# DIRECT LIQUID-LIQUID LIPID EXTRACTION METHOD FOR BIODIESEL PRODUCTION FROM SEWAGE AND PETROCHEMICAL INDUSTRY SLUDGES


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# Outline

- Aim of the Study
- Methodology
- Material and Methods
- Results and Conclusions

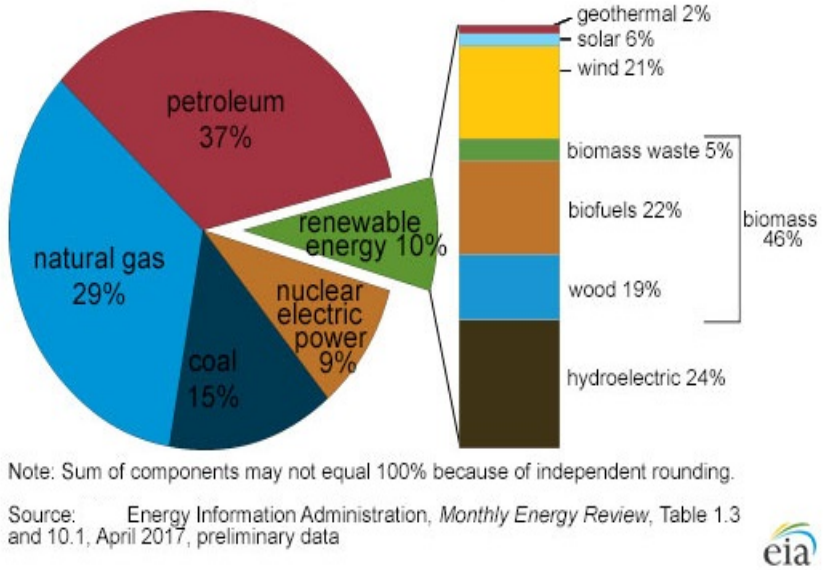
# Aim of this Study

- to explore lipid extraction from sewage and petrochemical industry WWTP sludges by using the novel **direct liquid-liquid extraction method**, which does not require expensive sludge dewatering/drying steps;

- to compare liquid-liquid method to standard reference drying method in terms of lipid and biodiesel yields

- to investigate the effect of acid pre-treatment on lipid yields

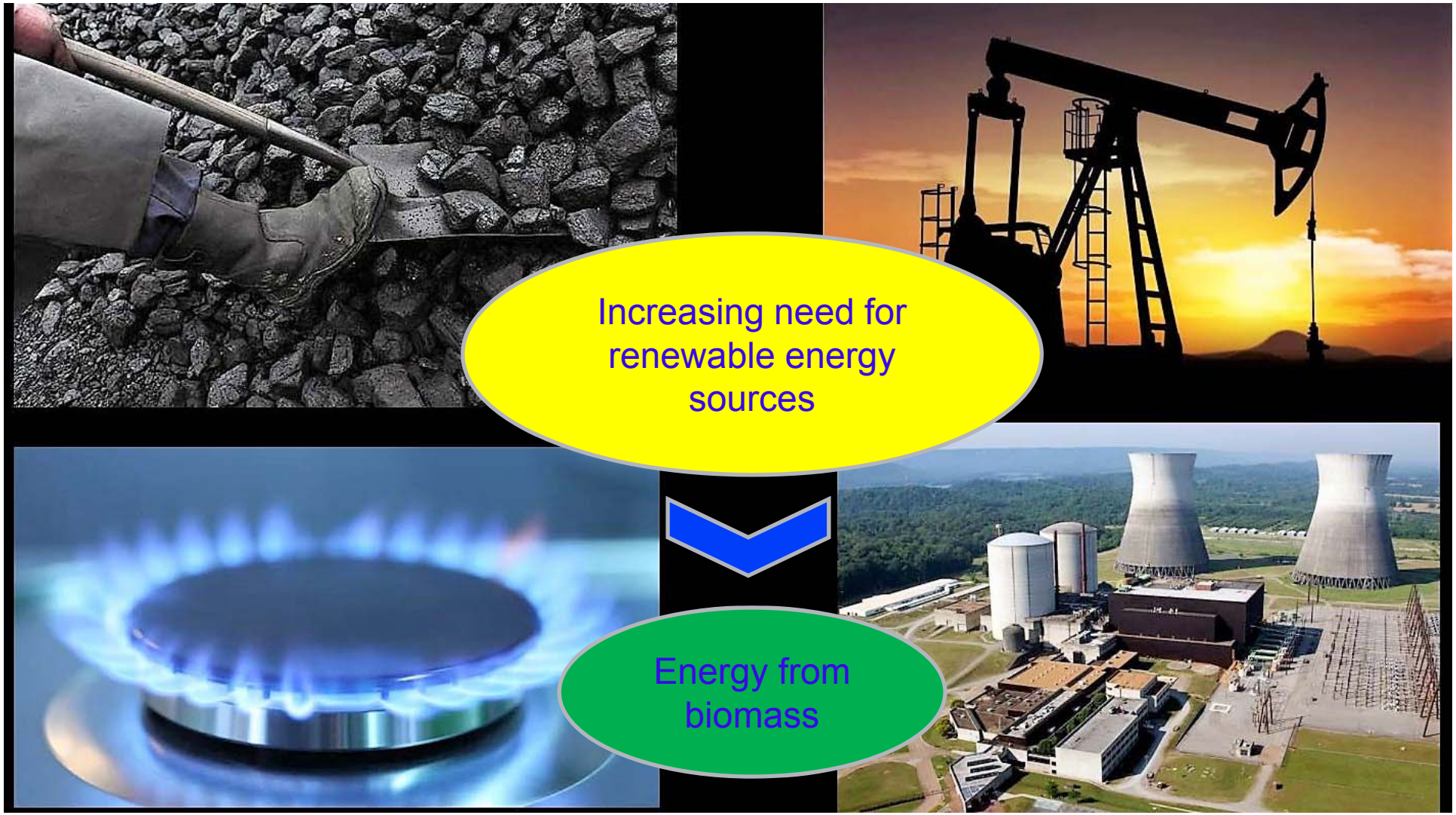
# World's Energy Scenario




- According to the International Energy Agency, the world will need 37% more energy in 2040 than today.

- As shown in figure, currently 81% of all energy consumed worldwide is from fossil fuel sources.

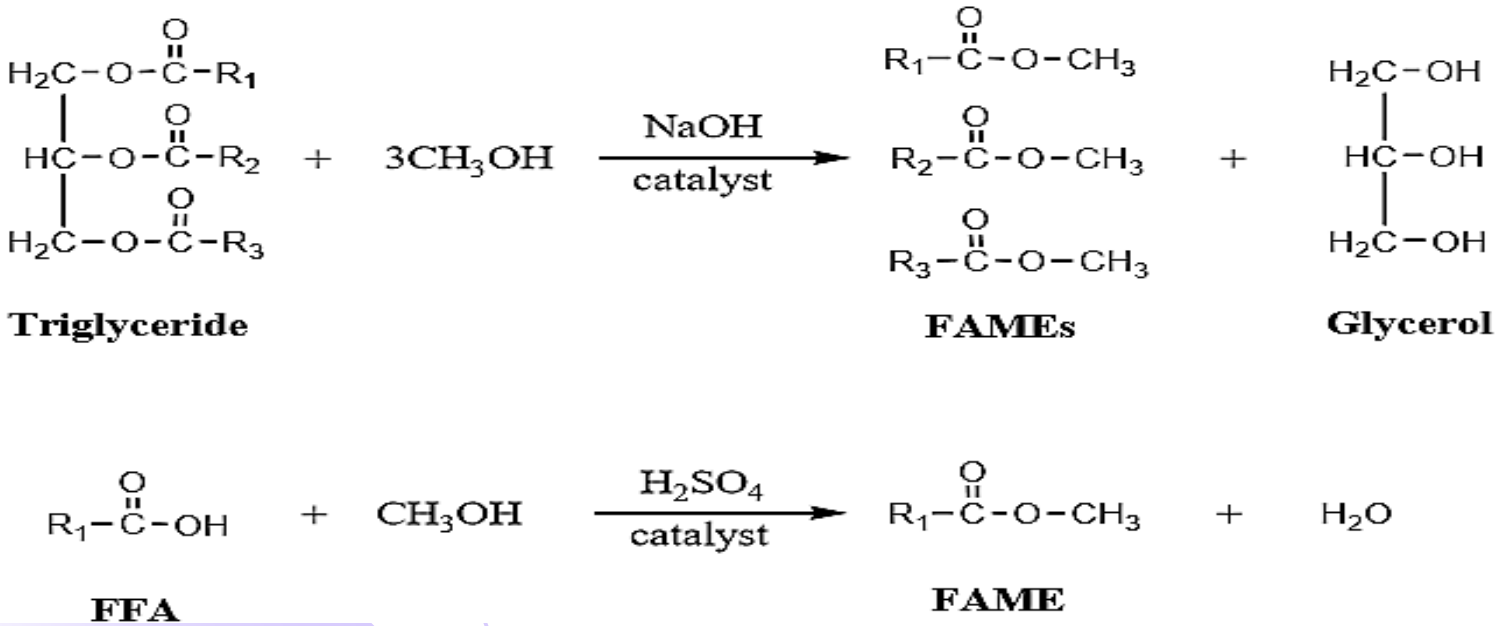
# The fossil fuel sources will be depleted in near future.



# What is Biodiesel?

- Biodiesel is an alternative fuel that may be derived from a variety of feedstock.

- Commonly produced by transesterification of pre-extracted oils with an alcohol in the presence of a catalyst to generate the fatty acid methyl esters (FAMEs).



# Benefits of Biodiesel

**Biodiesel**;

- o is renewable
- o is biodegradable
- o is non-toxic
- o is safe for storage and handling
- o increases lubricity of fuel
- o reduces global warming gases
- o has lower emission profiles

# Biodiesel Feedstocks

- ✓ Edible plant oils
  - rapeseed oil
  - sunflower oil
  - palm oil
  - soybean oil
  - coconut oil

high cost of pure vegetable or seed oils, 70-85% of the overall biodiesel production cost

- ✓ Non-edible plant oils
  - jatropha
  - castor
  - neem
  - karanja

excessive cultivation ⟶ deforestation and destruction of the ecosystem

- ✓ Animal fats
- ✓ Waste cooking oil

low quality ⟶ the biodiesel consistency

- ✓ Microalgae

high cost related with biomass production

- ✓ **Wastewater Sludge**

# Sludge as a Lipid Feedstock

- Municipal Wastewater (sewage) sludge is readily available in large quantities
- Wastewater sludge is cheap or practically costless feedstock generated in WWTPs, therefore the cost of biomass production and land requirement is eliminated.
- Wastewater sludge is non-edible lipid feedstock, therefore the competition with the food market is eliminated.

# MATERIALS AND METHODS

## Feedstocks:

- **Municipal WWTP Sludge**
  - Primary Sludge : from the bottom of the primary clarifier in a WWTP in İstanbul
  - Secondary Sludge : from the bottom of the secondary clarifier in a WWTP in İstanbul

- **Petrochemical Industry WWTP Sludge**
  - from the sludge thickening unit of a petrochemical industry WWTP in Turkey

# Municipal Sewage Sludge

## up to 30 wt.% of lipids

**Primary sludge**;

- is a combination of floating grease and solids
- contains high lipid content originated from the adsorption of lipids, in the form of triglycerides, diglycerides, monoglycerides, phospholipids, and free fatty acids (FFAs)

**Secondary sludge**;

- is composed of microorganisms whose cell membranes contain phospholipids

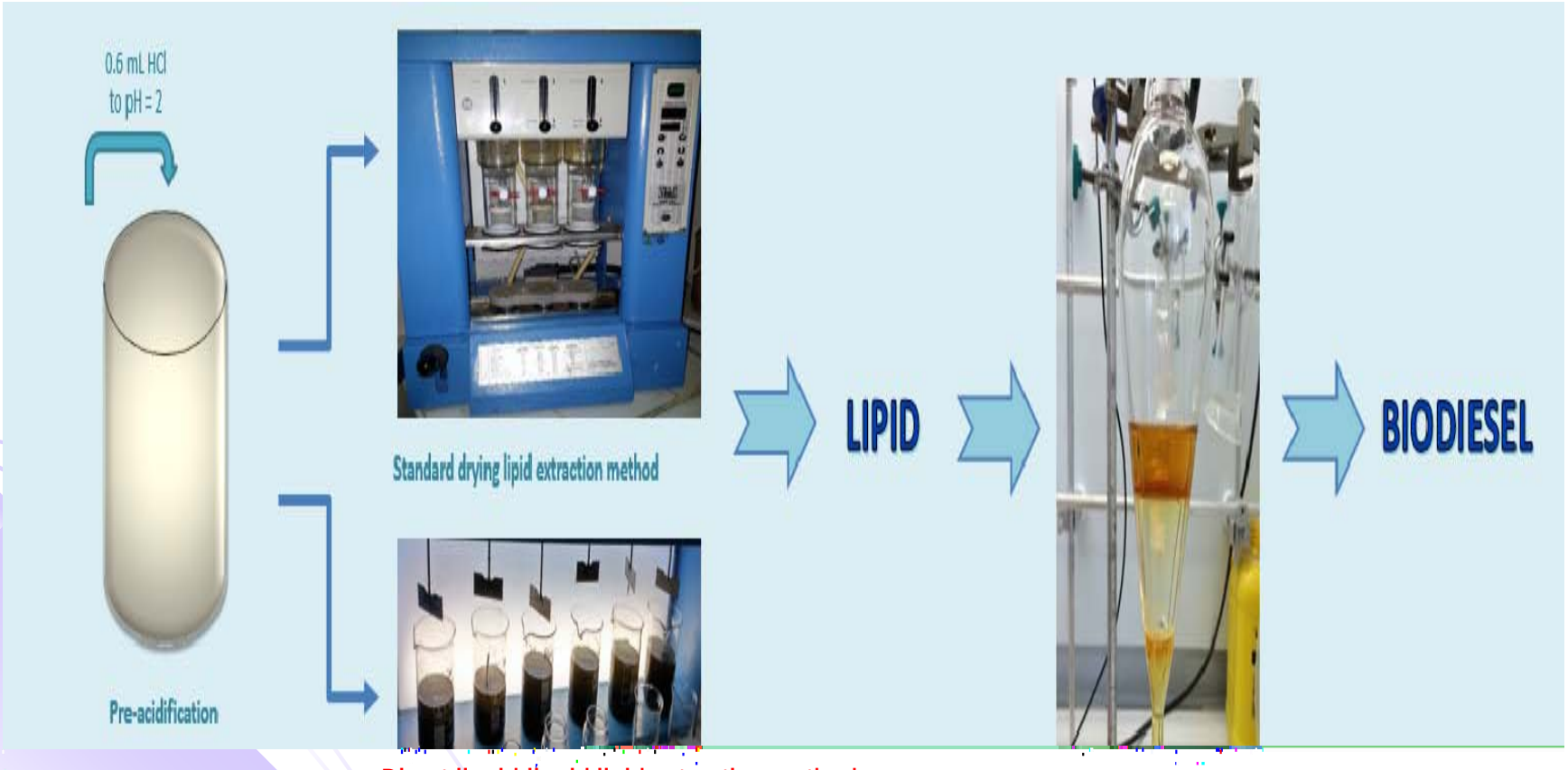
# Petrochemical Industry WWTP Sludge

## up to 40-60 wt.% of lipids

- contains sludges from:
  - oil separators,
  - primary clarifier
  - secondary clarifier (waste activated sludge)

- comprises of various organic and inorganic compounds:
  - suspended solids, salts, water-soluble metals
  - phospholipids,
  - petroleum hydrocarbons (PHCs),
  - neutral lipids and FFAs (sourced from waste activated sludge and the oils that cannot be removed in WWTP)

# Scheme of biodiesel production process



# Standard Drying Lipid Extraction Method

Sludge samples

↓

Acid pre-treatment (HCl)

↓

Drying with $MgSO_4.H_2O$

↓

Solvent addition (hexane)

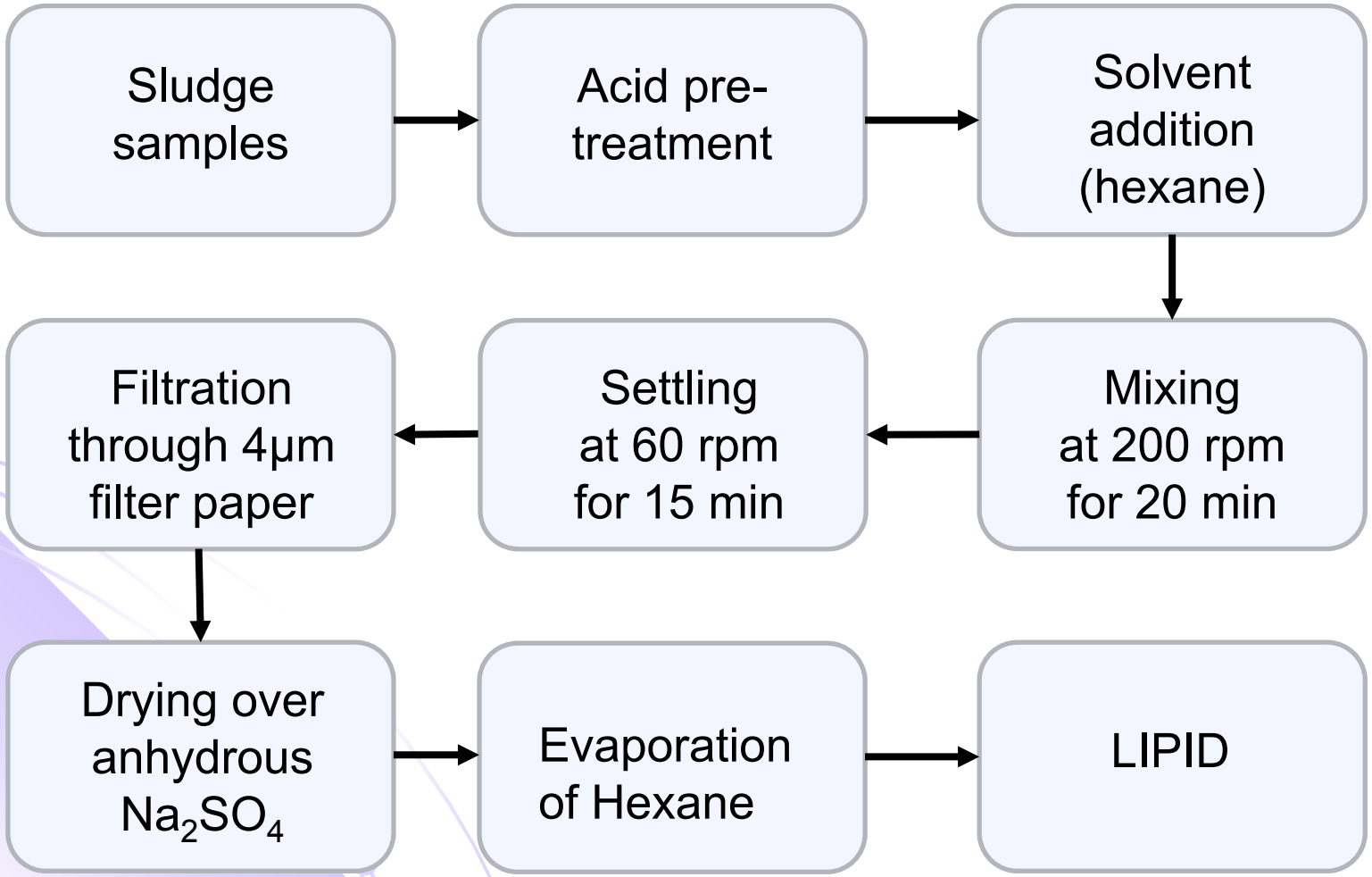
↓

Soxhlet extraction

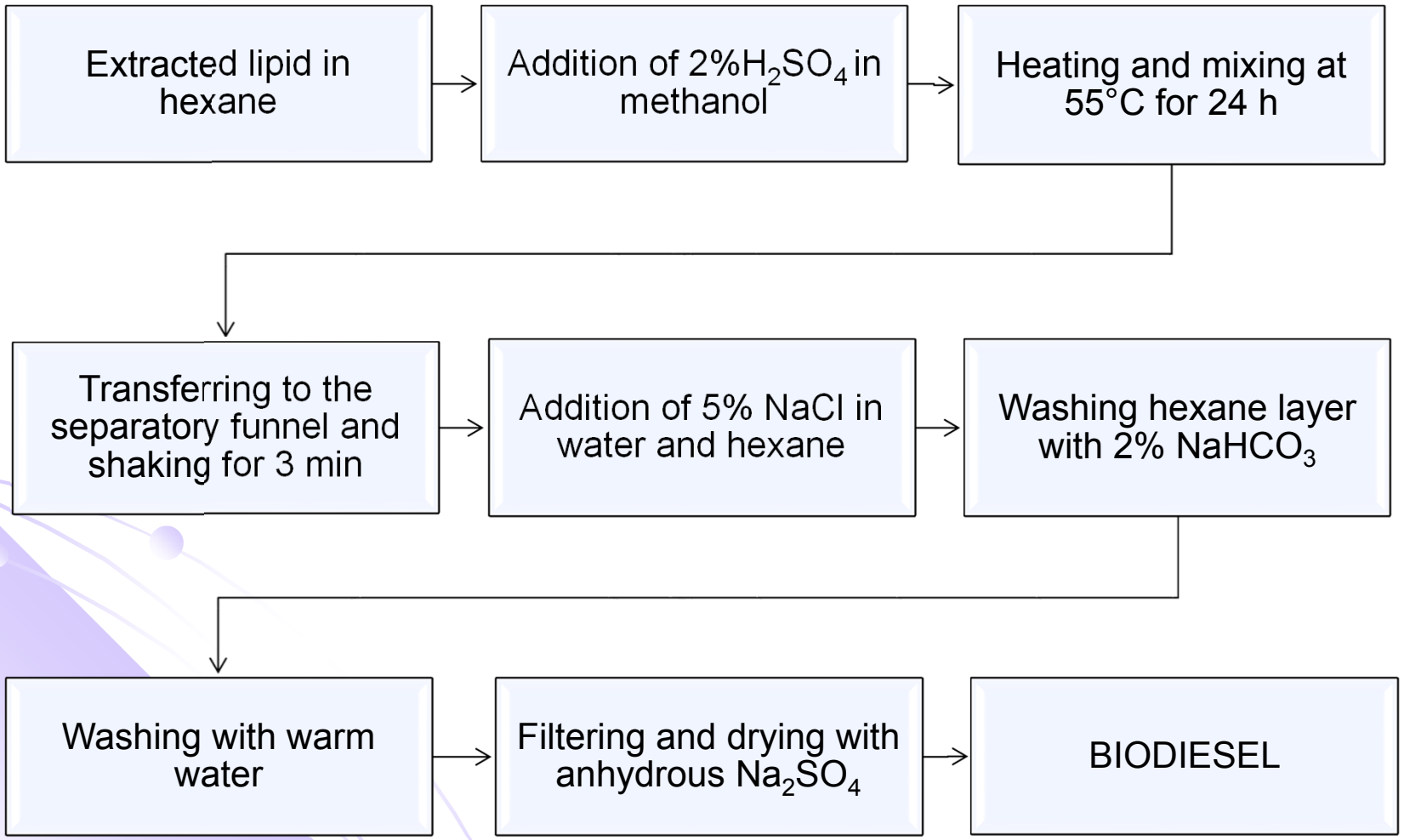
↓

LIPID

# Direct Liquid-Liquid Lipid Extraction Extraction Method



# Biodiesel Production



# RESULTS AND DISCUSSION

# Sludge Characteristics

| Parameter | Unit | Primary Sewage Sludge | Secondary Sewage Sludge | Petrochemical Industry WWTP sludge |
|---|---|---|---|---|
| TS | % | 4.2 | 1.3 | 3.4 |
| VS | % | 2.4 | 0.78 | 2.07 |
| COD | mg/L | 40280 | 8780 | 63220 |
| sCOD | mg/L | 3090 | 2240 | 10800 |
| pH | - | 6.2 | 6.1 | 6.8 |
| Viscosity | mPa.s | 8 | 6.4 | 29.2 |

# Effect of Solvent Type

| | LIPID YIELD (%) | | | |
|---|---|---|---|---|
| | Liquid-Liquid Method | | Drying Method | |
| **SOLVENT** | Primary sewage sludge | Secondary sewage sludge | Primary sewage sludge | Secondary sewage sludge |
| **Hexane** | 19.4 | 2.7 | 24.0 | 7.2 |
| **Petroleum ether** | 13.4 | 2.3 | 23.8 | 11.9 |
| **Chloroform** | 12.3 | 1.3 | 23.1 | 10.0 |
| **Toluene** | 11.3 | 1.3 | 22.9 | 10.3 |

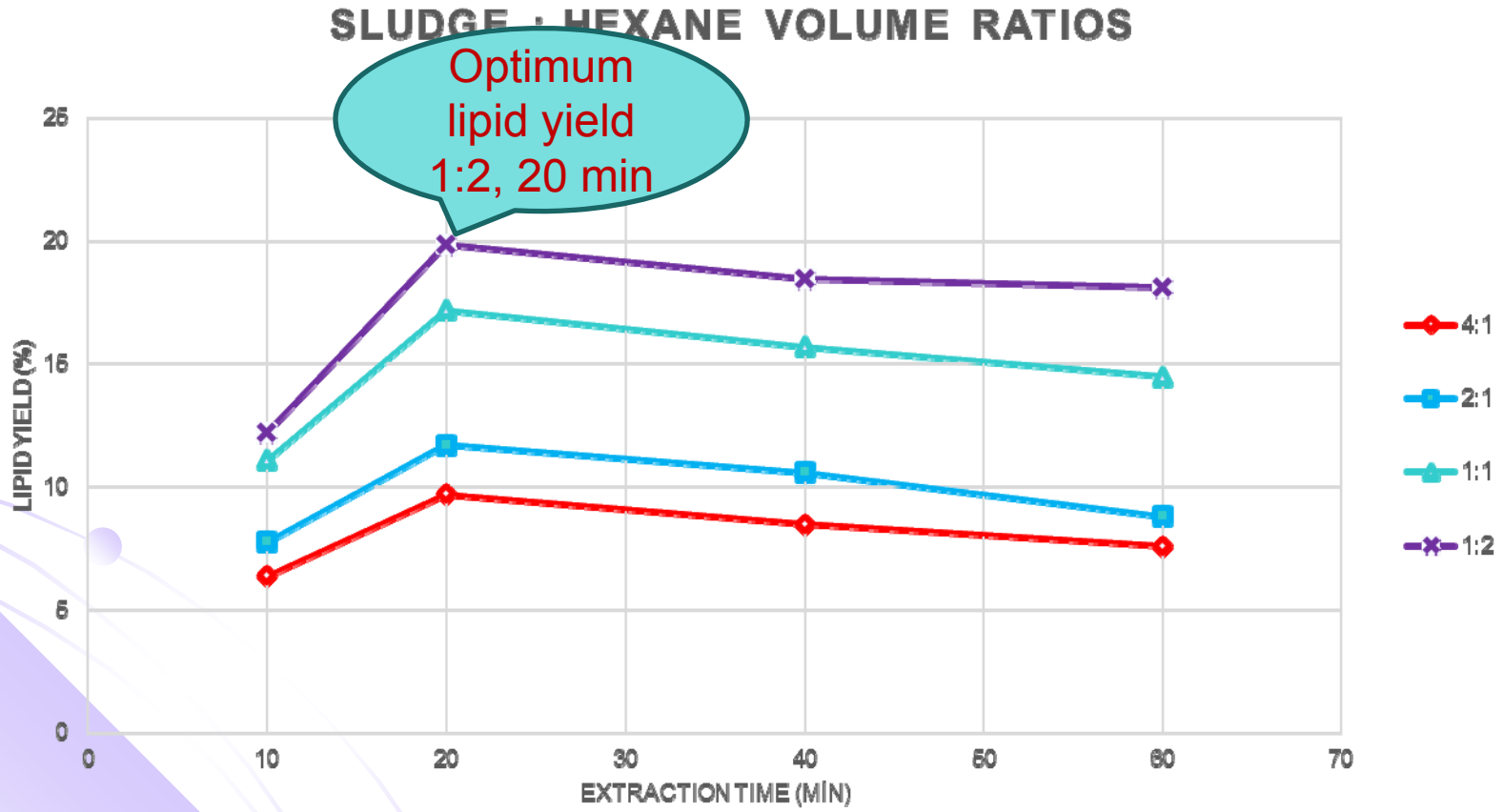
# Efficiency of Feedstocks

| Sludge type | Extraction method (Sludge:Hexane = 1:2) | Lipid yield (%)[a] | |
|---|---|---|---|
| | | Acidified | Non-acidified |
| Primary Sewage Sludge | Standard drying[b] | 24 | 22.8 |
| | Direct liquid-liquid extraction | 19.7 | 10.5 |
| Secondary Sewage Sludge | Standard drying[b] | 7.2 | 6.5 |
| | Direct liquid-liquid extraction | 2.9 | 1.6 |
| Petrochemical Industry WWTP Sludge | Standard drying[b] | 20.3 | 18.2 |
| | Direct liquid-liquid extraction | 31.2 | 27.3 |

[a] Each value is the average of at least 3 samples collected on different days.

[b] Extraction according to standard $MgSO_4.H_2O$ method, lipid yield on the basis of dry sludge.

# Optimization of the Liquid-Liquid Lipid Extraction Method for Primary Sludge



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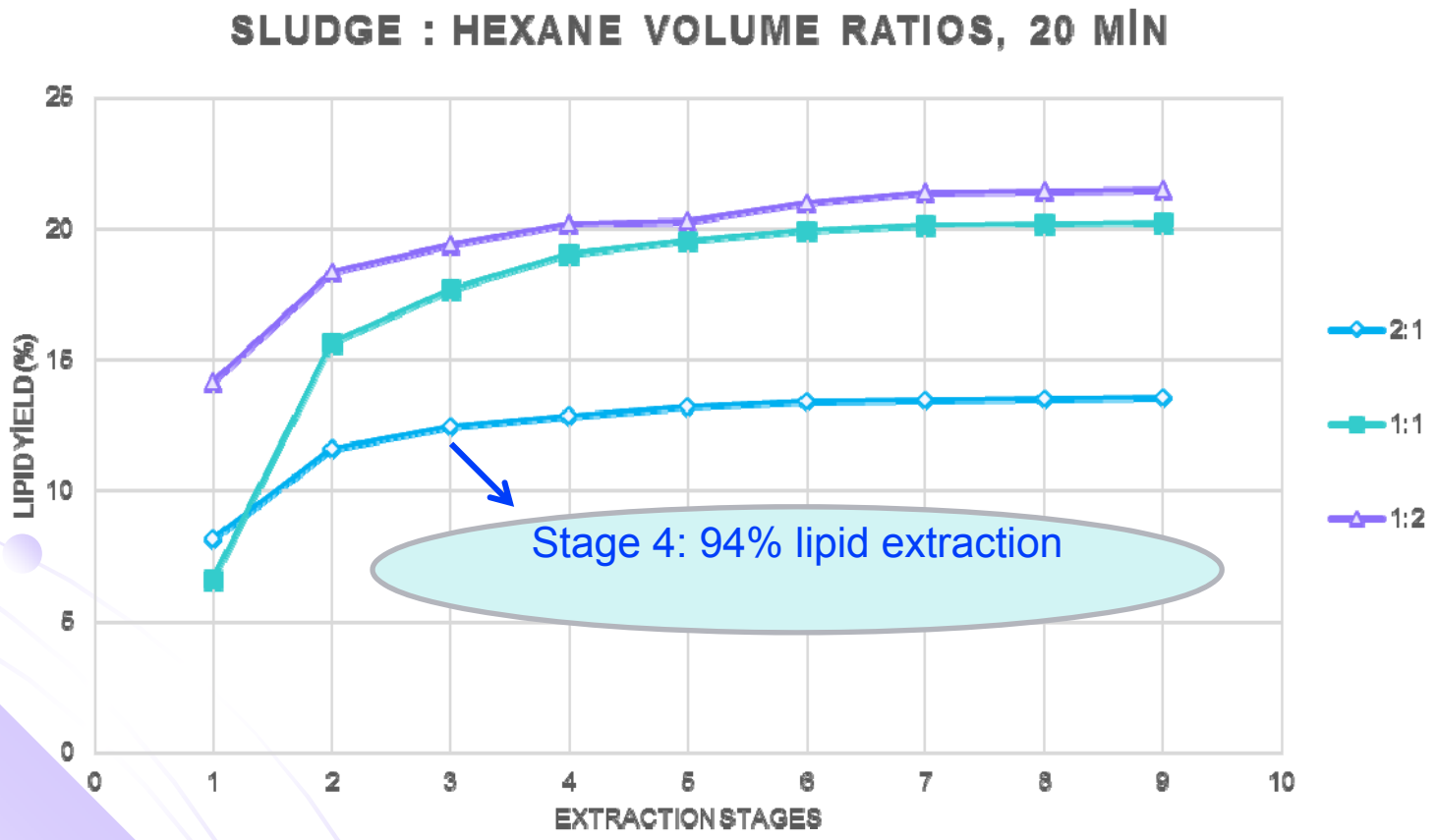
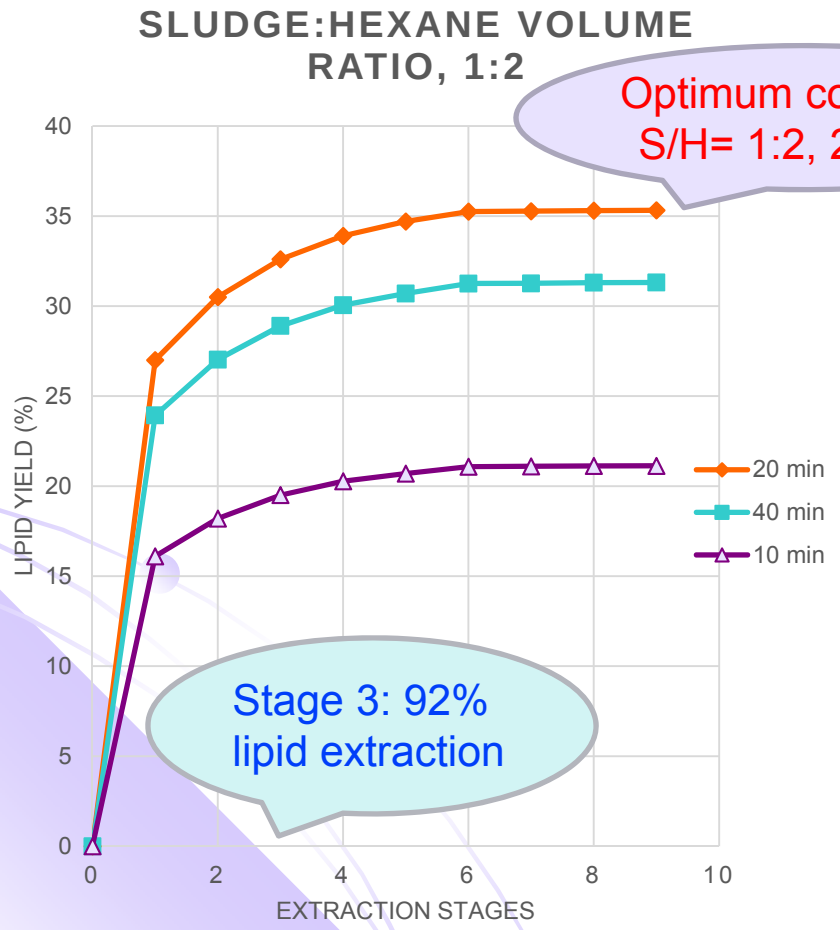
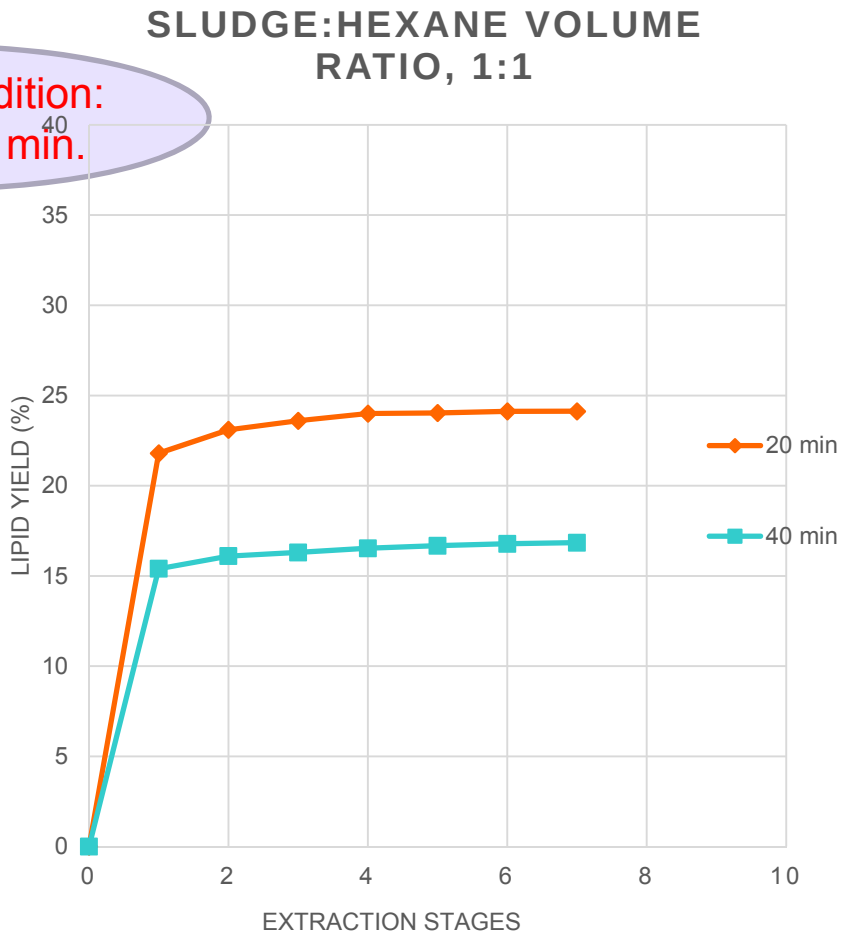


# Optimization of the Liquid-Liquid Lipid Extraction Method for Petrochemical Industry WWTP Sludge



# Biodiesel Production Yields

| Sludge type | Extraction method | Yield (%)[a] | Non Pretreated. | Acid Pretreated |
|---|---|---|---|---|
| Primary sewage sludge | Standard drying[b] | Lipid | 22.8 | 24.0 |
| | | Biodiesel | 12.5 | 14.5 |
| | Direct liquid-liquid extraction | Lipid | | |
| | | Biodiesel | 5.3 | 14.7 |
| Petrochemical Industry WWTP sludge | Standard drying[b] | Lipid | 18.2 | 20.3 |
| | | Bi | | 14.1 |
| | Direct liquid-liquid extraction | | | 31.2 |
| | | Biodiesel | 18.8 | 22.2 |

180% increase with pretreatment

57% increase with direct liquid-liquid extraction

[a] All transesterification experiments were performed at least twice.
[b] Extraction according to standard $MgSO_4.H_2O$ method, lipid yield on the basis of dry sludge.

# CONCLUSIONS

- Oily sludges from petrochemical industry WWTPs and primary sludges are lipid-rich feedstocks for biodiesel production.

  - *Secondary* sewage sludge samples were found to be an inefficient to be used as lipid feedstock for biodiesel production.

# CONCLUSIONS

- Direct liquid-liquid lipid extraction method resulted with higher lipid and biodiesel yields for *petrochemical industry WWTP sludge* samples than that of obtained by standard drying method.

- Almost the same biodiesel yields were achieved for primary sewage sludge by both of the extraction methods.

- Taking into consideration economy of the process, liquid-liquid lipid extraction method may be preferred as it eliminates dewatering/drying steps, contributing to 50% of the whole biodiesel production cost.

# CONCLUSIONS

- Acidification pre-treatment increased the lipid and biodiesel yields.

- Biodiesel production from sludge is an alternative reuse/disposal method, decreasing the amount of WWTP sludges, requiring high treatment and handling costs.

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**THANK YOU ☺**