Design of a Ɛ-caprolactam-based ionic liquids for the extraction of lipids from starved *Parachlorella kessleri* microalgae for sustainable aviation fuels (SAF)

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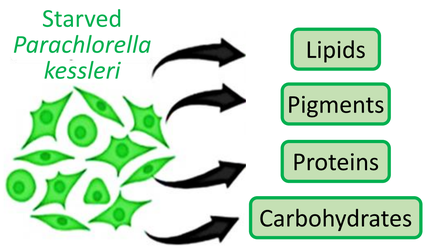
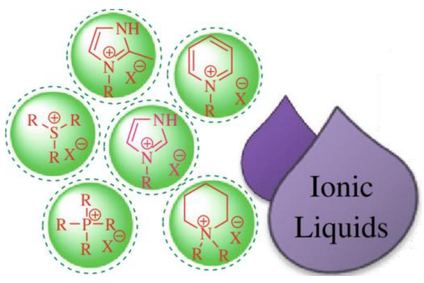
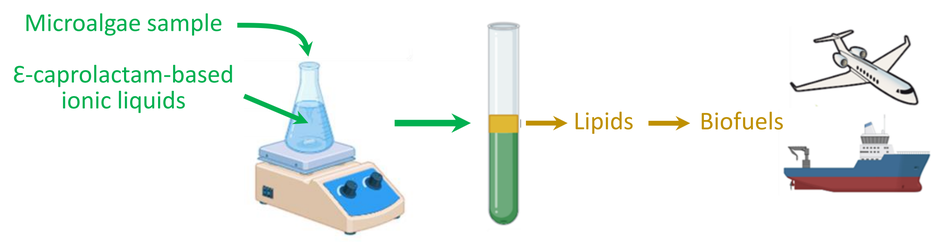
Introduction



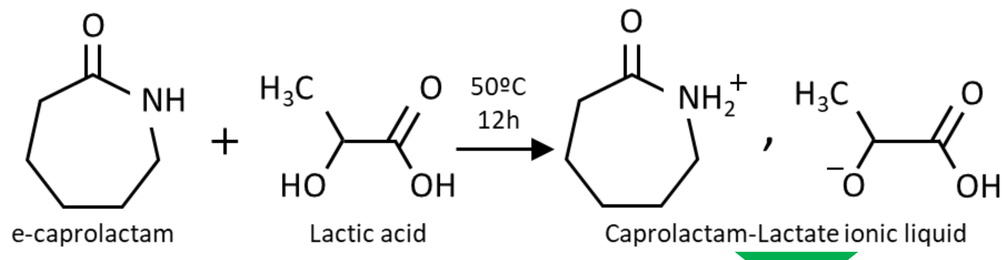


- Molten salts M.P. < 100ºC
- (Semi)-organic
- Non-volatile
- Non-flammable
- Moderate viscosity
- High Polarity
- High ionic conductivity
- Thermal and chemical stability



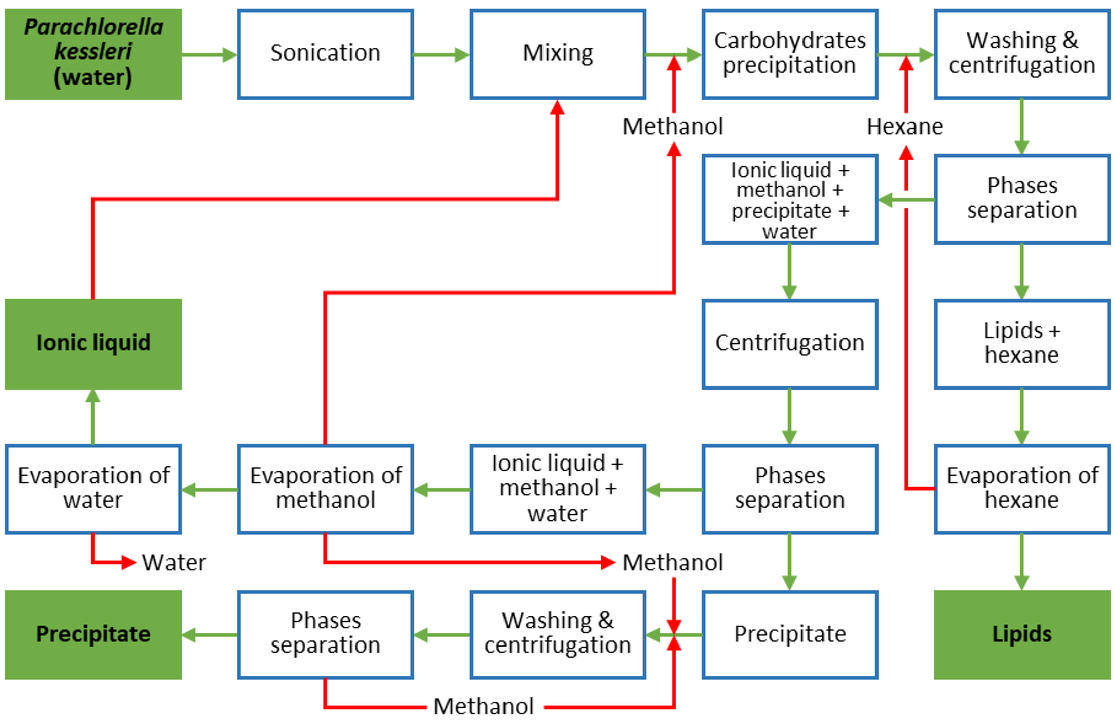
Experimental procedure

Synthesis of ionic liquid based on ε-caprolactam



Characterized by: NMR, TGA, DSC and FTIR

Microalgae lipids extraction procedure



Experimental design: response surface diagram

Table 1. Values and codes used in the experimental design.

Factor	Code				
	-2	-1	0	1	2
Temperature (°C)	20	40	60	80	100
Ratio (microalgae:IL)	1:1	1:4	1:7	1:10	1:13
Sonication time (min)	0	10	20	30	40
Stirring time (min)	0	2	4	6	8

Conclusions

ε-caprolactam-lactate (CPL-Lac) ILs stood out as the most efficient in extracting lipids from starved Parachlorella kessleri, with a recovery percentage of 89.8%

Lipid recovery reached up to 96,24% under conditions: 40°C, 1:10 biomass/IL, 6 h of stirring, and 30 min of ultrasound

The optimal conditions proposed by the Minitab® software (42 °C, 1:11, 8 h, 19 min sonication) resulted in lower yields between 89.6-91.8%

Palmitic (C16:0), stearic (C18:0), oleic (C18:1 cis + trans), linoleic (C18:2 trans) and α-Linolenic (C18:3 n3) acids predominate

Experimental Results

Table 2. Screening of lipids extraction using different ε-caprolactam-based ionic liquids.

Ionic Liquid	% Lipids recovered
Caprolactam-Lactate	89.82
Caprolactam-Formate	85.86
Caprolactam-Citrate	86.72
Caprolactam-Acetate	88.61

Table 3. Best results of the extraction experimental design of starved *Parachlorella kessleri* using ε-caprolactam-lactic acid ionic liquids.

Run Order	Ratio (g)	Temperature (ºC)	Stirring (hours)	Sonication time (min)	% Lipids recovered
1	1:4	80	2	30	90.87
4	1:10	40	6	30	96.24
10	1:7	60	4	20	87.81
11	1:7	60	4	20	85.19
15	1:7	60	4	20	88.71
16	1:7	60	8	20	92.92
18	1:4	80	6	30	94.03
19	1:7	20	4	20	81.85
22	1:10	80	2	30	94.35
23	1:7	60	4	20	85.74
28	1:7	60	4	20	82.52
31	1:10	80	6	30	81.54

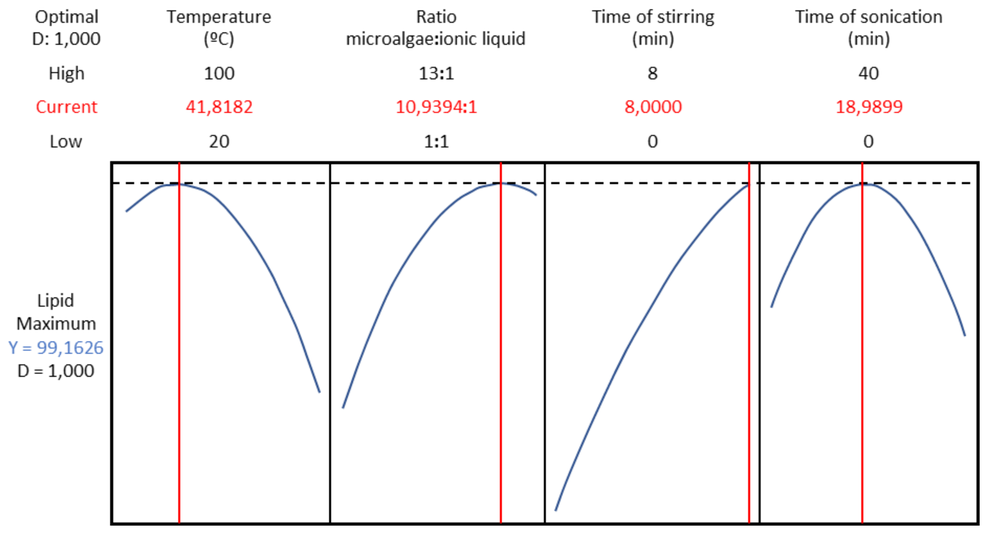


Figure 2. Predicted process optimization of lipid extraction from starved *Parachlorella kessleri* using ε-caprolactam-lactic acid ionic liquid.

Table 4. Lipid recovery results obtained from the optimal conditions of the experimental design of extraction of starved *Parachlorella kessleri* using ε-caprolactam-acid lactic ionic liquids.

Run Nº	Experimental conditions				Recovery of lipids (%)
	Ratio biomass: ionic liquid	Temperature (ºC)	Time (min)		
			Stirring	Sonication	
Opt. 1	1:11	42	8	19	89.6
Opt. 2	1:11	42	8	19	91.8

Acknowledgements

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