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# Enhancing bioenergy yields from sequential bioethanol and biomethane production by means of solid–liquid separation of the substrates

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# INTRODUCTION

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- Lignocellulosic biomass is an important feedstock for biofuel production
- In the ethanol production process a large quantity of sidestream are generated
- Sidestreams have energetic and commercial value
- AD of the sidestreams improves the total energetic output from biomass and removes the environmental load of the waste
- Sequential fermentation and anaerobic digestion is a promising solution [1-2].

1. Rocha-Meneses, L.; Raud, M.; Orupõld, K.; Kikas, T. Potential of bioethanol production waste for methane recovery. *Energy* 2019, 173, 133–139, doi:10.1016/j.energy.2019.02.073.

2. Rocha-Meneses, L.; Ivanova, A.; Atouguia, G.; Ávila, I.; Raud, M.; Orupõld, K.; Kikas, T. The effect of flue gas explosive decompression pretreatment on methane recovery from bioethanol production waste. *Ind. Crops Prod.* 2019, 127, 66–72, doi:10.1016/j.indcrop.2018.10.057.

# INTRODUCTION

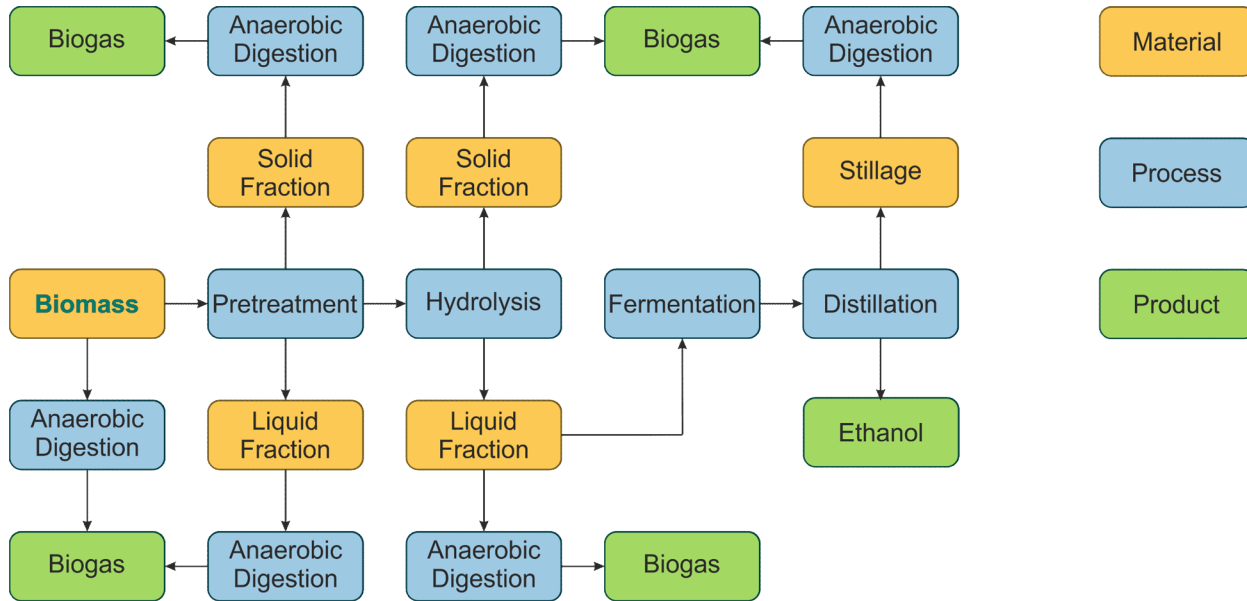
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- There is a continuous search for strategies that aim to improve the efficiency of second-generation bioethanol production.
- Solid–liquid separation of the substrates has also been reported as a solution to improve the overall biogas yields [3,4].
- **How can solid-liquid separation affect the process flow of bioethanol production?**

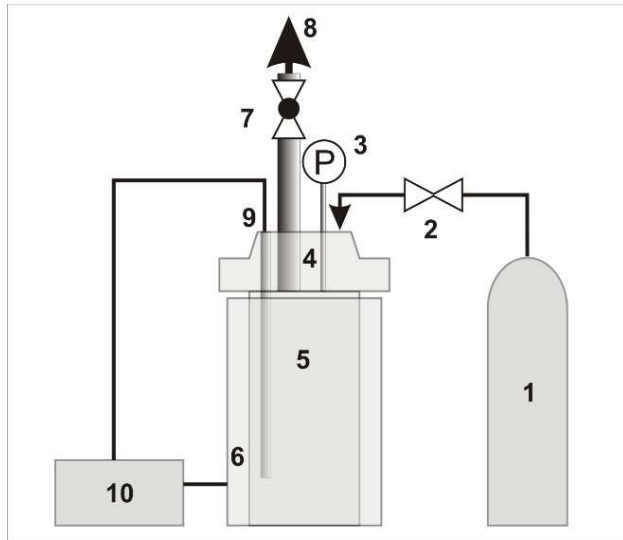
3. Cestonaro do Amaral, A.; Kunz, A.; Radis Steinmetz, R.L.; Scussiato, L.A.; Tápparo, D.C.; Gaspareto, T.C. Influence of solid–liquid separation strategy on biogas yield from a stratified swine production system. *J. Environ. Manag.* **2016**, *168*, 229–235, doi:10.1016/j.jenvman.2015.12.014.

4. Anjos, I.D.; Toneli, J.T.C.L.; Sagula, A.L.; Lucas Junior, J.d. Biogas production in dairy cattle systems, using batch digesters with and without solids separation in the substrates. *Eng. Agric.* **2017**, *37*, 426–432.

# Experimental design



# Pretreatment



- 1 – N<sub>2</sub> tank
- 2 - pressure control valve
- 3 - manometer
- 4 - modified pressure vessel cap
- 5 - Parr instruments pressure vessel
- 6 - ceramic contact heater
- 7 - pressure release valve
- 8 - ventilation system
- 9 - thermocouple
- 10 - temperature controller unit.

# Biomethane potential

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- BMP test based on Owen *et al.* [6] and Angelidaki *et al.* [7] before and after each process step
- Experiments during 42-45 days
- Analysis of the TS and VS
- Methane content: Gas chromatograph CP-4900 Micro-GC, Varian Inc.
- Measurements performed in triplicate

[6] Owen, W.F., et al., *Bioassay for monitoring biochemical methane potential and anaerobic toxicity*. Water Research, 1979. **13**(6): p. 485-492.

[7] Angelidaki, I., et al., *Defining the biomethane potential (BMP) of solid organic wastes and energy crops: a proposed protocol for batch assays*. Water Science and Technology, 2009. **59**(5): p. 927-934.

# Characterisation of materials

Fraction	Variable	TS (g/kg)	VS (g/kgTS)
-	Untreated	931 ± 0	963 ± 0
Liquid Fraction	Pretreated	18.0 ± 0.07	997 ± 0
	Hydrolyzed	34.2 ± 0.09 <sup>a</sup>	997 ± 0
	Fermented	20.1 ± 0.8	997 ± 0 <sup>b</sup>
	Sidestream	23.4 ± 0.7 <sup>a</sup>	997 ± 0 <sup>b</sup>
Solid Fraction	Pretreated	118 ± 0	996 ± 0 <sup>b</sup>
	Hydrolyzed	139 ± 2	995 ± 0 <sup>b</sup>
	Fermented	123 ± 1	995 ± 0
	Sidestream	128 ± 6	995 ± 0

Component	Content (%)
Hemicellulose	32.6 ± 0.5
Cellulose	45.7 ± 0.2
Lignin	5.2 ± 0.0
Ash	3.8 ± 0.1

# Results and Discussion

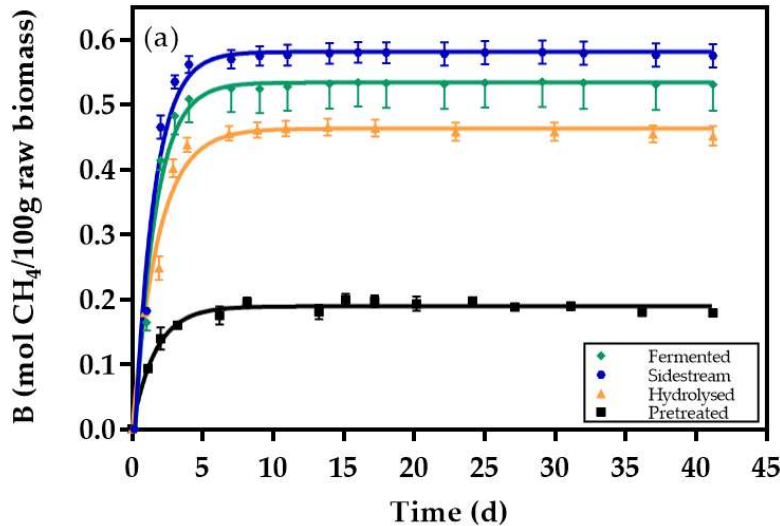
Concentrations of glucose, xylose, glycerol, acetic acid, and ethanol (g/L) in samples from different stages of bioethanol production that has been pretreated with NE.

	Glucose (g/L)	Xylose (g/L)	Glycerol (g/L)	Acetic acid (g/L)	Ethanol (g/L)
Pretreated	0.48 ± 0.02	0.6 ± 0.4	< 0.25 <sup>a</sup>	1.53 ± 0.03	-
Hydrolysed	13.7 ± 0.8	4.06 ± 0.18	< 0.25 <sup>a</sup>	1.81 ± 0.01	1.15 ± 0.05
Fermented	0.25 ± 0.09	3.6 ± 0.4	0.73 ± 0.10	2.2 ± 0.2	8.3 ± 0.7
Stillage	0.8 ± 0.3	3.8 ± 0.6	0.71 ± 0.06	2.6 ± 0.3	-

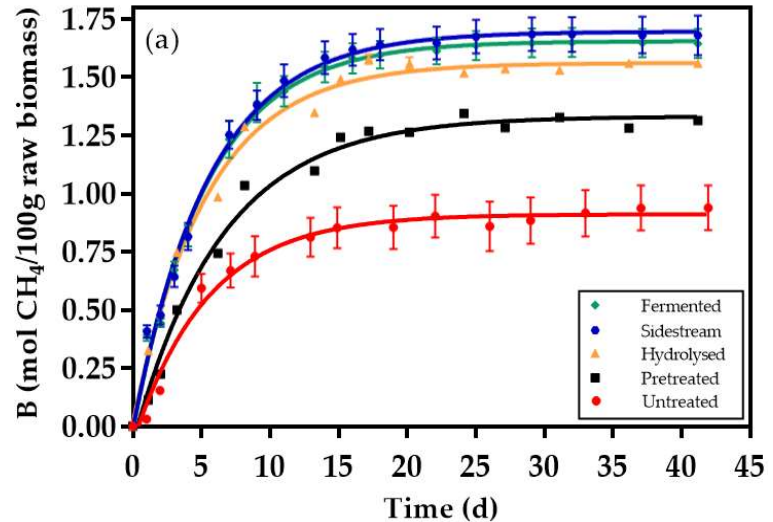


# Results and Discussion

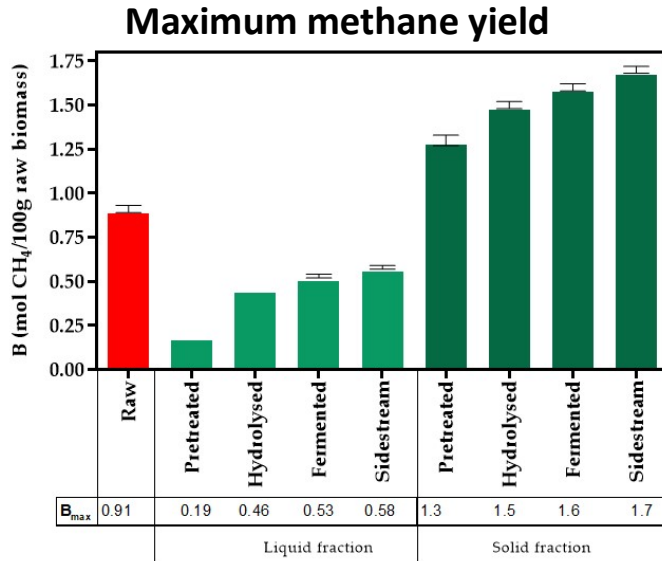
## ▪ Samples from the liquid fraction



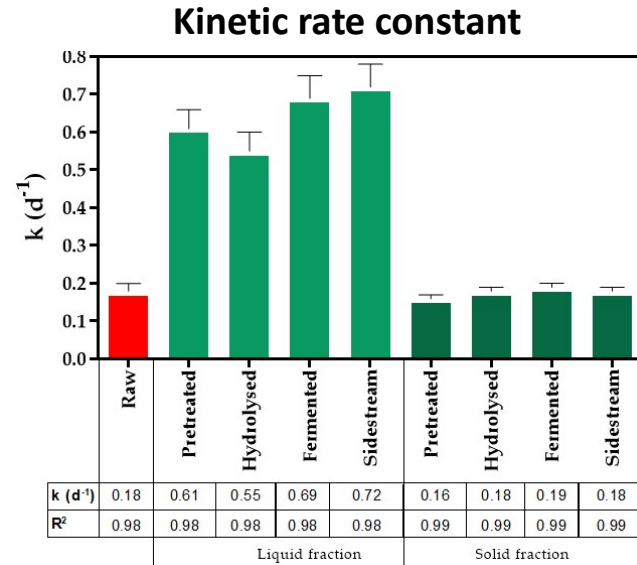
## ▪ Samples from the solid fraction



# Results and Discussion



**Fig 3.** Maximum methane yield ( $B_{max}$ ) for the fitting curves of samples taken from the liquid and solid fraction of pretreated, hydrolyzed, fermented material, and the bioethanol sidestream, pretreated with NED.



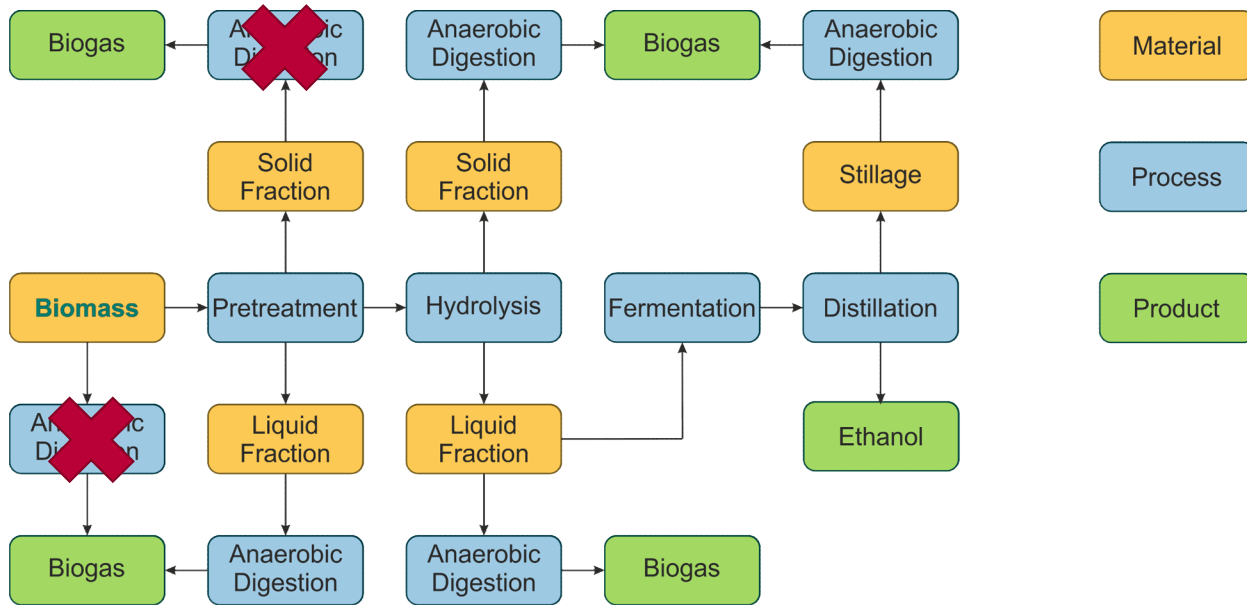
**Fig 4.** Kinetic rate constant ( $k$ ) and correlation coefficient ( $R^2$ ) for the fitting curves of samples from the liquid and solid fraction of pretreated, hydrolyzed, fermented material, and the bioethanol sidestream, pretreated with NED.

# Results and Discussion

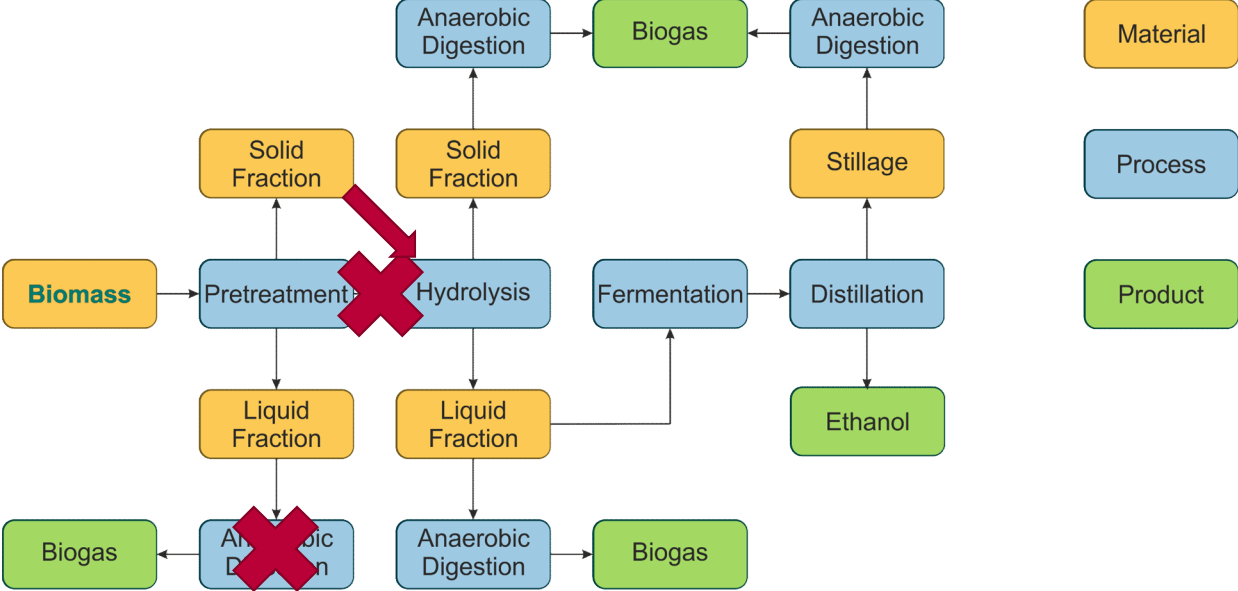
**Table 2.** Digestion time (85% B<sub>max</sub> and 95% B<sub>max</sub>) for samples from the liquid and solid fraction of pretreated, hydrolyzed, fermented material, and the bioethanol sidestream, pretreated with NED and flue gas.

Fraction	Variable	85% B <sub>max</sub>		95% B <sub>max</sub>	
		mol CH <sub>4</sub> /100 g	Days	mol CH <sub>4</sub> /100 g	Days
Untreated	-	0.88	14.0	0.98	21.7
Liquid Fraction	Pretreated	0.16	2.6	0.18	4.1
	Hydrolyzed	0.39	2.8	0.44	4.5
	Fermented	0.45	2.4	0.51	3.8
	Sidestream	0.49	2.4	0.55	3.6
Solid Fraction	Pretreated	1.1	12.5	1.3	18.9
	Hydrolyzed	1.3	10.2	1.5	16.1
	Fermented	1.4	10.2	1.6	16.1
	Sidestream	1.4	10.5	1.6	16.6

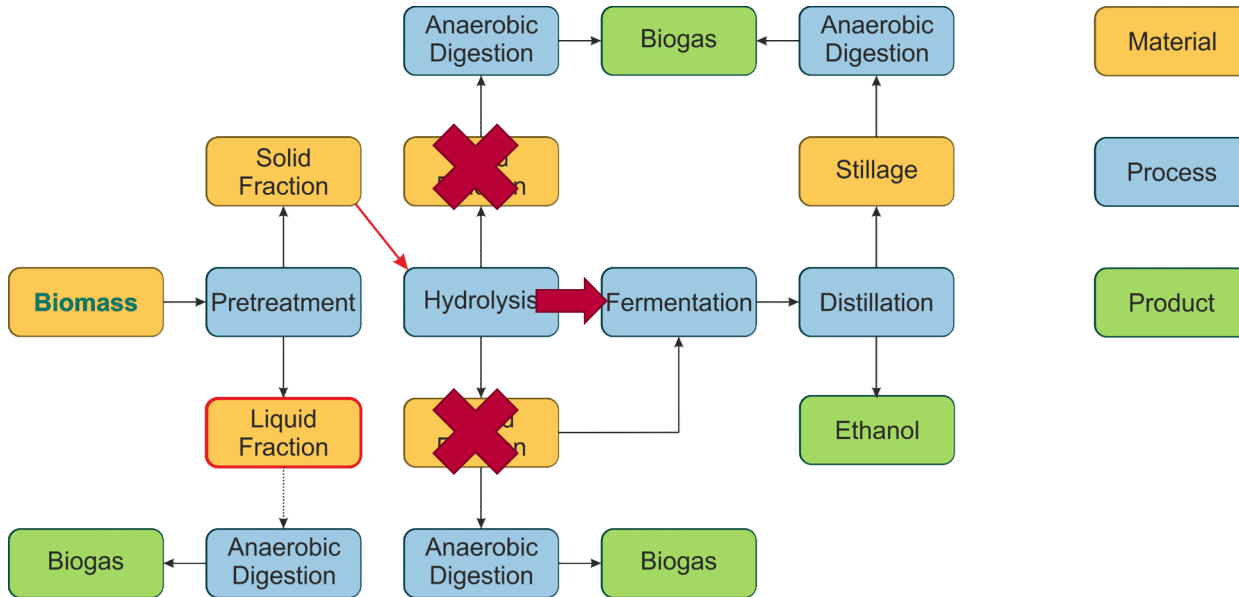
# Production flow analysis



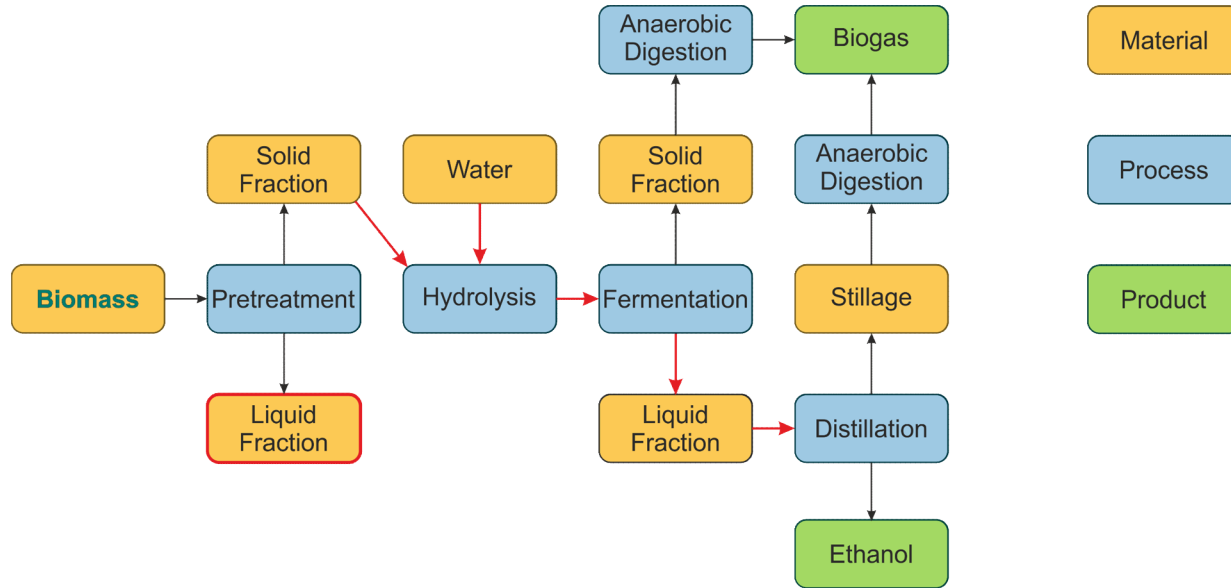
# Production flow analysis



# Production flow analysis



# Production flow



# CONCLUSION

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- Sidestreams have high energy potentials
- Solid–liquid separation is an effective strategy for enhancing methane yields
- The methane yields from the liquid fractions were between 60–88% lower than those that were obtained from solid fractions.
- The kinetics of liquid AD were more than 3 times higher
- Using liquid phase from pretreatment in downstream processes is questionable
- Separating solids after hydrolysis is not reasonable
- Highest energy output comes from integrated bioethanol biogas production process where stillage and solids are combined in AD





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# Thank you for your attention!