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School of Chemical Engineering
Unit of Environmental Science & Technology

Stepwise verification and upscaling process for bioethanol production from source separated food waste

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- Source-Separated Food Waste
- Valorization of Food Waste (BioEthanol)

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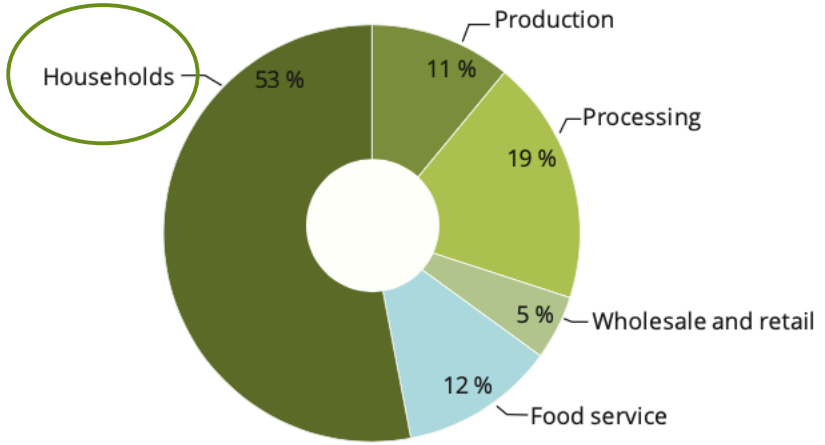
BioWaste - Food Waste

Environmental Impact

Economic Impact

Social Impact

Biowaste: 34% of municipal waste in Europe

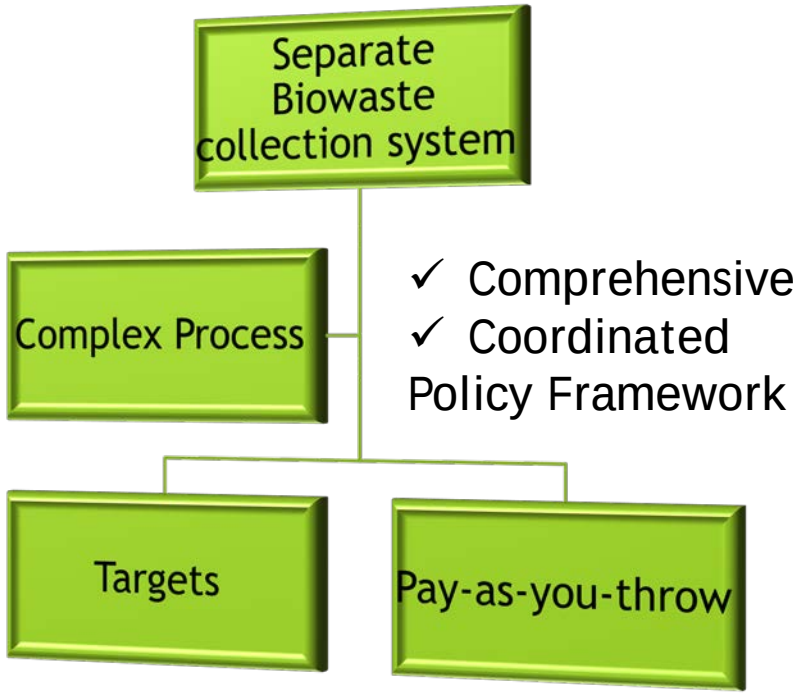


FoodWaste accounts for 60% of all Biowaste

Source-Separated Food Waste

High-quality value-added products needs:

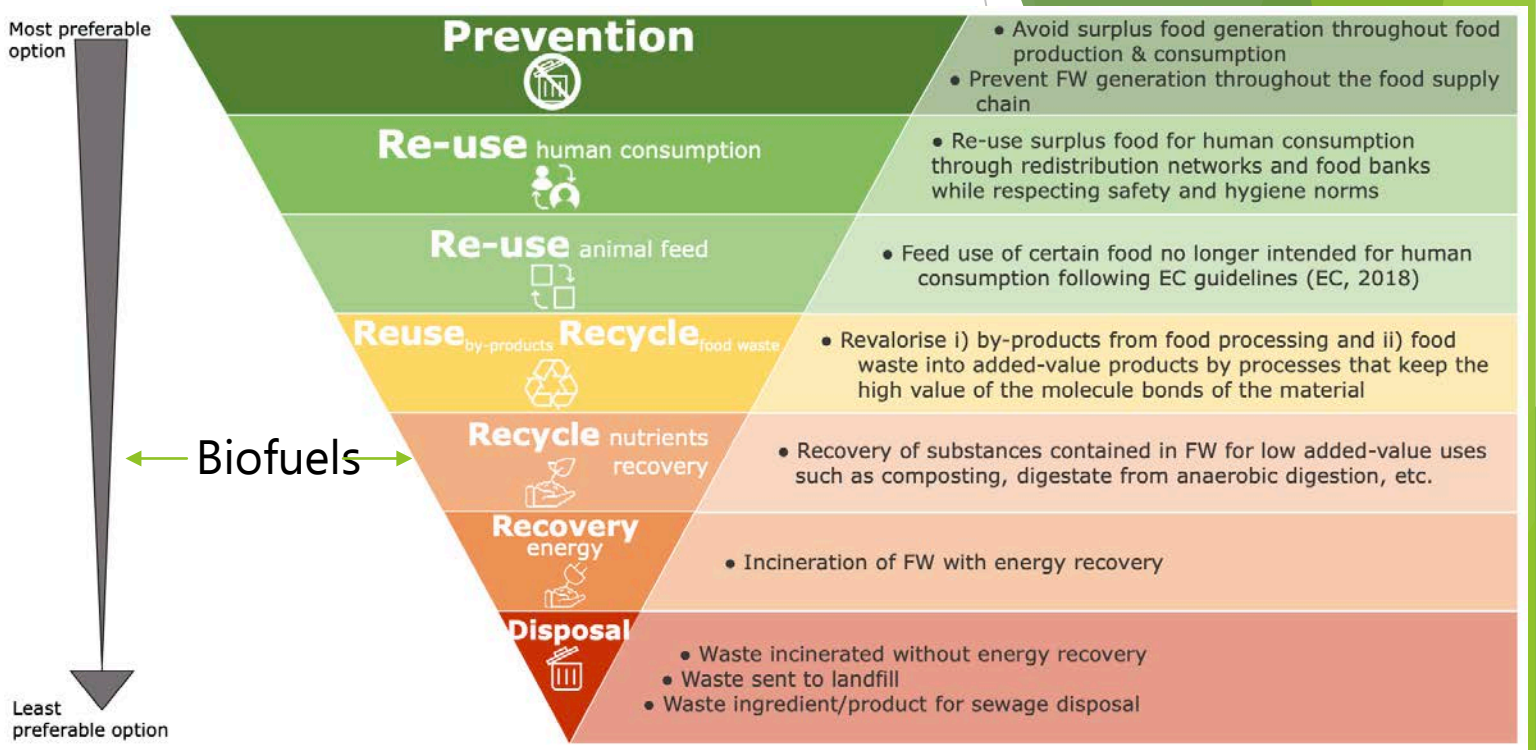
- Collected separately at source



Food Waste Hierarchy

NOT ALL FOOD WASTE WILL BE PREVENTED :

- ✓ Technologies about valorizing them must be developed



Biofuels

Climate change

Fossil Fuels

Biofuels

Key role

Low carbon Transport

Bioethanol

EXPERIMENTAL

Source-Separated Food Waste

SUSTAINABLE CITY
CITY NETWORK

Cleaning service of the Municipality of VVV

- ✓ Collection
- ✓ Transportation
- ✓ Feedstock Delivery

- o Delivery Frequency: 15 days
- o Feedstock Quantity: 200 kg

Feedstock Delivery

Food Waste Code	Delivery Date	Quantity (kg)
«K»	15/09/20	129,00
«L»	20/09/20	153,72
«M»	13/10/20	90,00
«N»	27/10/20	190,00
«O»	01/12/20	199,50
«P»	01/12/20	173,00
«Q»	26/01/21	199,50
«R»	09/02/21	195,00
«S»	20/04/21	190,84
«T»	25/05/21	135,74
«U»	08/06/21	155,92
«V»	12/10/21	76,00
«W»	02/11/21	170,91
«X»	23/11/21	121,08
«Y»	07/12/21	150,65
SUM		**2330,86**

Drying and Milling

GAIA GC-100 Organic Waste Dryer

50 kg Wet Solids

75% Moisture

15-18h, 120 ^{o}C

12,5 kg Dry Solids (25% TS)

Residual Moisture 5%

Composition of Feedstocks

		Dry Basis (%)								
A/A	Date of Delivery	Moisture	Fats & Oils	Water Soluble Solids	Ash	Cellulose	Hemi-cellulose	Starch	Acid Soluble Lignin	Acid Insoluble Residue
1	15/9/2020	75,71	11,52	39,37	12,25	14,24	35,32	3,70	1,28	9,16
2	29/9/2020	77,98	13,07	33,66	13,81	20,50	7,04	2,98	1,41	10,35
3	13/10/2020	76,19	13,81	37,47	12,31	19,99	4,19	4,77	1,47	9,53
4	27/10/2020	76,84	12,27	36,31	13,27	24,71	4,58	3,92	1,25	9,47
5	24/11/2020	76,00	9,17	35,39	13,09	16,82	4,38	4,66	1,01	16,14
6	1/12/2020	75,09	14,49	39,89	12,88	14,67	4,15	3,62	1,03	6,64
7	26/1/2021	73,17	15,22	37,28	7,82	17,25	9,76	8,30	1,14	8,89
8	9/2/2021	72,61	11,23	25,50	11,83	18,42	8,69	9,70	2,98	13,19
9	20/4/2021	73,90	14,64	27,68	7,46	15,98	8,50	10,36	1,41	17,88
10	25/5/2021	71,49	15,44	29,08	7,80	12,84	5,14	18,49	2,38	15,14
11	8/6/2021	76,68	9,65	40,50	10,45	10,92	14,12	9,63	1,16	11,01
12	12/10/2021	79,15	17,04	31,25	11,10	10,73	8,78	7,67	1,74	12,67

Physicochemical Characterization

	Composition (% w/w d.b.)
Moisture (Wet basis)	75,89 ± 2,24
Oils	13,14 ±2,14
WS	34,43 ± 4,45
Ash	10,83 ± 2,39
Cellulose	16,13 ±3,60
Hemicellulose	9,07 ± 7,23
Starch	7,63 ± 3,94
ASL	2,11 ± 2,46
AIR	11,90 ± 4,38
Free Glucose	2,35 ±1,17

Experimental Protocol

Fermentation mode: SSF at 35 ^{o}C (Simultaneous Saccharification Fermentation)

Factorial experiment

Process Variables:

- Amylase Dosage (Spirizyme XL)
- Cellulose Dosage (NS87014)
- Yeast Dosage (S.cerevisiae)
- Ethanol yield

1st Scale up

4L Reactor

Optimal conditions of factorial experiment

Variable testing:

- Solid Loadings

2nd Scale up

Pilot Trials

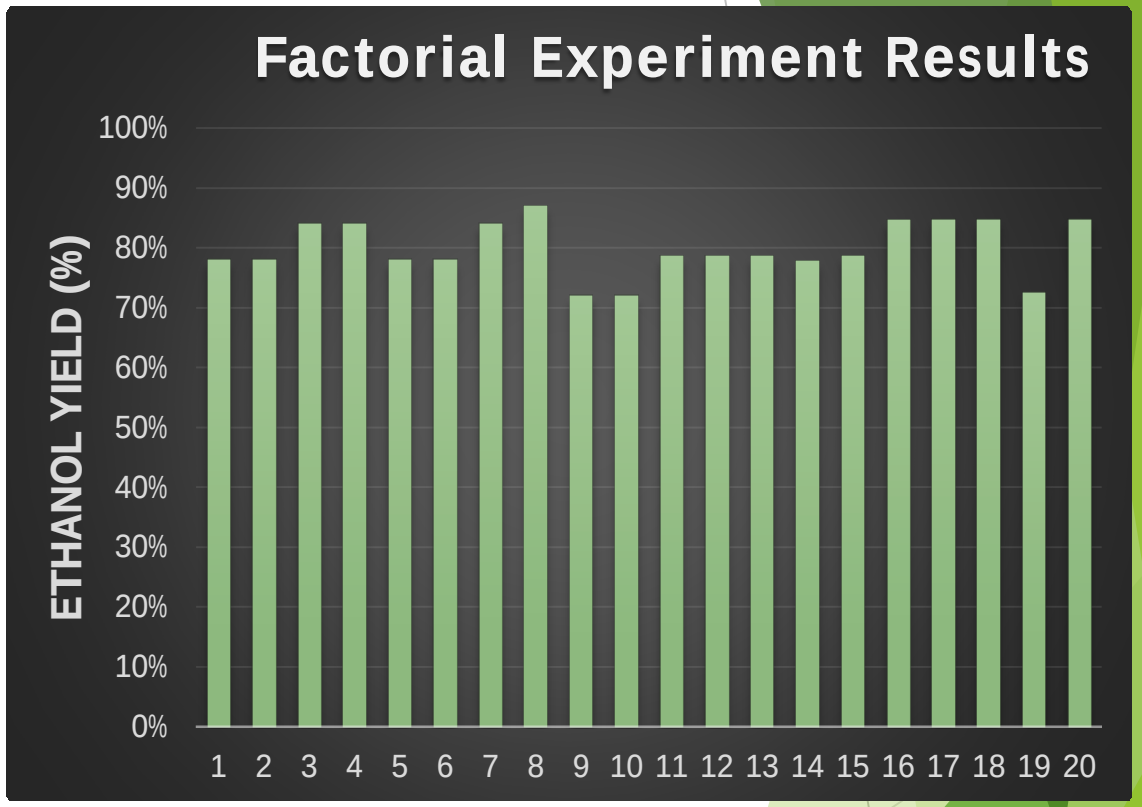
- SSF mode
- Optimal conditions of the previous experiments

Lab-Scale Experiments (100 mL)

Controlling variables and levels of the factorial experiment:

Parameter	Low Level (-)	High Level (+)	Center
SpirizymeXL (µL/g starch)	20	60	40
NS87014 (µL/g cellulose)	100	250	175
S. Cerevisiae (%)	1	3	2

- ✓ SSF mode
- ✓ 35 °C
- ✓ 10% Solid Loading



Results:

- ➢ Average Ethanol Yield: 80,1 %
- ➢ Highest Ethanol Yield: 84,7%

Lab-Scale Experiments (4L)

1st Scale up to 4L:

- At optimum conditions of the factorial

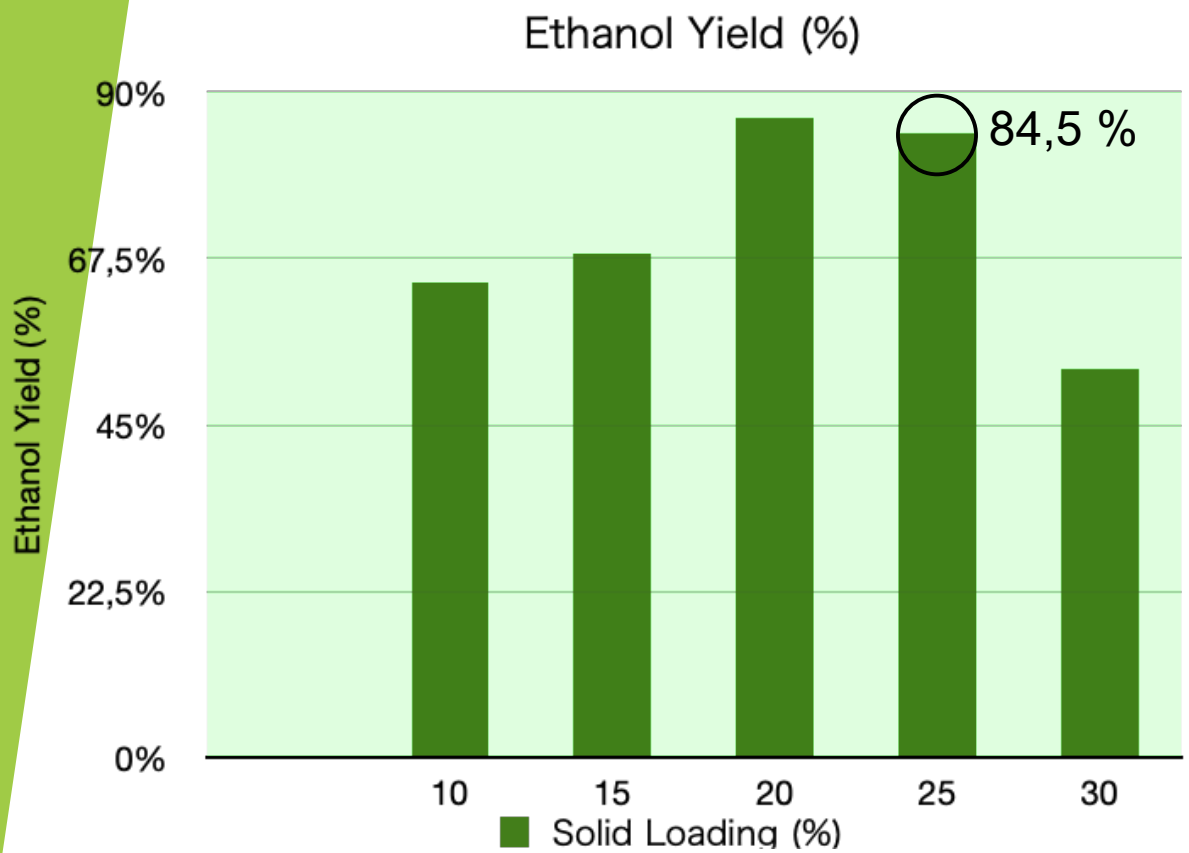
Experiments (No)	Solid Loading (%)	Spirizyme (μL/g starch)	NS87014 (μL/g cell)	S.Cerevisiae (%)
1	10	40	175	2
2	15	40	175	2
3	20	40	175	2
4	25	40	175	2
5	30	40	175	2

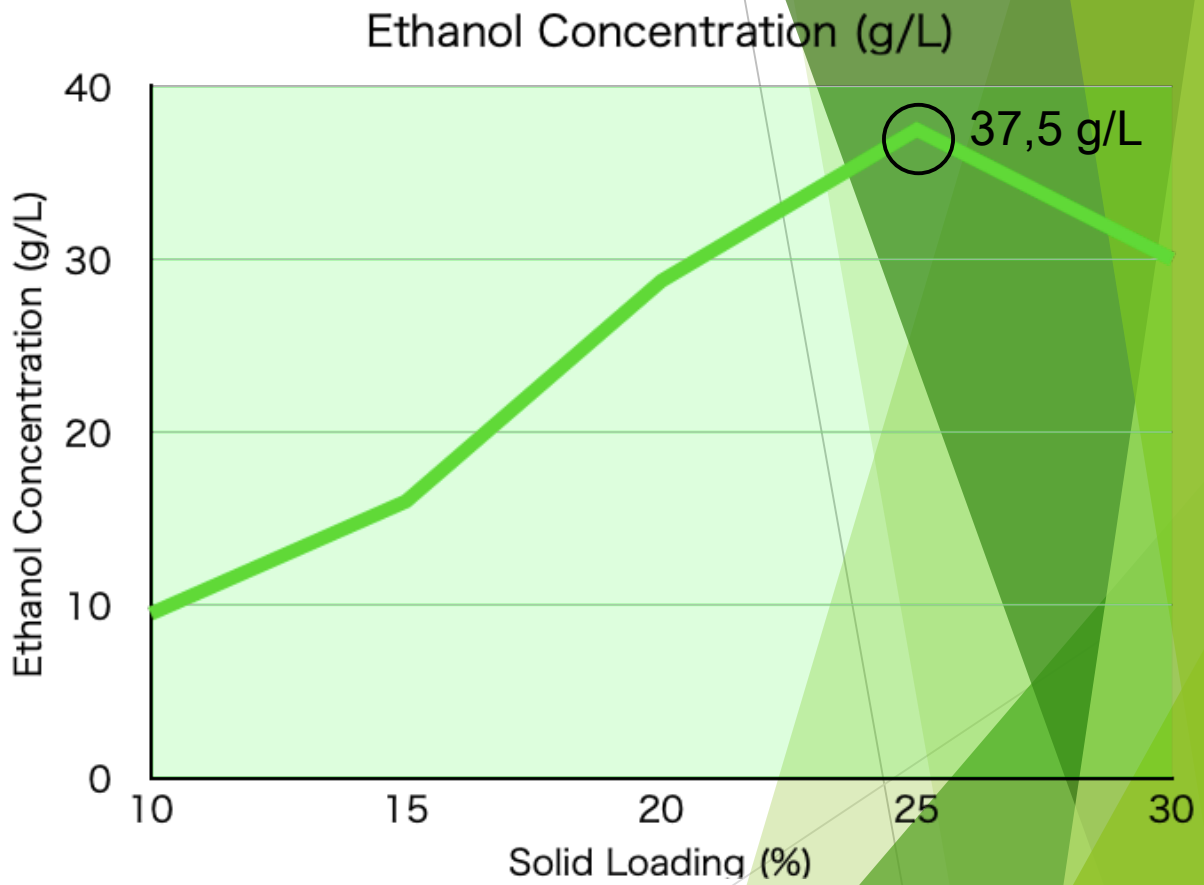
Results:

✓ Optimum Results:

- 25 % Solid Loading
- Ethanol Yield: 84,5 %
- Ethanol Concentration: 37,5 g/L

Lab-Scale Experiments (4 L)





Pilot Scale

- ✓ Fermentation mode: SSF
- ✓ Temperature : 35 ^{o}C
- ✓ Solid Loading: 25 %
- ✓ Residence time: 24 h

Optimum Conditions:

Amylase: 40 µL/g_{starch}
Cellulase: 170 µL/$g_{cellulose}$
Yeast: 2 % of TS

Hydrolysis - Fermentation

Characteristics:

- Two rotating vessels
- Double walls
- Temperature control with water
- Controlled by PLC

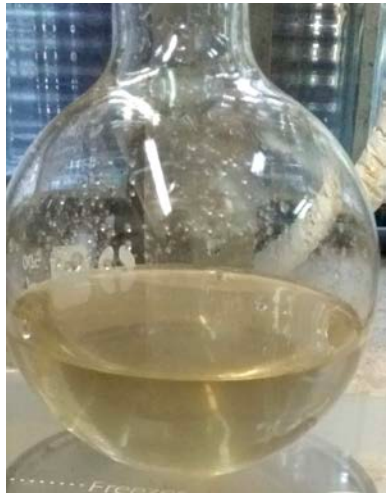
PLC:

Interior of Reactor:

Distillation - Dehydration

Distillation Unit:
- 70 ^{o}C
- Vacuum pump

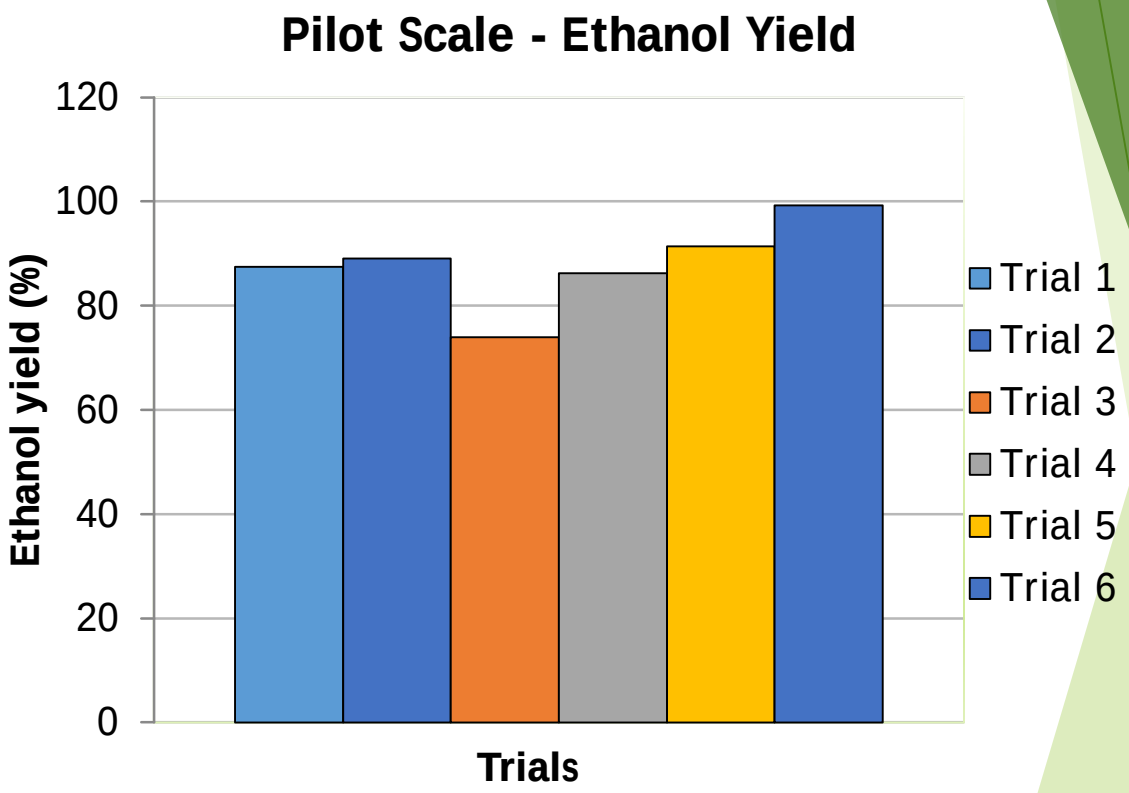
1st Distillate:
35 % v/v



Lab-scale two stage distillation:
94-95 % v/v

Zeolite 3A:
99,55 % v/v

Ethanol Yield



Ethanol yield as the main target was calculated as:

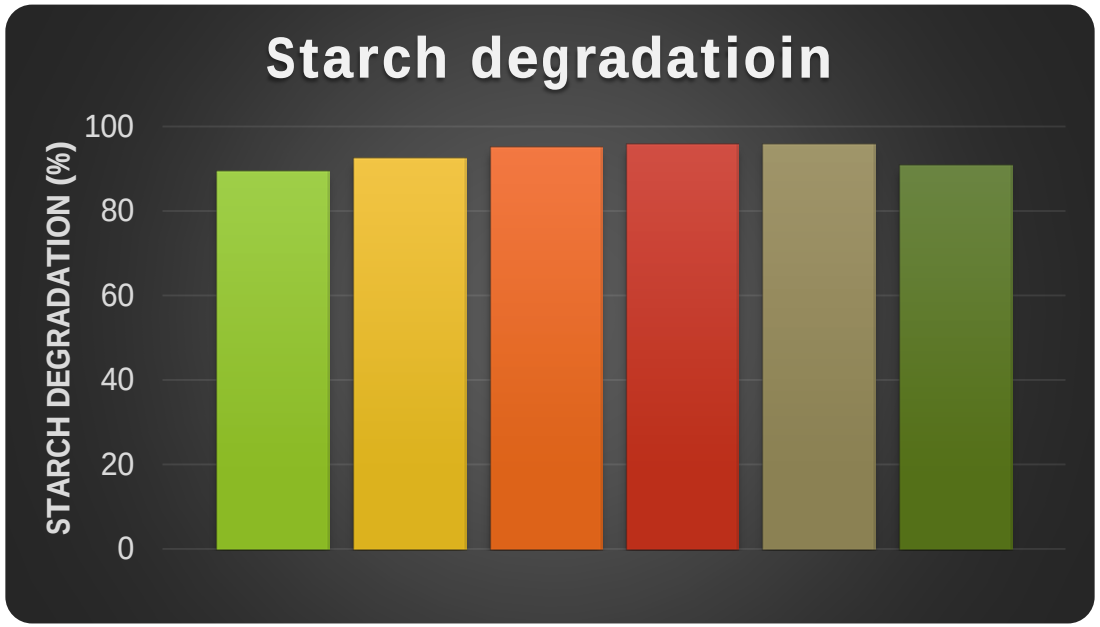
$$Y_{EtOH} = \frac{Maximum\ Ethanol\ Concentration}{Theoretical\ Ethanol\ Cocnentration}$$

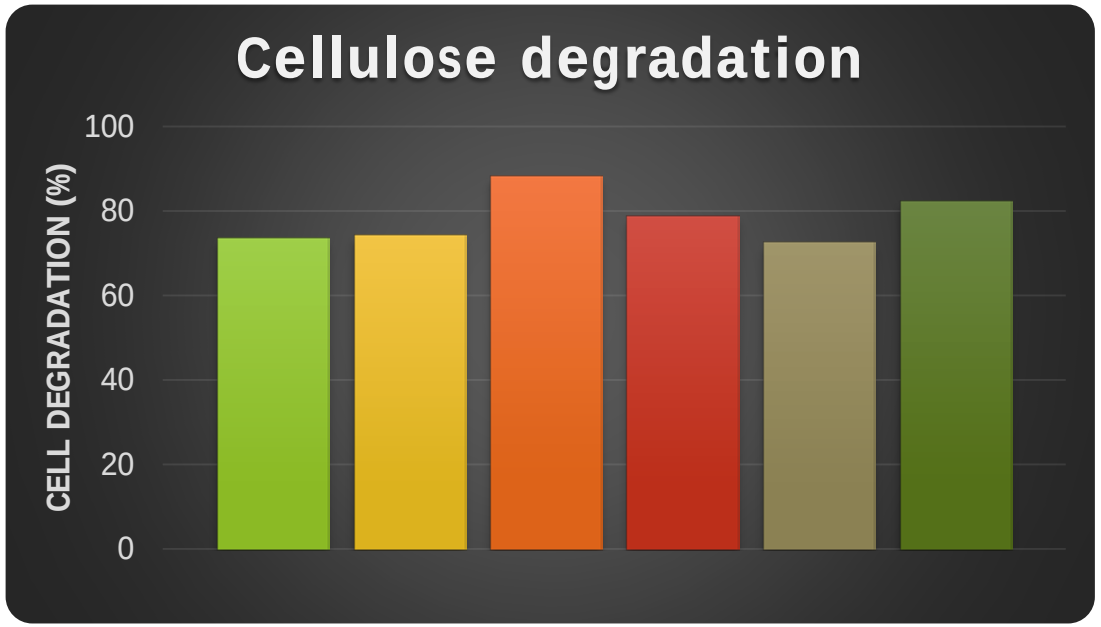
Notes:

1. Maximum Ethanol concentration was about at 8 hours.
2. Ethanol yields were verified by polysaccharides' degradation.

Conclusions:

1. The results were successful and repeatable.
2. The Ethanol yield was between 73 to 90 %.





Conclusions:

1. Starch Degradation is very high and is between 89 and 95 %.
2. Cellulose Degradation is significantly higher than other studies, from 72 to 88 %.
3. Repeatable results are obtained.

Polysaccharides Degradation

Total Results

	Pilot Trials						Average
Degradations	**1**	**2**	**3**	**4**	**5**	**6**	
Solid Degradation (%)	46,28	63,86	70,2	67,96	64,27	68,54	**63,52 ± 8,8**
Cellulose Degradation (%)	73,61	74,27	88,31	78,78	72,48	82,33	**78,30 ± 6,14**
Starch Degradation (%)	89,46	92,34	95,17	95,65	95,85	90,77	**93,20 ± 2,73**
Ethanol Yields							
Max Ethanol Conc (g/L)	34,66	39,26	39,01	42,12	30,03	40,05	**37,49 ± 4,38**
Ethanol yield (%)	87,46	89,01	73,92	86,23	91,45	99,24	**87,89 ± 8,25**
EtOH yield by Cell,Starch,Glucose Degradation (%)	75,14	83,09	91,4	86,92	83,72	86,59	**84,48 ± 5,44**
EtOH yield by Cell and Starch Degradation (%)	69,3	68,37	83,67	76,58	81,99	77,16	**76,18 ± 6,31**

BioEthanol Analysis

Analysis by HELPE

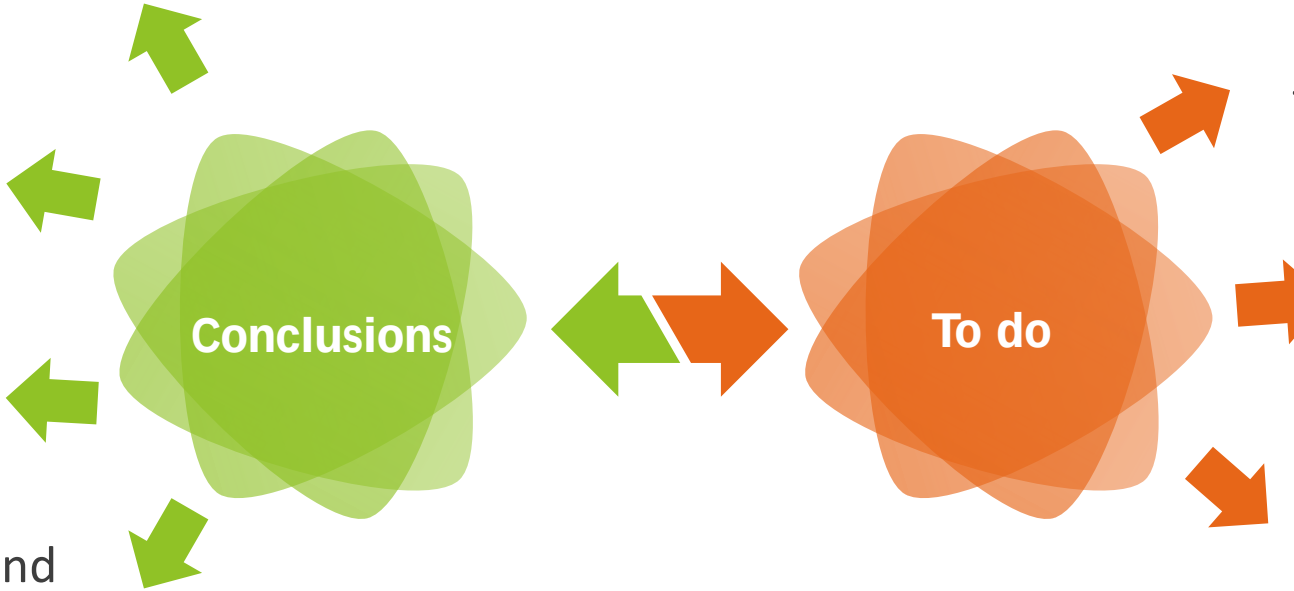
- High quality Ethanol
- High purity (99,55% v/v), according to EN 15376:2012
- Production of biofuel E10 (Gasoline 95 RON + 10% Bioethanol) agree with the properties of unleaded gasoline 95 RON according to EN 228:2012

Conclusions & Challenges

Upscaling of bioethanol production at pilot scale

The **new enzymatic cocktails** were very efficient at 35 ^{o}C

Very **high** and **repeatable** results



- High **quality** and **purity** of BioEthanol
- **Properties of E10** Biofuel in line with the limits

Use of **defatted raw material** will increase the efficiency

Mix substrates for better ethanol yield

Energy optimization

THANK YOU

This project has received funding from the European Union's Horizon 2020 research and innovation program under grant agreement no.818308

WaysTUP!

VALUE CHAINS FOR DISRUPTIVE TRANSFORMATION OF URBAN BIOWASTE INTO BIOBASED PRODUCTS IN THE CITY CONTEXT

Pure Ethanol

Test	Meas. Unit	Method	Result
Density at 15 oC	g/ml	EN ISO 12185	0.7951
Methanol	%w/w	EN 15721	0.01
Propan-1-ol	%w/w	EN 15721	0.15
Butan-1-ol	%w/w	EN 15721	0.01
Butan-2-ol	%w/w	EN 15721	0.27
2-Methylpropan-1-ol	%w/w	EN 15721	0.00
2-Methylbutan-1-ol	%w/w	EN 15721	0.15
3-Methylbutan-1-ol	%w/w	EN 15721	0.00
Higher saturated (C3-C5) mono-alcc	%w/w	EN 15721	0.57
Ethanol and higher saturated alcohol	%w/w	EN 15721	99.91
Water	%w/w	EN 15489	0.450
Total acidity (expressed as acetic aci	%w/w	EN 15491	0.001
Electrical conductivity at 25 oC	uS/cm	EN 15938	0.20
Appearance	---	EN 15769	Clear
Colour	---	EN 15769	Colourless
Inorganic chloride	mg/kg	EN 15484	0.1
Sulfate	mg/kg	prEN 15492	1.0
Involatile material	mg/100 ml	EN 15691	1
Total sulphur	ppm-w	ASTM D-5453	2.1

E10 Biofuel

Test	Meas. Unit	Method	Result
Density at 15 oC	g/ml	EN ISO 12185	0.7506
Vapour pressure at 100oF-Mini	kPa	EN 13016-1	77.3
Total sulphur	ppm-w	EN ISO 20846	0.6
* Research Octane Number, RON	---	EN ISO 5164	97.4
* Benzene	%v/v	EN ISO 22854	0.80
* Aromatics	%v/v	EN ISO 22854	34.7
* Olefins	%v/v	EN ISO 22854	1.4
* bio-Ethanol	%v/v	EN ISO 22854	10.0
* Ether>5C (MTBE-ETBE-TAME)	%v/v	EN ISO 22854	1.9
* Oxygen content	%w/w	EN ISO 22854	3.1
* Tert-butyl alcohol	%v/v	EN ISO 22854	0.0
* Appearance	---	VISUAL	Clear & Bright
* Colour	---	VISUAL	Undyed
* Vapour Lock Index	---	Calculated	1122
* Water	%w/w	EN 15489	0.061
I.B.P.	oC	EN ISO 3405	30.7
F.B.P.	oC	EN ISO 3405	190.2
Evaporated at 70 oC	%v/v	EN ISO 3405	49.8
Evaporated at 100 oC	%v/v	EN ISO 3405	58.9
Evaporated at 150 oC	%v/v	EN ISO 3405	87.8
Residue	%v/v	EN ISO 3405	1.0

Unleaded Gasoline 95 RON

Density at 15 oC	g/ml	EN ISO 12185	0.7463
Vapour pressure at 100oF-Mini	kPa	EN 13016-1	70.7
Total sulphur	ppm-w	EN ISO 20846	0.4
* Research Octane Number, RON	---	EN ISO 5164	*** 93.6
* Benzene	%v/v	EN ISO 22854	0.90
* Aromatics	%v/v	EN ISO 22854	*** 38.0
* Olefins	%v/v	EN ISO 22854	1.5
* bio-Ethanol	%v/v	EN ISO 22854	0.0
* Ether>5C (MTBE-ETBE-TAME)	%v/v	EN ISO 22854	2.0
* Oxygen content	%w/w	EN ISO 22854	0.4
* Tert-butyl alcohol	%v/v	EN ISO 22854	0.0
* Appearance	---	VISUAL	Clear & Bright
* Colour	---	VISUAL	Undyed
* Vapour Lock Index	---	Calculated	937
* Water	%w/w	EN 15489	0.005
I.B.P.	oC	EN ISO 3405	29.3
F.B.P.	oC	EN ISO 3405	193.3
Evaporated at 70 oC	%v/v	EN ISO 3405	32.9
Evaporated at 100 oC	%v/v	EN ISO 3405	52.8
Evaporated at 150 oC	%v/v	EN ISO 3405	86.6
Residue	%v/v	EN ISO 3405	1.0

1st Energy Analysis

The distillation took place for 2h and the recovery of ethanol was 63 % and 2.2 L.
The measurement is until the 1st distillate.

Energy Analysis for a pilot scale experiment with 25 kg of dry solids	
Drying	61,5 kWh
SSF	17 kWh
Distillation	30,3 kWh
Total	108,8 kWh
Total per unit	48,5 kWh/$L_{ethanol}$