EFFECT OF ACID AND ULTRASONICATION PRE-TREATMENTS ON THE LIPID EXTRACTION FROM PETROCHEMICAL INDUSTRY AND MUNICIPAL WASTEWATER SLUDGES

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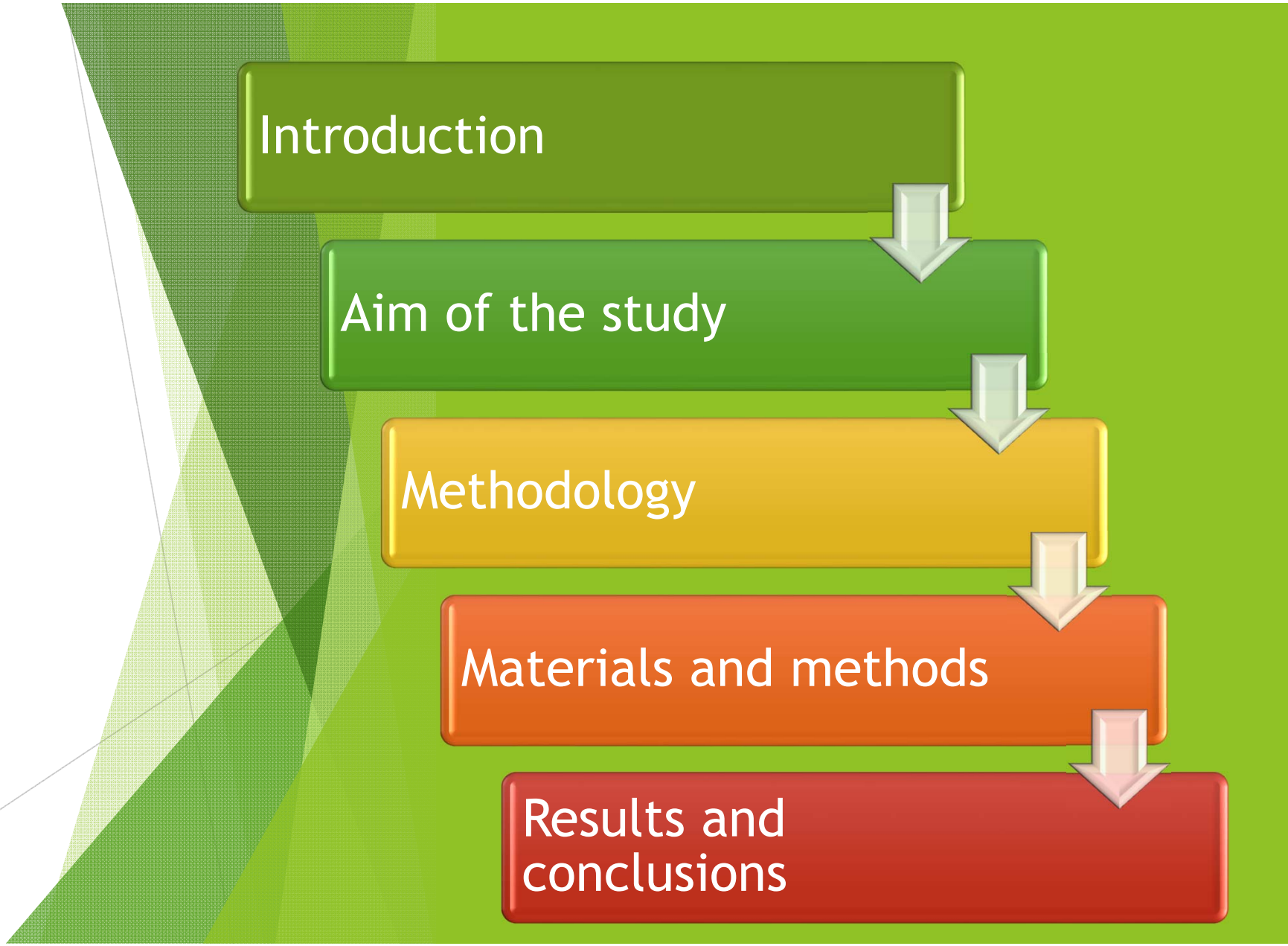
Outline


Introduction
Aim of the study
Methodology
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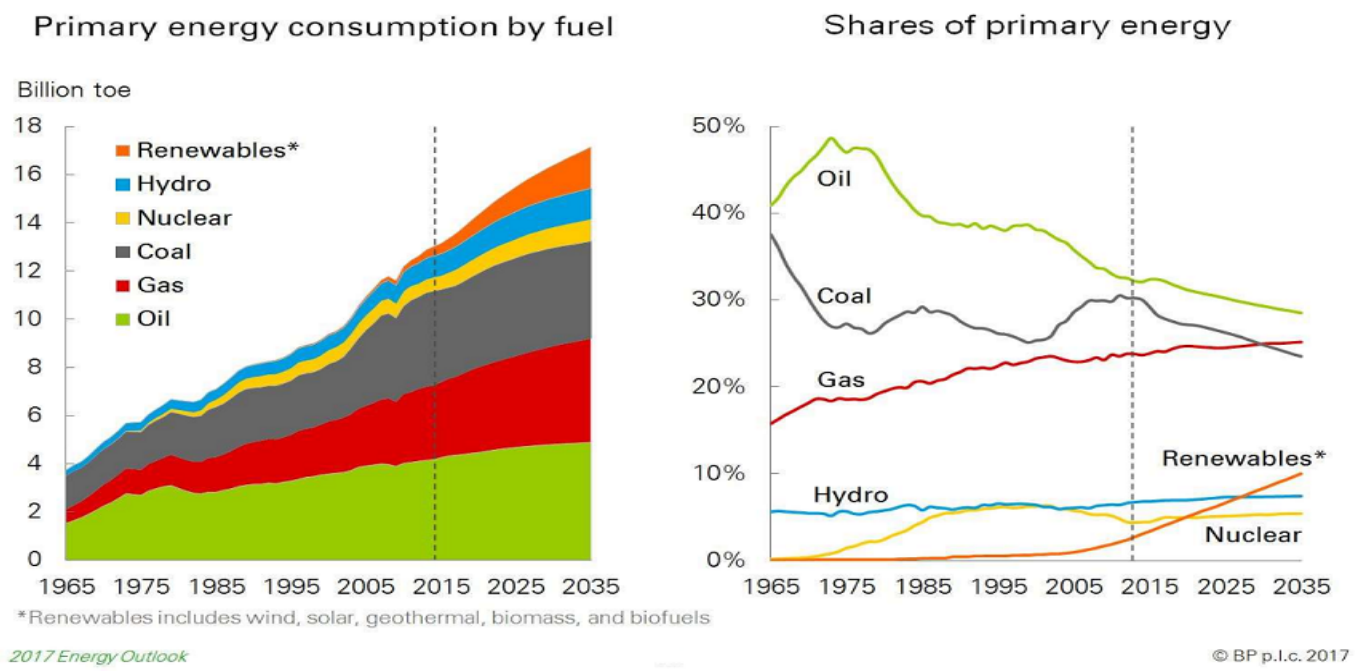
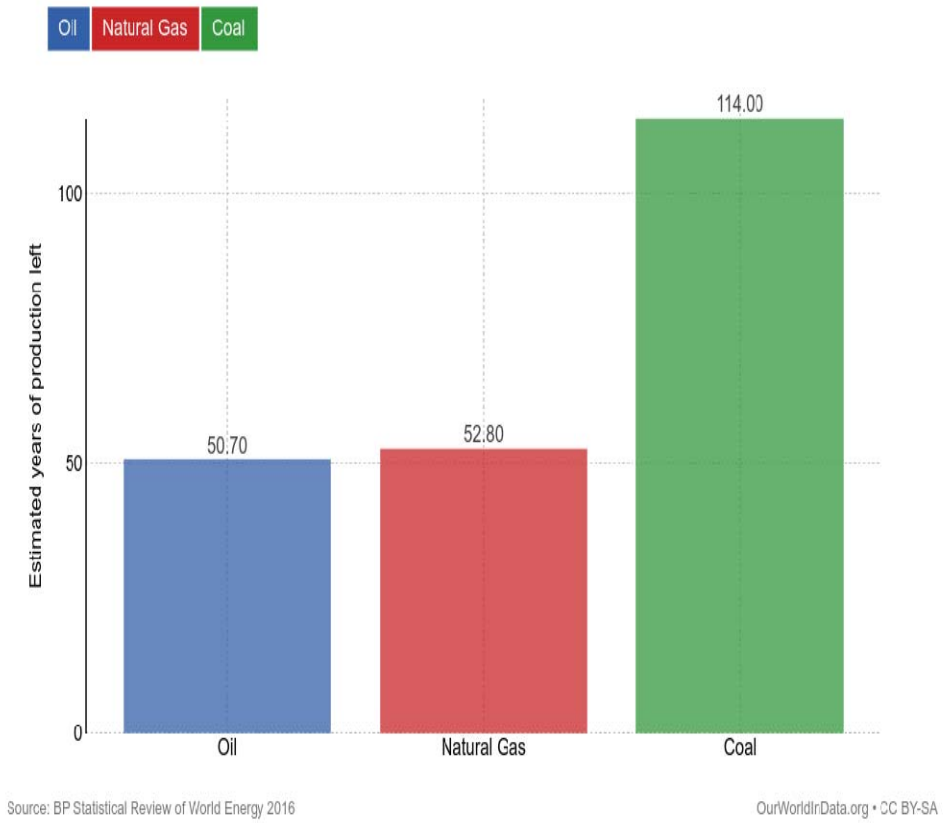
Energy Scenario in the World



- Currently 81% of all energy consumed worldwide is from fossil fuel sources.
- Renewable energy sources are expected to be 16.1% of world energy consumption in 2040.

Energy Scenario in the World



- Fossil fuel reserves are rapidly declining, especially oil and natural gas reserves approach critical levels.

The fossil fuel sources will be depleted in near future.

Increasing need for renewable energy sources

Energy from BIOMASS

What is Biodiesel?

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

Lipid extraction is the first step of biodiesel production.

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)

Lipid 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

Lipid Feedstocks

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

excessive cultivation

⬇

deforestation and destruction of the ecosystem

Lipid Feedstocks

- ✓ Animal fats
- ✓ Waste cooking oil

low quality ⇒ the biodiesel consistency

- ✓ Microalgae ⇒ high cost related with biomass production

✓ **Wastewater Sludge**

in Turkey

Sludge production ≈ 299 ktons in 2017

Expected to ⟶ 911 ktons in 2040

Sludge as a Lipid Feedstock

- Wastewater 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.

Sludge as a Lipid Feedstock

- **Municipal (Domestic) wastewater sludge** → **lipids up to 30 % (by wt)**
- **Petrochemical WWTP sludge** → **lipids up to 40-60 % (by wt)**

Aim of this Study

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

to investigate the effect of acid and ultrasonic pre-treatments on the lipid yields

to compare direct liquid-liquid lipid extraction method to standard reference drying method

Materials and Methods

Sludge Samples:

- **Municipal (Domestic) Wastewater Sludge**
  - Primary Sludge : from the bottom of the primary clarifier in a biological wastewater treatment plant in İstanbul

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

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,
  - free fatty acids (FFAs)

Petrochemical Industry WWTP Sludge

- contains sludges from oil separators, primary clarifier, and the waste activated sludge from secondary clarifier

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

Sludge Characteristics

Parameter	Unit	Primary sewage sludge	Petrochemical industry WWTP sludge
TS	%	4.2	3.4
VS	%	2.4	2.1
COD	mg/L	40280	63220
sCOD	mg/L	3090	10800
Viscosity	mPa.s	8.0	52.8
pH	-	6.2	6.8

Pre-treatments

Acid Pre-treatment

- 0.1N HCl application to reach pH:2

Ultrasonication Pre-treatment

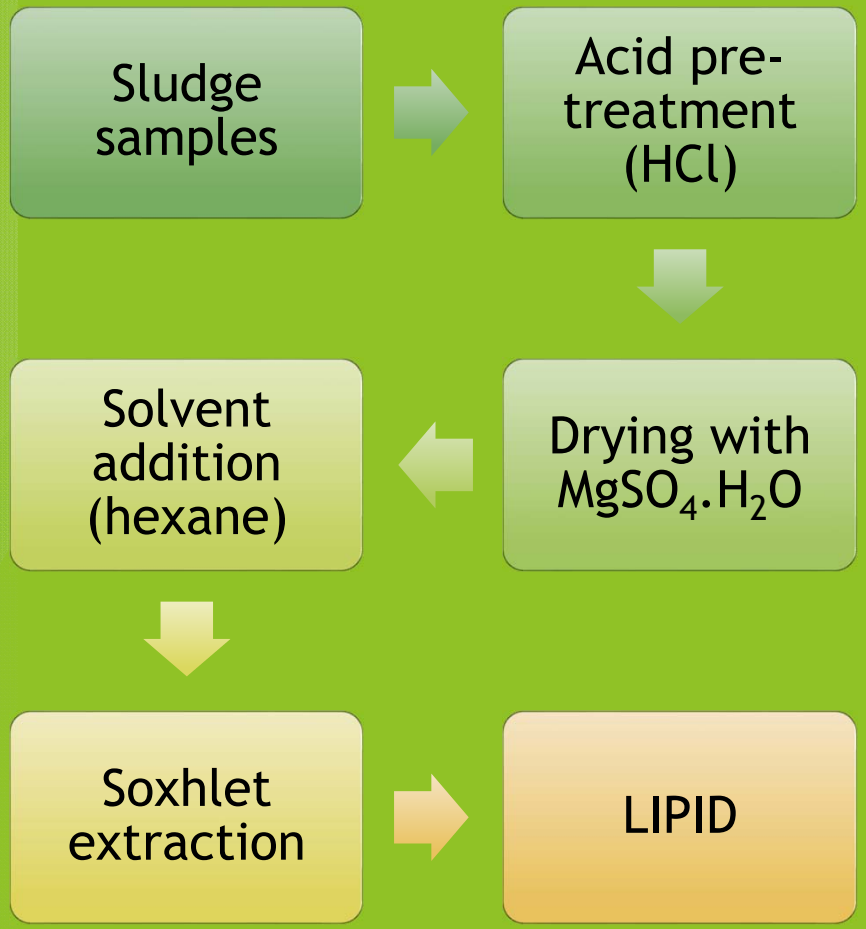
- 20 kHzfrequency
- 200 W power output
- 70% amplitude
- 13 min
- 15000 kJ/kg TS

Lipid Extraction Methods

1. **Direct liquid-liquid lipid extraction method**

2. **Standard drying method**

Standard Drying Method



Direct Liquid-Liquid Lipid Extraction Method

- Sludge samples
- Acid pre-treatment
- Solvent addition (hexane)
- Mixing at 200 rpm for 20 min
- Settling at 60 rpm for 15 min
- Filtering with 4µm filter paper
- Application of anhydrous Na_2SO_4
- Evaporating
- LIPID

RESULTS AND DISCUSSION

Effect of Extraction and Pre-treatment Methods

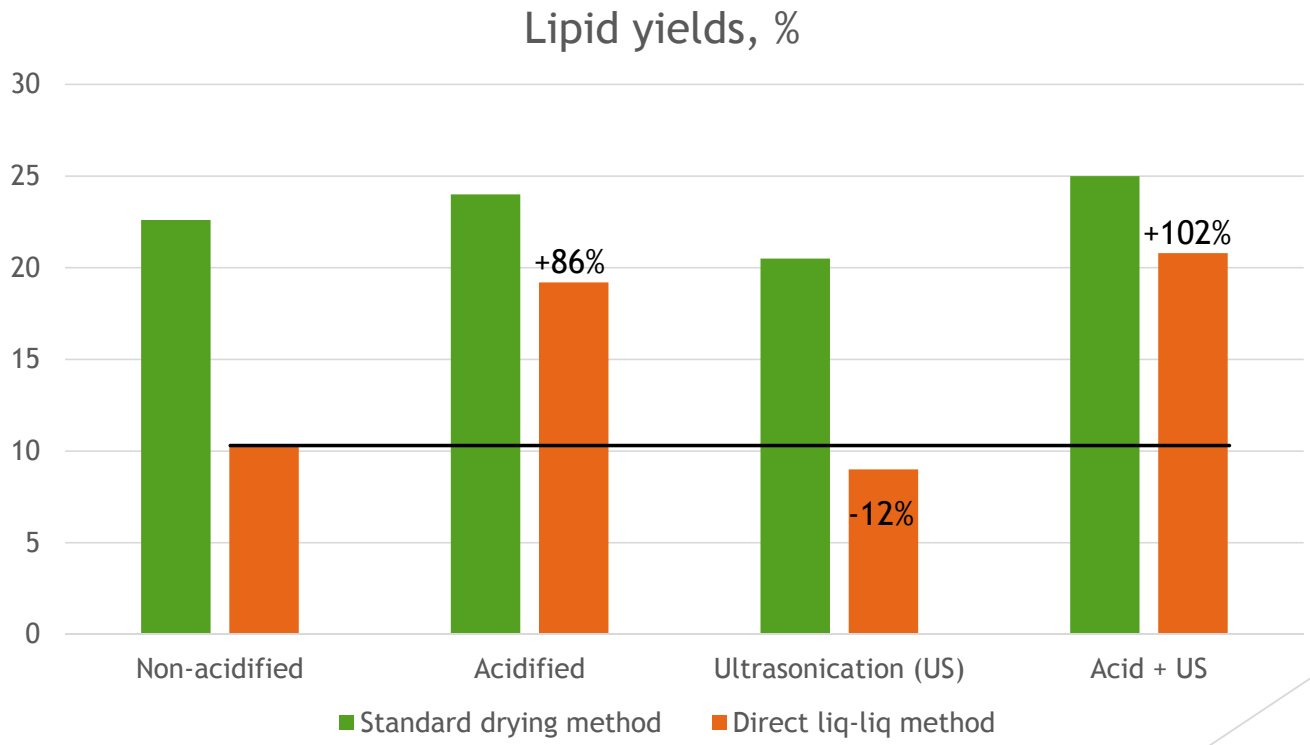
Sludge type	Extraction method	Lipid Yield (%)(a)			
		Pre-treatment method			
		No-pretreatment	Acid	US (Ultrasonic)	Acid+US
Primary sewage sludge	Standard drying(b)	22.6	24.0	20.5	25.0
	Direct liquid-liquid extraction	10.3	19.2	9.0	20.8
Petrochemical industry WWTP sludge	Standard drying(b)	21.4	23.7	29.1	33.1
	Direct liquid-liquid extraction	30.5	34.8	30.9	45.2

+50%

(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.

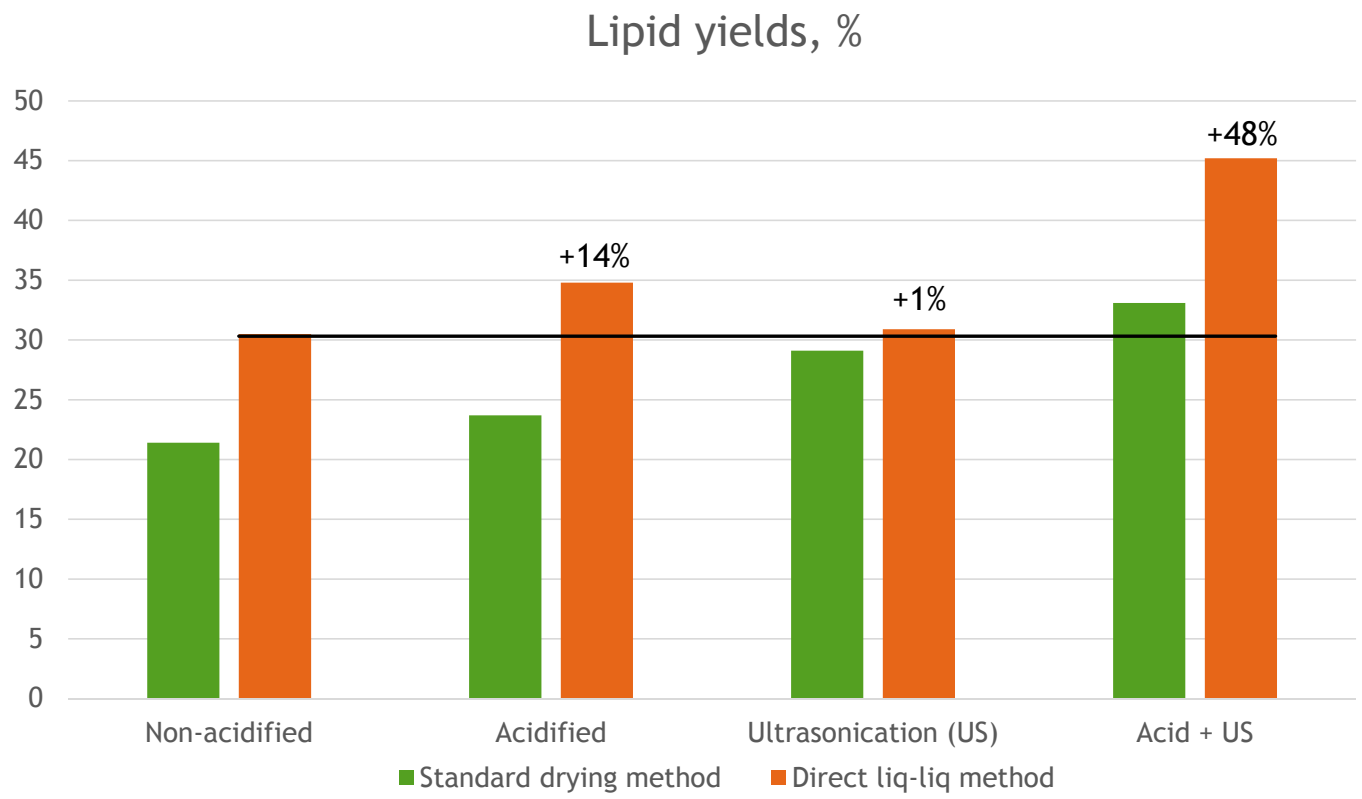
Comparison of Lipid Yields

- **Primary Wastewater Sludge**

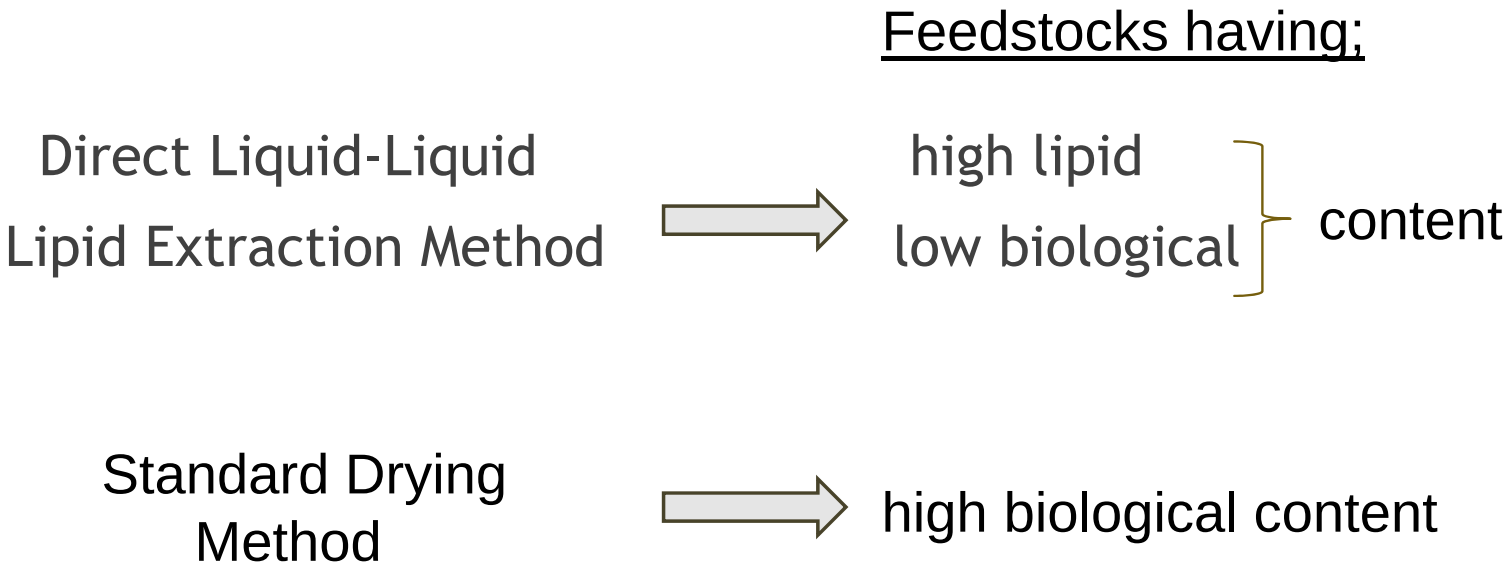


Comparison of Lipid Yields

- **Petrochemical Industry WWTP Sludge**



Efficiency of Extraction Methods



CONCLUSIONS

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

Direct liquid-liquid lipid extraction method resulted with higher lipid yields for the *petrochemical industry WWTP sludge* having high lipid low biological content

Standard drying method is found to be more efficient for the municipal sludge having high biological and relatively low lipid content.

CONCLUSIONS

Acid pre-treatment increased the lipid yields.

The ultrasonication pretreatment alone did not improve the lipid yields of municipal wastewater sludge samples.

The combined acidification/ultrasonication pretreatment caused to an important increase in the lipid yields of both sludge types.

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

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THANK YOU ☺

Biodiesel Production Yields

Sludge type	Extraction method	Yield (%)[a]	Non-acid.	Acid	US	US+Acid
Primary Wastewater sludge	Standard drying[b]	Saponifiable	58.0	55.0	50.0	56.0
		Biodiesel	13.1	13.2	10.2	14.0
	Direct liquid-liquid extraction	Saponifiable	54.0	68.0	61.0	67.0
		Biodiesel	5.6	13.1	5.5	14.0
Petrochemical Industry WWTP sludge	Standard drying[b]	Saponifiable	65.0	66.0	67.0	68.0
		Biodiesel	13.9	15.7	19.7	22.5
	Direct liquid-liquid extraction	Saponifiable	68.0	71.0	69.0	69.0
		Biodiesel	21.0	24.7	21.4	31.1

[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.