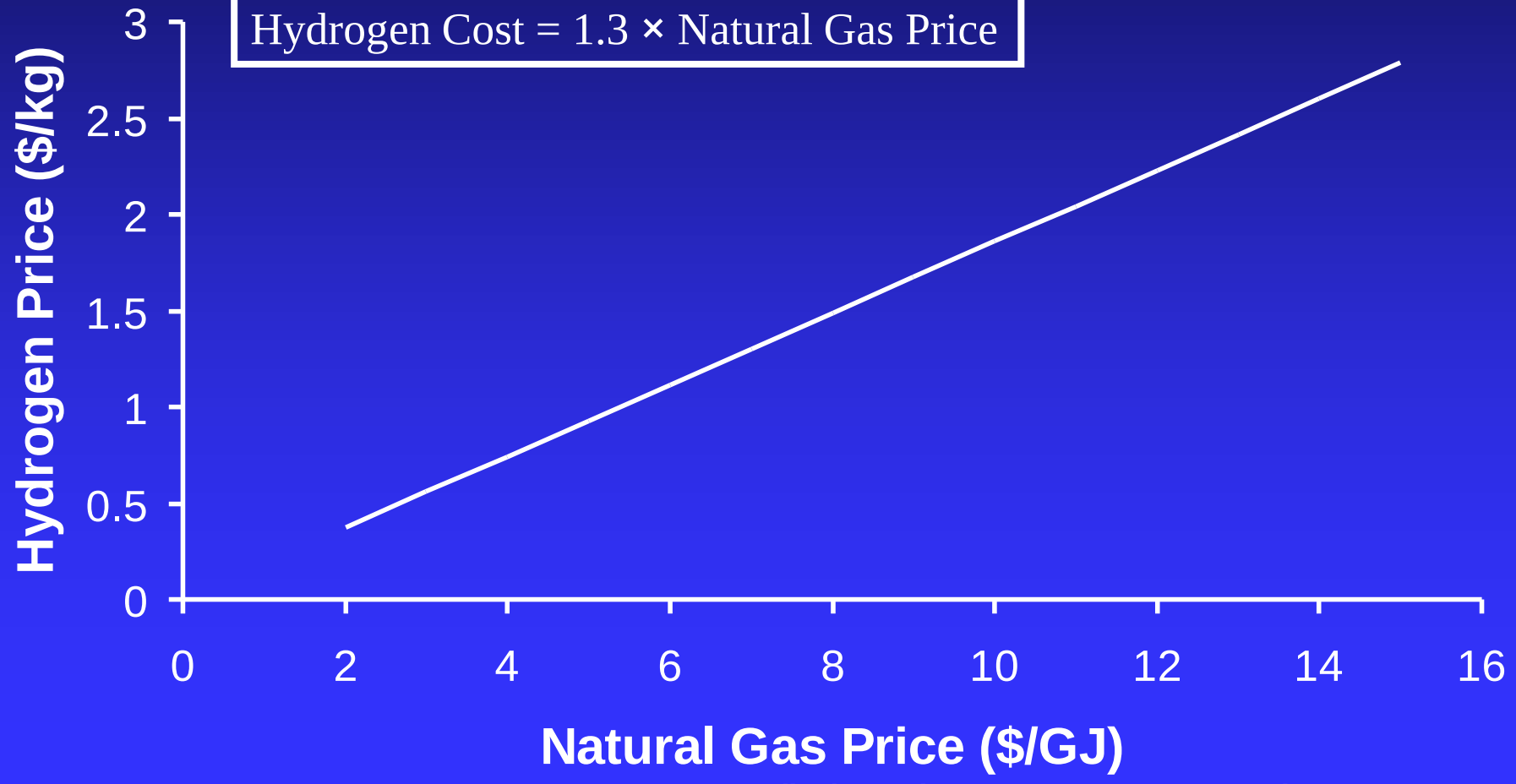
Source: *U.S. Energy Information Administration*

Hydrogen Sources

- Methane reforming
- Biomass gasification
- Electrolyzer

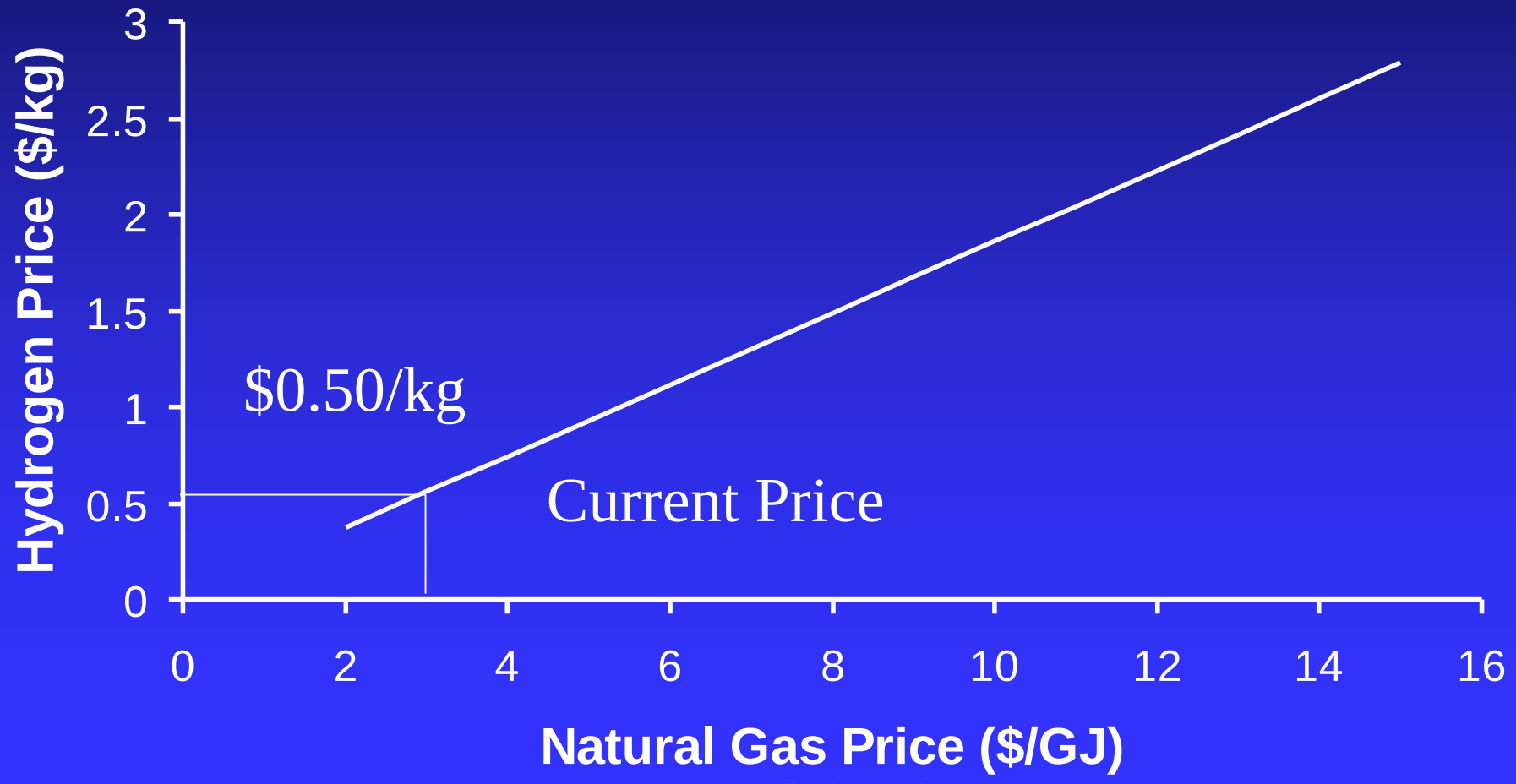
Methane reforming

On an energy basis…
Hydrogen Cost = 1.3 × Natural Gas Price



J. Gillingham, Valero Energy Corporation, personal communication (2008).

Methane reforming



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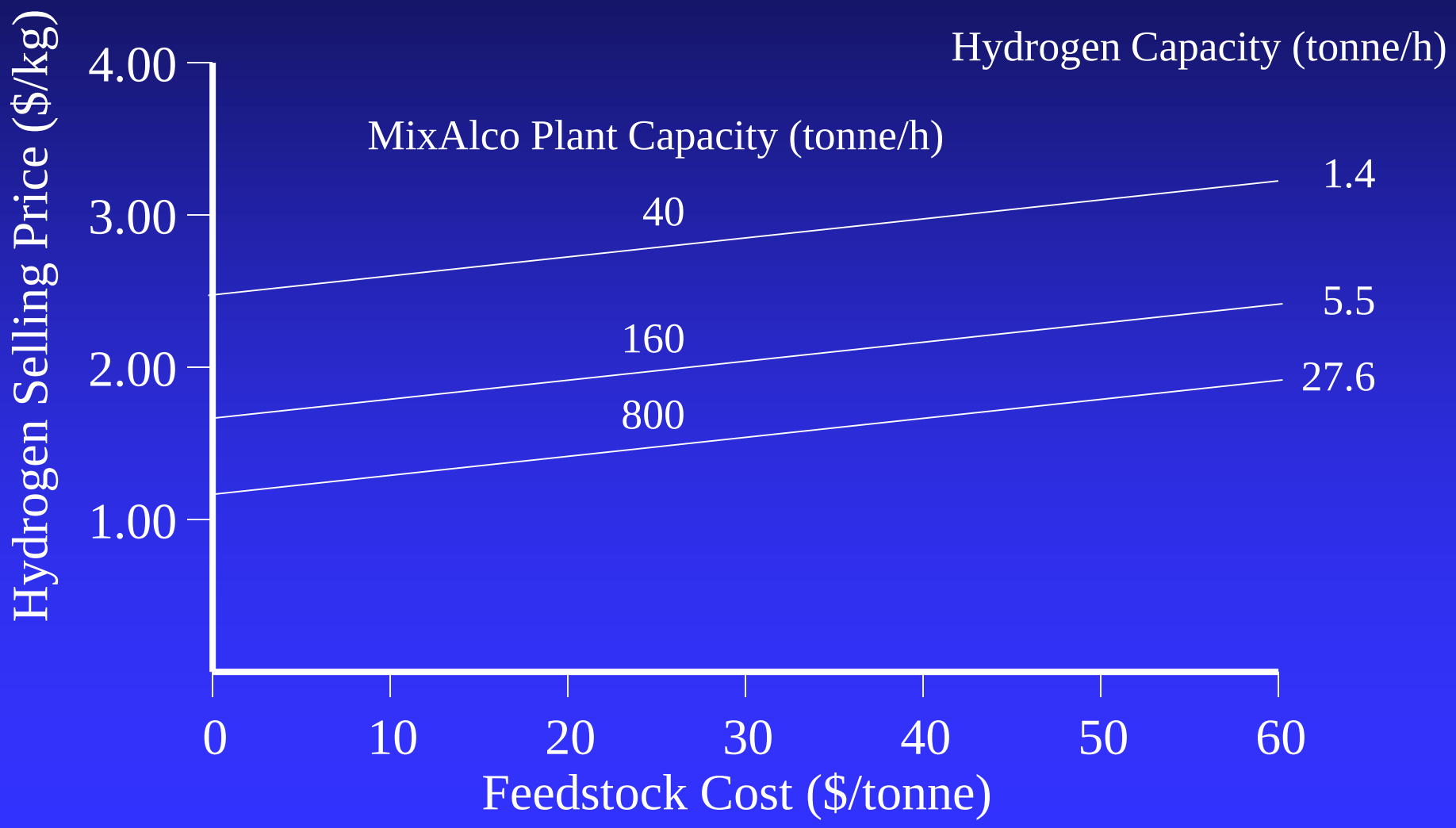
Hydrogen Sources

- Methane reforming
- Biomass gasification
- Electrolyzer

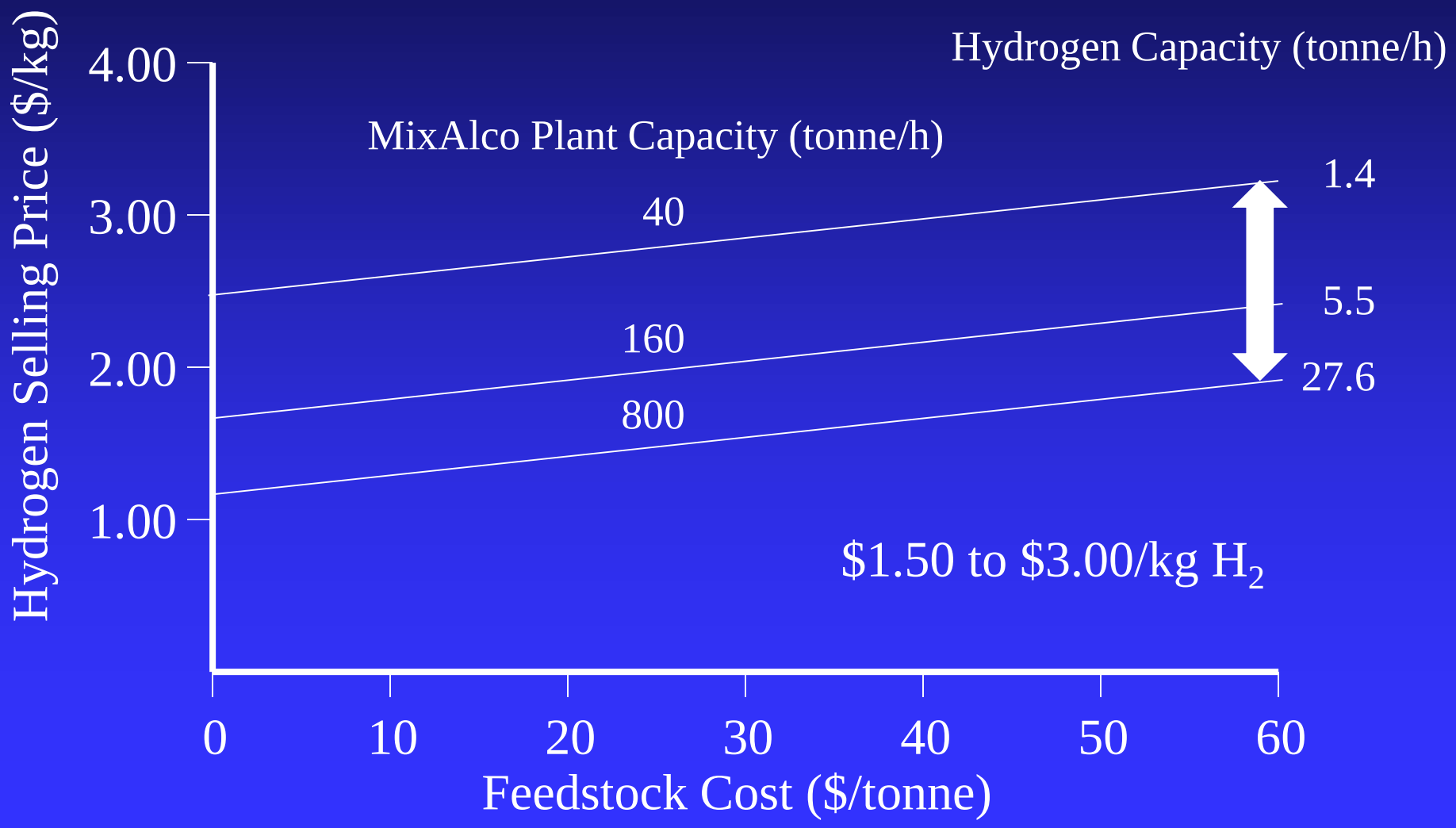
Biomass Gasifier

ROI = 15%



Biomass Gasifier

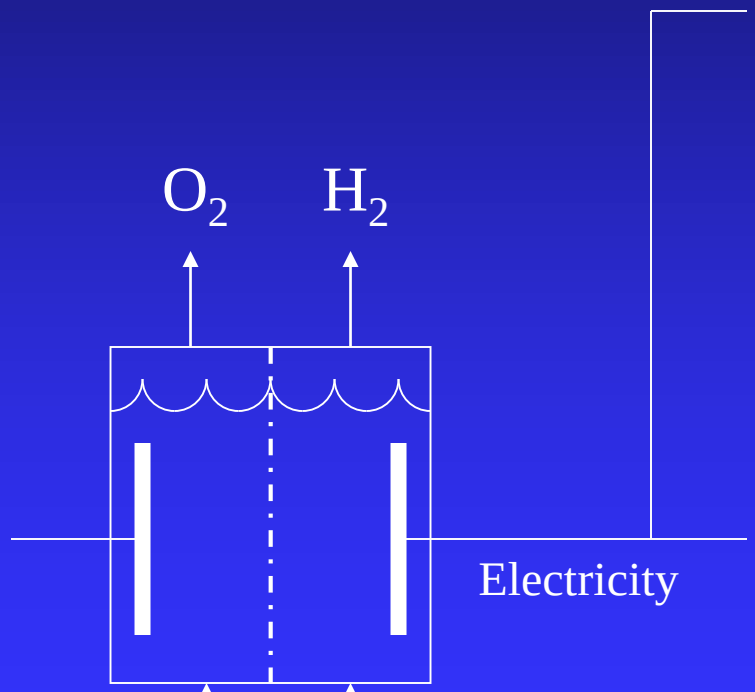
ROI = 15%



Hydrogen Sources

- Methane reforming
- Biomass gasification
- Electrolyzer

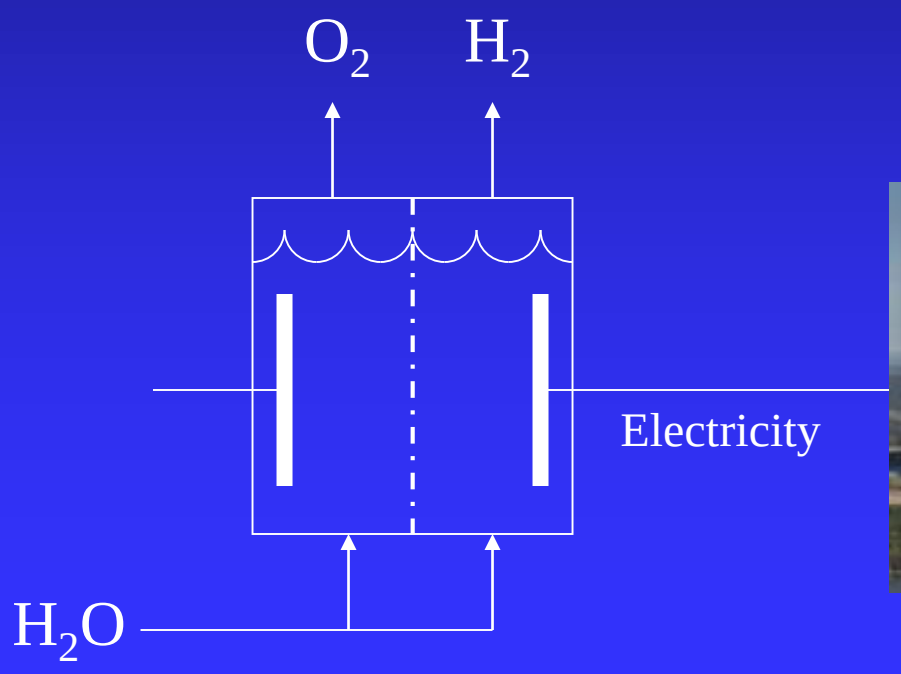
Water electrolysis



Solar

Wind

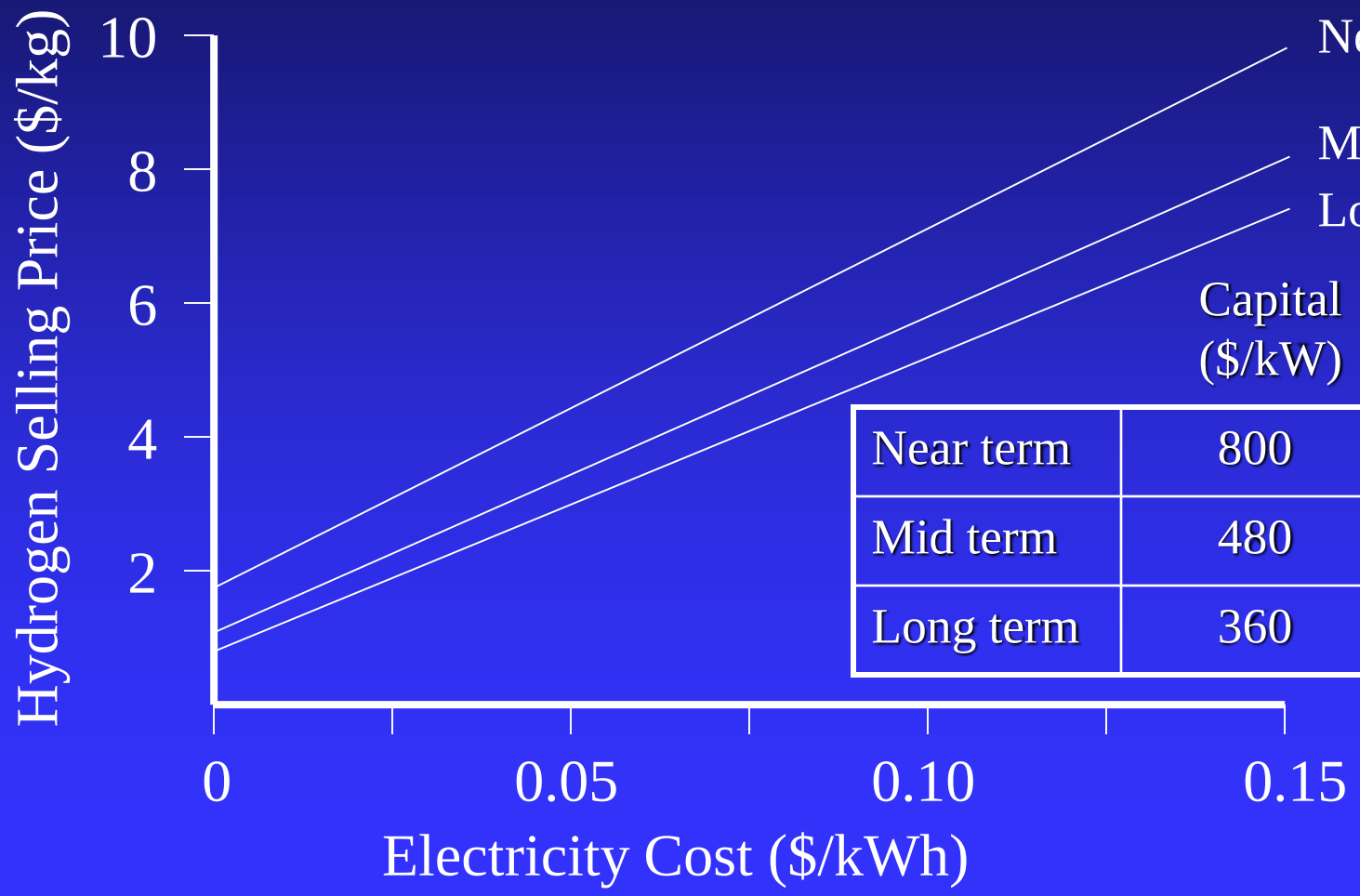
Water electrolysis



Nuclear

Electrolyzer Hydrogen

ROI = 15%

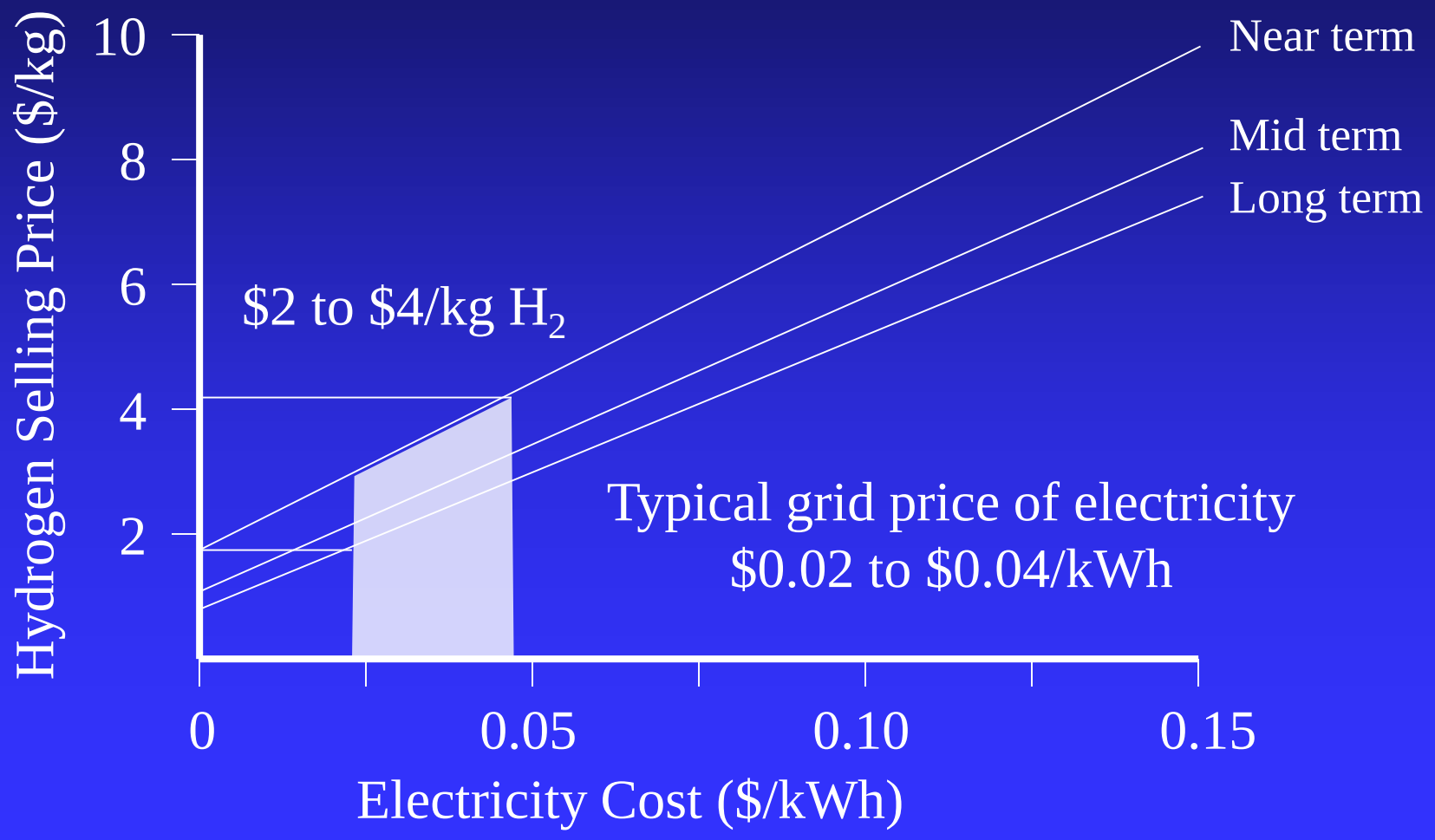


	Capital ($/kW)	Energy (kWh/kg H_2)
Near term	800	53.4
Mid term	480	47.9
Long term	360	44.7

http://www.nrel.gov/docs/fy05osti/38210.pdf

Electrolyzer Hydrogen

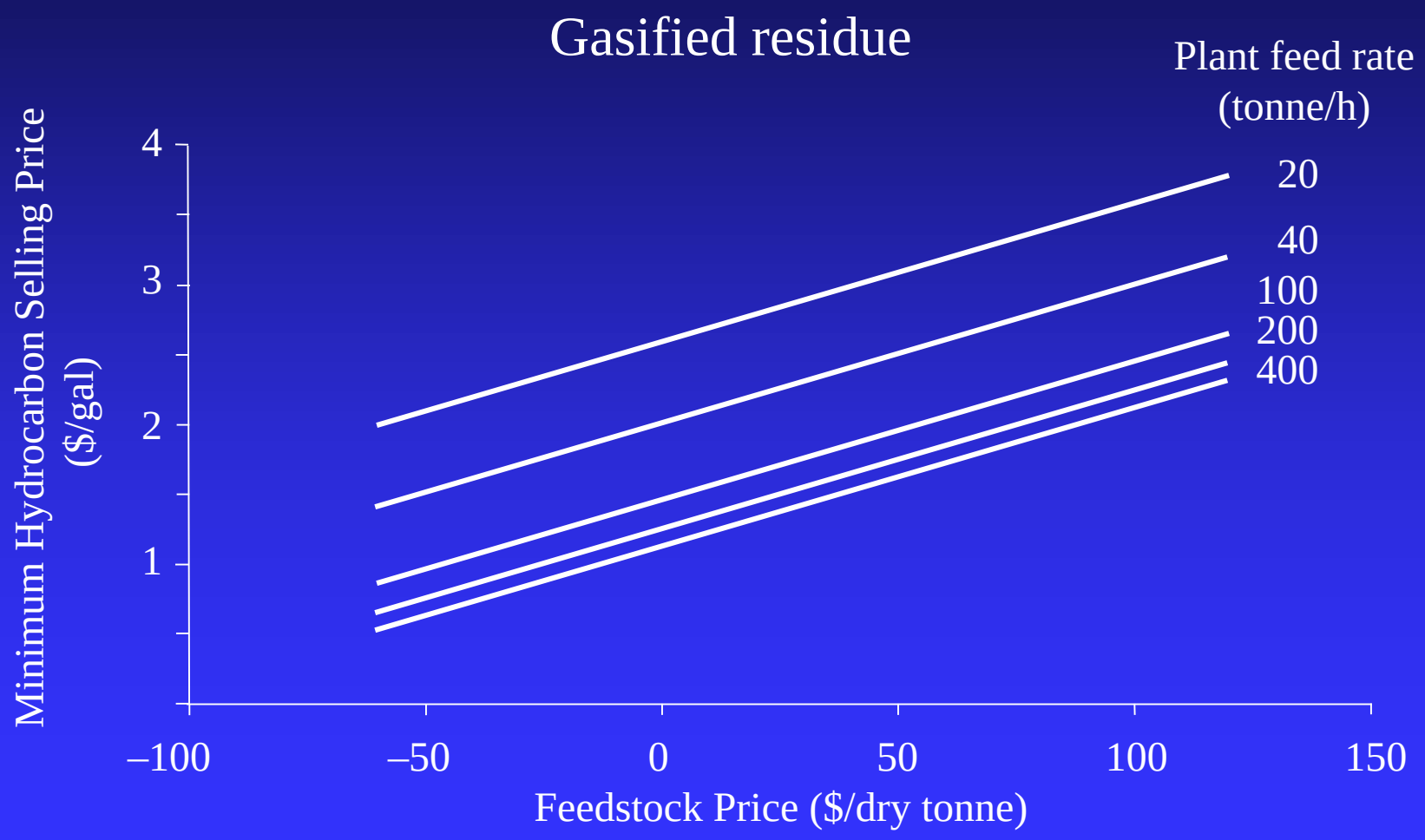
ROI = 15%



http://www.nrel.gov/docs/fy05osti/38210.pdf

Sensitivity to Feedstock Price

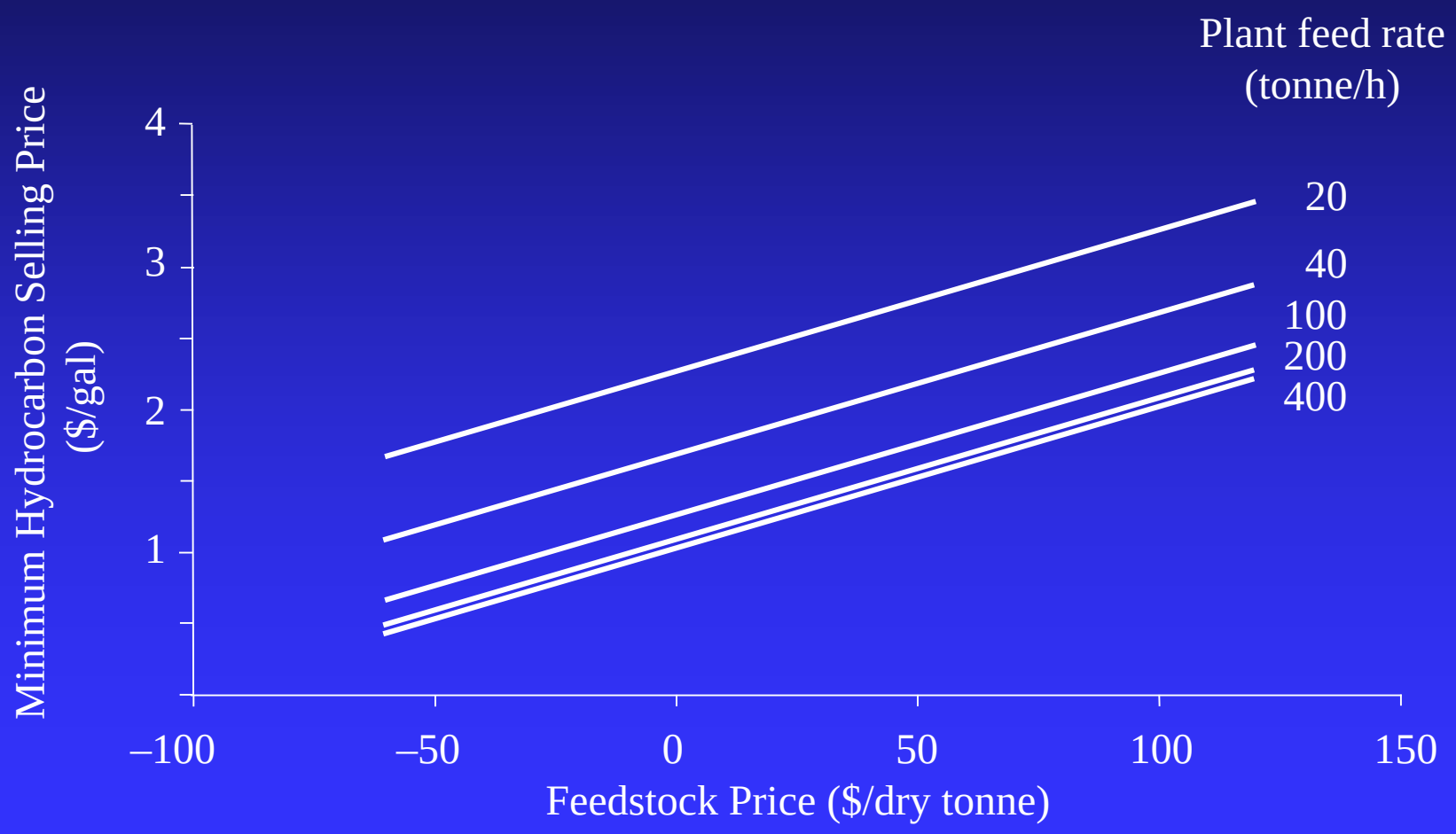
Discount rate = 10% (after tax) Internal hydrogen



Note: Negative feedstock prices mean tipping fees for MSW

Sensitivity to Feedstock Price

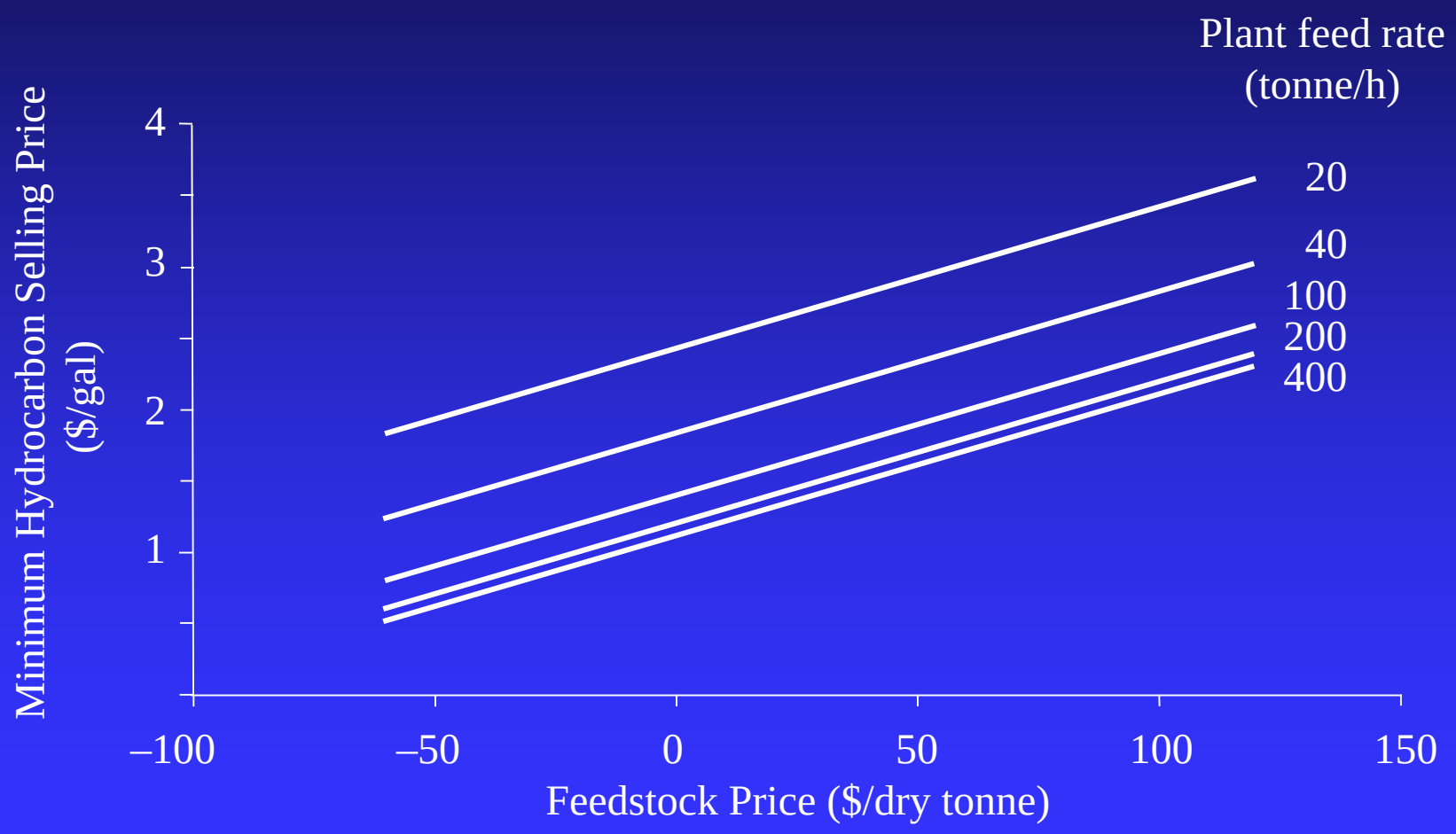
Discount rate = 10% (after tax) External hydrogen = $1/kg



Note: Negative feedstock prices mean tipping fees for MSW

Sensitivity to Feedstock Price

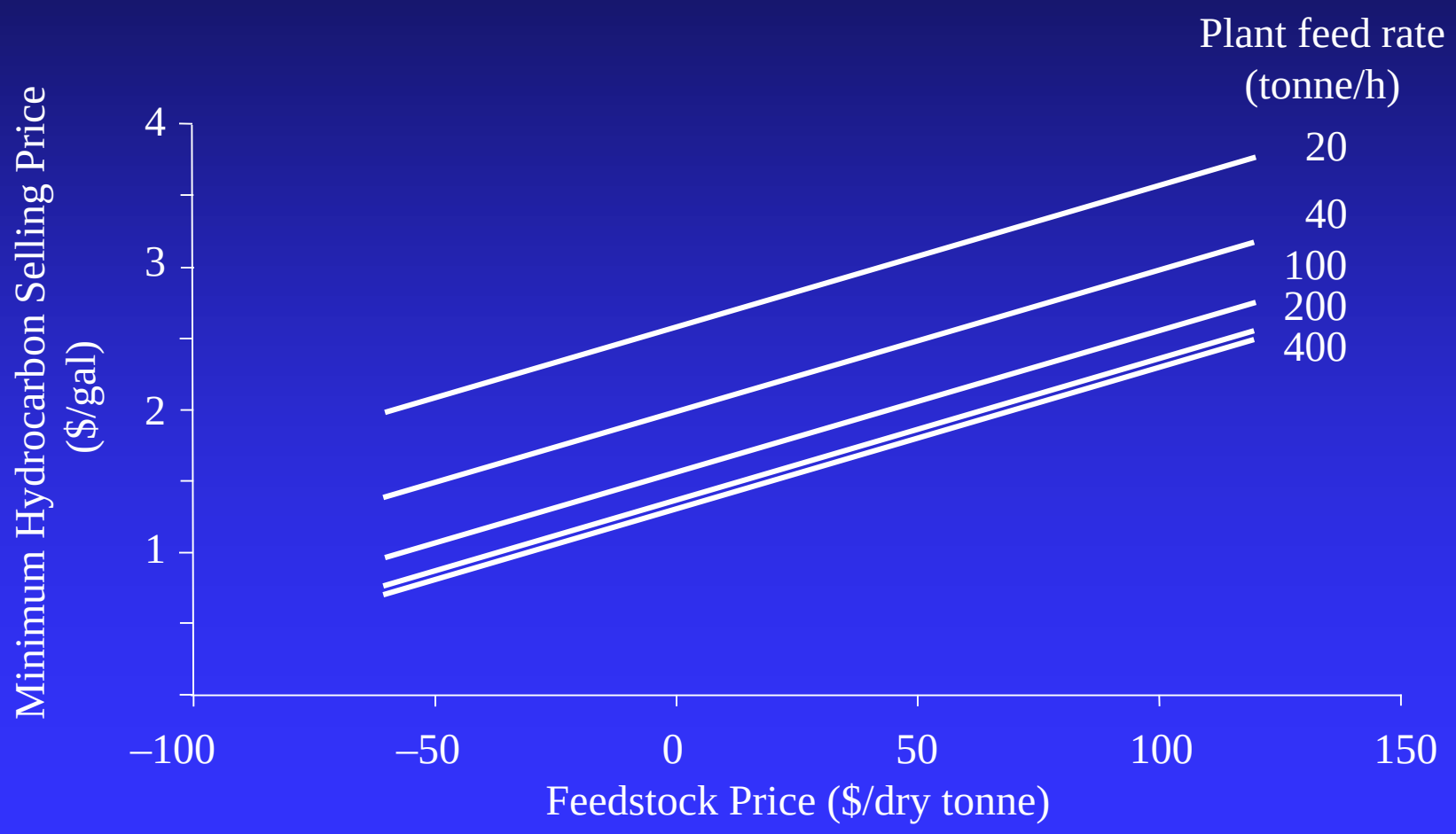
Discount rate = 10% (after tax) External hydrogen = $2/kg



Note: Negative feedstock prices mean tipping fees for MSW

Sensitivity to Feedstock Price

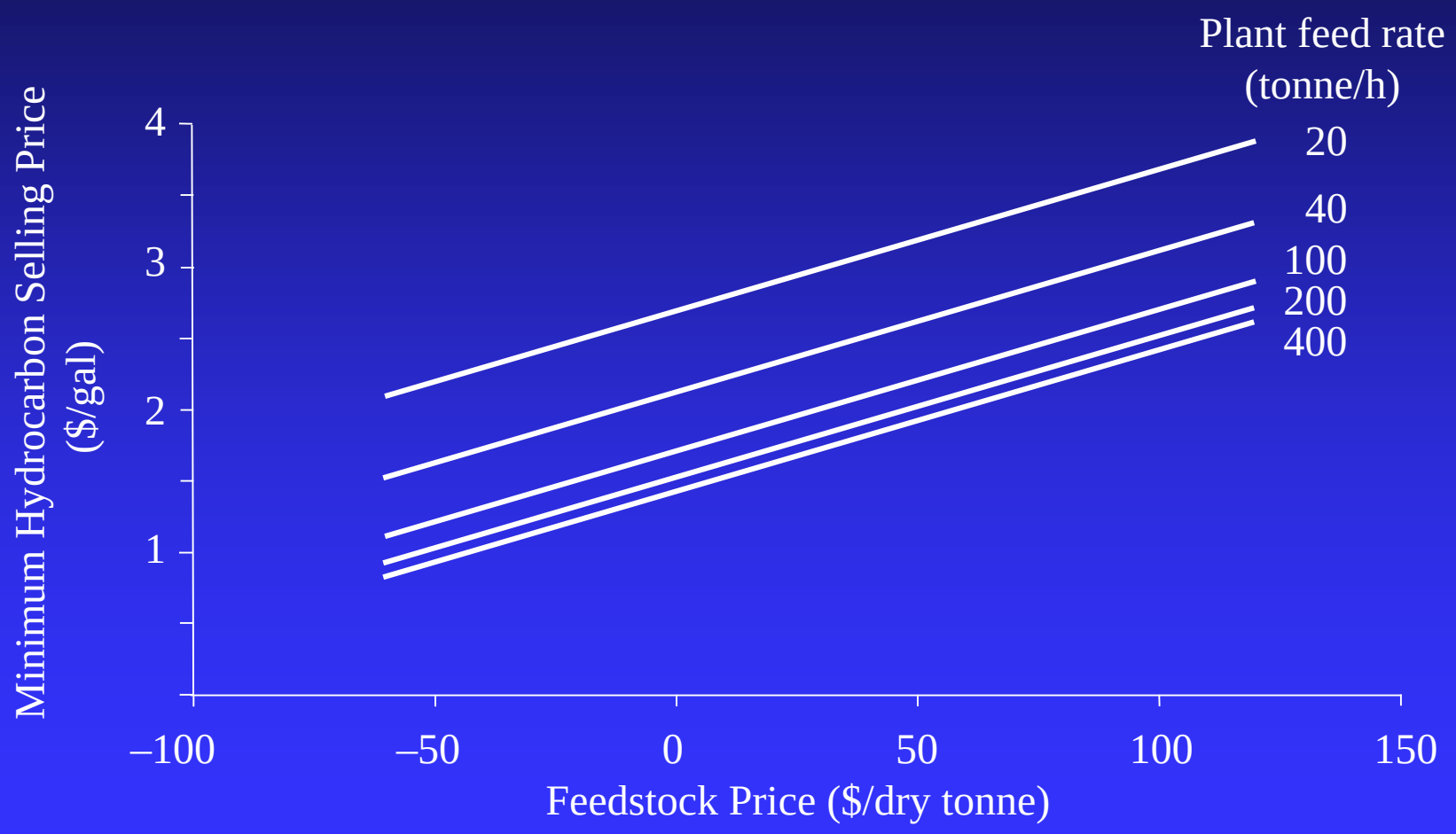
Discount rate = 10% (after tax) External hydrogen = $3/kg



Note: Negative feedstock prices mean tipping fees for MSW

Sensitivity to Feedstock Price

Discount rate = 10% (after tax) External hydrogen = $4/kg



Note: Negative feedstock prices mean tipping fees for MSW

Outline

- Biomass processing
- Carboxylate platform (MixAlco)
  - Primary alcohol route
  - Secondary alcohol route
- Economics
- History
- Conclusions

Brief History of MixAlco Process

1991 – Laboratory research began

2000 – Pilot plant construction began

2008 – Demonstration plant construction begins

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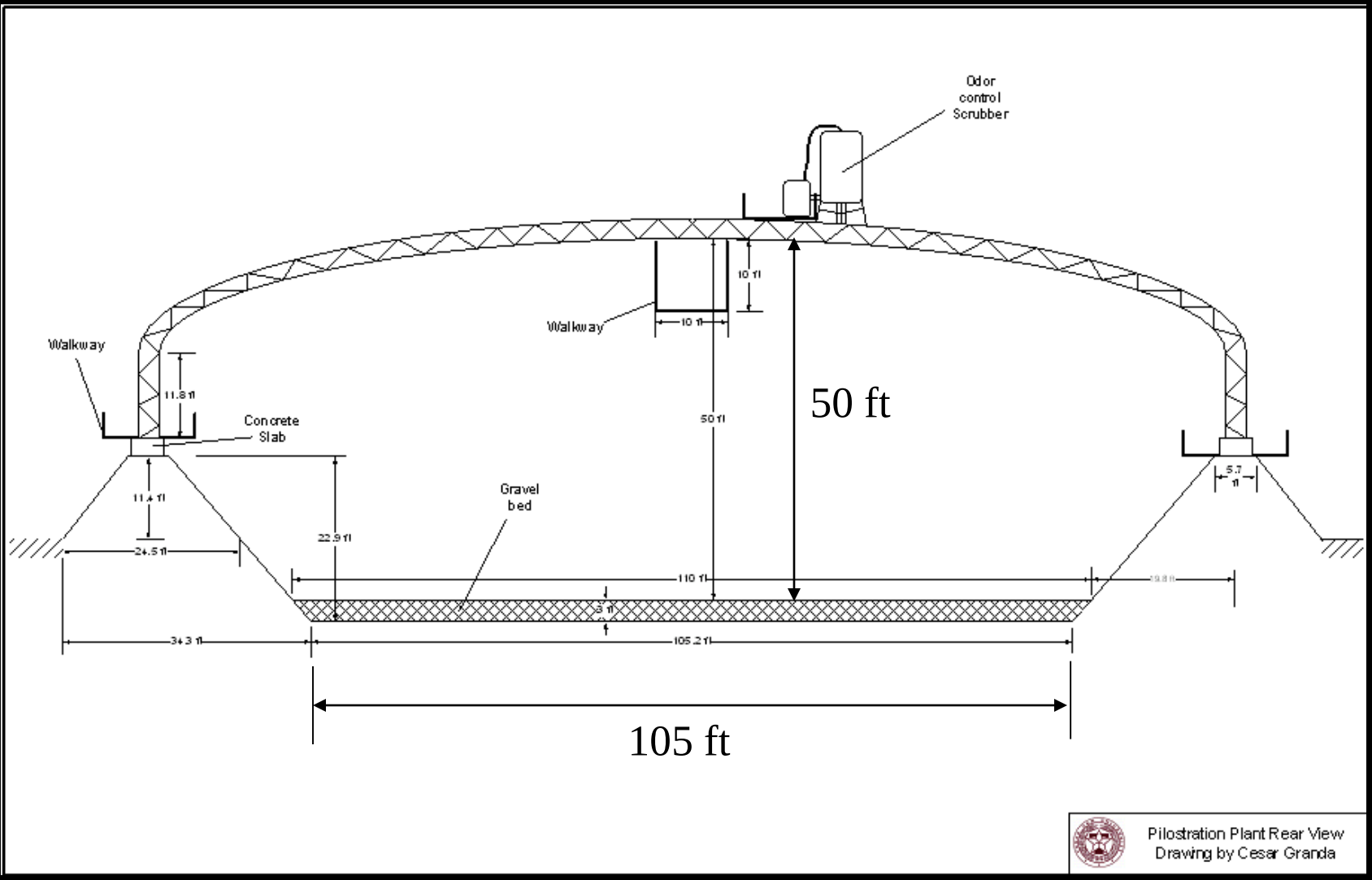
Fermentor Structure



Construction has begun!!

Construction

Construction

Construction

Construction

Construction

Construction

Fermentor Interior

Completed Fermentor

Giant Cattle Rumen

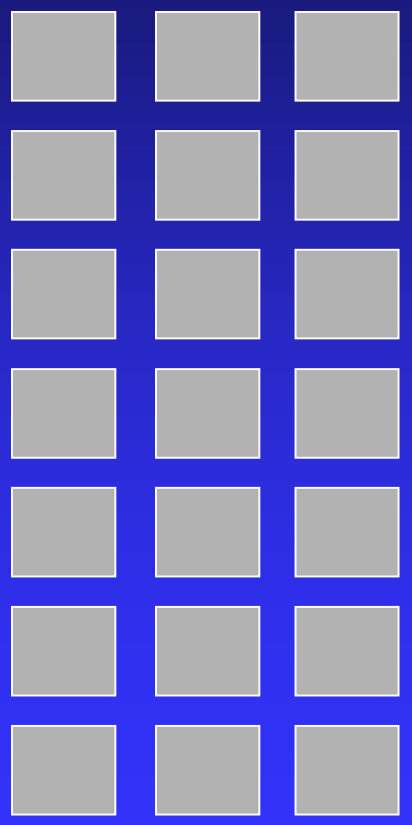
Demonstration Plant

Demonstration Plant

Demonstration Plant

DARPA Project – Phase 1

Start



21 organizations

Meet milestones

End

Texas A&M

Terrabon

GE

Carboxylate platform
(100 L jet fuel)

DARPA Project – Phase 2

Start

End

Terrabon

2 organizations

Carboxylate platform
(6000 L jet fuel)

Outline

- Carboxylate platform (MixAlco)
  - Primary alcohol route
  - Secondary alcohol route
  - Kolbe electrolysis
- Economics
- History
- Conclusions
- Future improvements

Hydrogen Requirements

Percentage of energy from hydrogen

	Fermentor Temperature	
	40°C	55°C
Kolbe Electrolysis	0%	0%
Secondary Alcohols	8.9%	12.3%
Primary Alcohols	27.5%	37.2%

Hydrogen Requirements

Percentage of energy from hydrogen

Fermentor Temperature

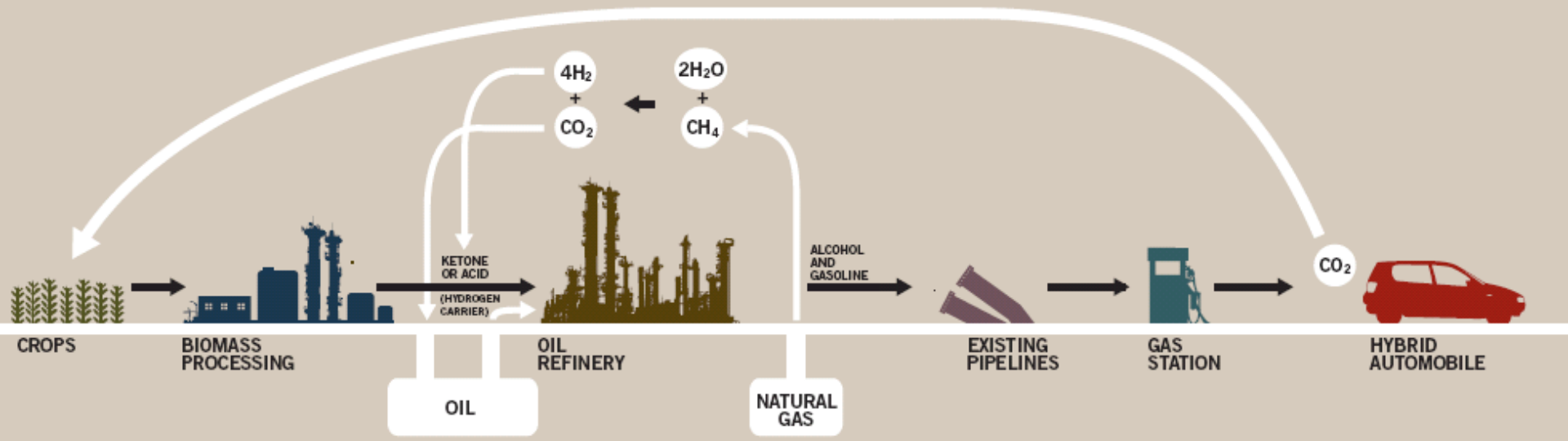
	40°C	55°C
Kolbe Electrolysis	0%	0%
Secondary Alcohols	8.9%	12.3%
Primary Alcohols	27.5%	37.2%

Improved carbon efficiency

Conclusions

Attractive economics

Capacity (tonne/h)	Biomass Cost ($/tonne)	Hydrogen	Minimum Selling Price ($/gal)	
			Hydrocarbon	Ethanol Equiv.
40	60	Internal	2.56	1.74
400	60	Internal	1.76	1.20
40	–45	$1/kg	1.24	0.84



The Grand Vision

Outline

- Carboxylate platform (MixAlco)
  - Primary alcohol route
  - Secondary alcohol route
  - Kolbe electrolysis
- Economics
- History
- Conclusions
- Future improvements

Future Improvements

- Improve rates
  - Catle feed rate = 250 g/(L·d)
  - MixAlco feed rate = 4 to 10 g/(L·d)
- Exploit bioprospecting
- Enhance chain elongation
- Explore round-robin fermentation
- Develop extraction processes
- Develop fermentation-compatible pretreatments
  - Replace calcium cation

The MixAlco process is simple

Mixed-culture fermentation is complex