



**National Technical University of Athens  
School of Chemical Engineering  
Unit of Environmental Science &  
Technology**

# **Assessing straw digestate from anaerobic digestion as feedstock for sugars production**

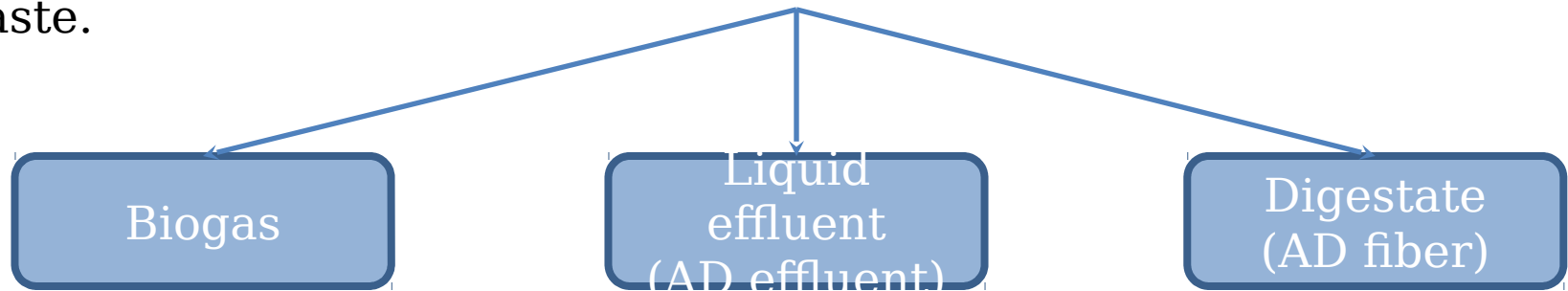
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**7<sup>th</sup> International Conference on Sustainable Solid Waste Management, 26-29 June 2019, Heraklion, Crete Island, Greece**

# Anaerobic Digestion (AD)

Biological conversion process for organic residues into renewable energy, while alleviating environmental concerns associated with the waste.



- Soil amendment
- Animal bedding

# *Aim*

To assess the potential of straw digestate as sugar source



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# *Materials and Methods*



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# Materials and Methods (I)



**Raw material:** Wheat straw from Aspropyrgos province, Greece

**AD fiber sample:** Solid digestate after S/L separation from a pilot scale CSTR anaerobic digester (37°C, HRT= 20 days) that treats wheat straw in NTUA.

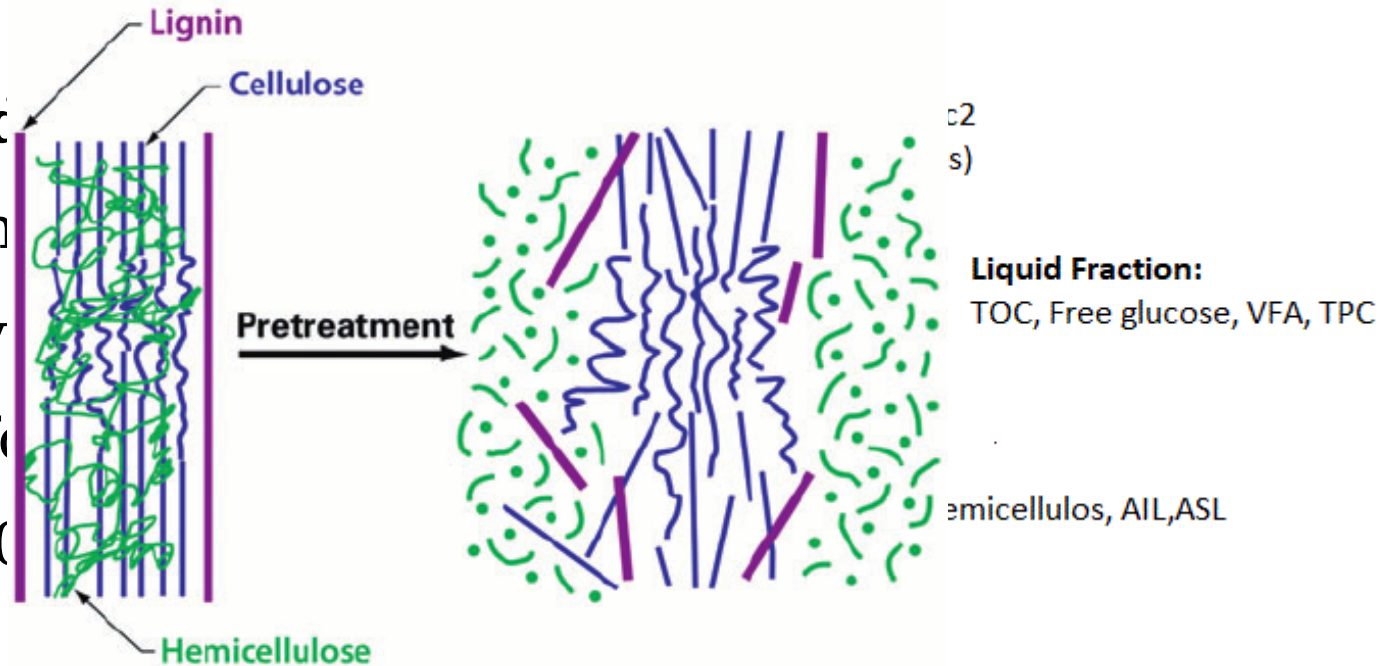
Parameter		Value (% w/w)
Cellulose		24.78
Hemicellulose		11.99
Lignin		18.58
	Klason lignin	17.30
	Acid-soluble lignin	1.28
Ash		27.31

# Materials and Methods (II)

**Chemical pretreatment:** The CSTR AD fiber was pretreated in autoclave with/ without dilute NaOH or H<sub>2</sub>SO<sub>4</sub> at 120°C.

## Enzymatic hydrolysis

- Untreated and pretreated
- 10% w/w dry
- Cellulolytic fungi
- 50°C and 300 rpm



## Materials and Methods (III)

**Factorial experimental procedure:** 2<sup>3</sup> factorial experiment

**Optimization parameter:** Saccharification efficiency SG  
$$SG\% = \frac{\text{Theoretical glucose produced from the total conversion of carbohydrates}}{\text{Theoretical glucose produced from the total conversion of carbohydrates}} \cdot 100\%$$

### ***Controlling parameters***

- Chemicals' concentration
- Autoclave retention time
- Enzyme loading during enzymatic hydrolysis



# Factorial experiments

Alkaline pretreatment prior to enzymatic

Controlling Parameter	Variation Intervals		
	Low level	High Level	Center
Time autoclave, $t_{\text{auto}}$ (h)	1	1.5	1.25
NaOH (%)	2	4	3
CellicCTec2, $C_{\text{enz}}$ ( $\mu\text{L}/\text{g}$ cellulose)	100	400	250

Acidic pretreatment prior to enzymatic hydrolysis

Controlling Parameter	Variation Intervals		
	Low level	High Level	Center
Time autoclave, $t_{\text{auto}}$ (h)	1	1.5	1.25
$\text{H}_2\text{SO}_4$ (%)	1	3	2
CellicCTec2, $C_{\text{enz}}$ ( $\mu\text{L}/\text{g}$ cellulose)	100	400	250





# *Results and Discussion*



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# Alkaline pretreatment

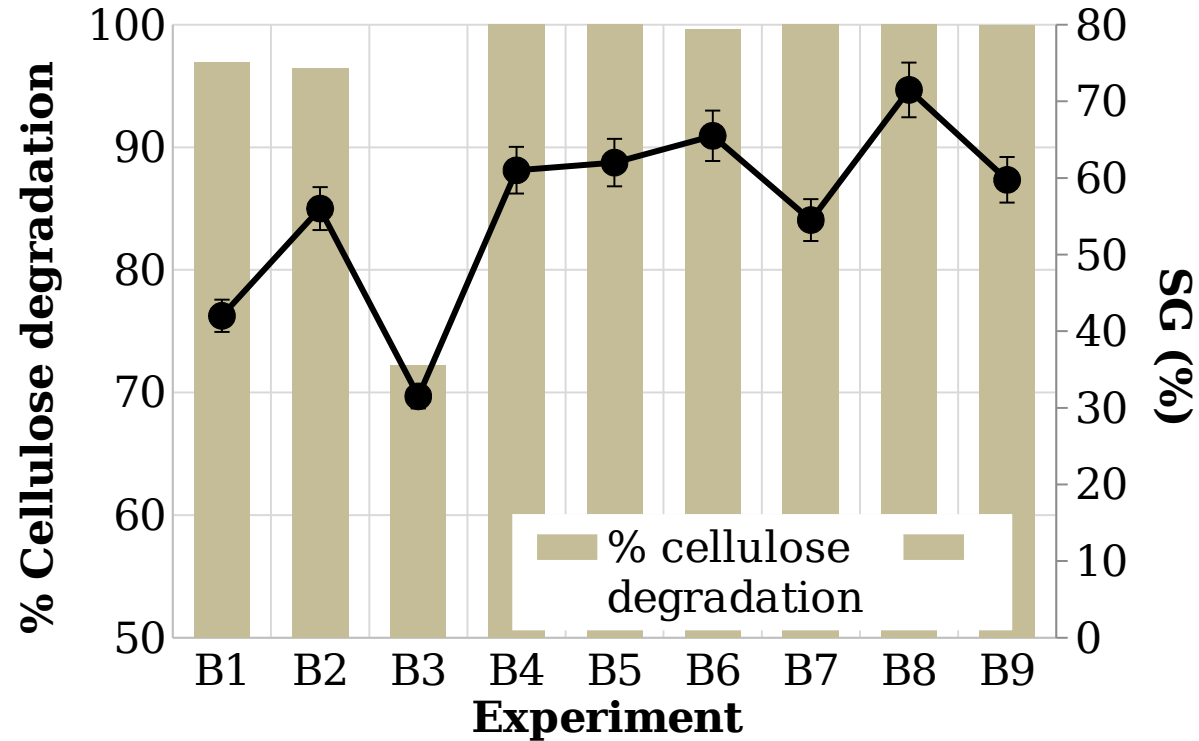
$t_{\text{auto}}$ (h)	NaOH (%)	%TS hydrolysis			%cellulose degradation			%AIL degradation			%ASL degradation			%hemicellulose degradation		
1	2	30.67	±	2.18	1.76	±	3.68	8.23	±	3.75	62.28	±	2.11	3.45	±	5.92
2	4	23.59	±	2.17	2.20	±	6.83	77.14	±	4.39	75.82	±	1.27	74.36	±	1.59
1.5	3	24.01	±	0.89	5.48	±	9.04	74.27	±	3.48	68.62	±	4.16	43.78	±	16.62
1	4	29.02	±	4.76	1.64	±	7.04	76.40	±	4.77	75.16	±	2.41	44.07	±	13.20
2	3	19.84	±	3.33	4.14	±	1.73	71.28	±	2.75	65.15	±	1.42	44.71	±	2.42

- in lignin content
- Slight change in cellulose content
- Glucose 1.11-4.78 mg/g digestate
- Volatile Fatty acids 56.95-84.17 mg/g digestate
- Phenolic compounds 2.50-4.61 mg/g digestate



# Alkaline pretreatment

Exp	Time (h)	NaOH (%)	Cellic C Tec 2 ( $\mu\text{L/g}$ )
B1	1	2	100
B2	1.5	4	100
B3	1	4	100
B4	1.5	2	100
B5	1	2	400
B6	1.5	4	400
B7	1	4	400
B8	1.5	2	400
B9	1.25	3	250



# Alkaline pretreatment

$$SG_{\text{NaOH}} = 3.975 + 32 * t_{\text{auto}} + 0.0525 * C_{\text{enz}}$$

↑ autoclaving time and/or enzyme loading ↑  
saccharification yield

Max  $SG_{\text{NaOH}} = 76\%$  for

**1.5 h** autoclaving time

**2%** NaOH and

**400**  $\mu\text{L}$  CellicCTec2/ g cellulose



# Acidic pretreatment

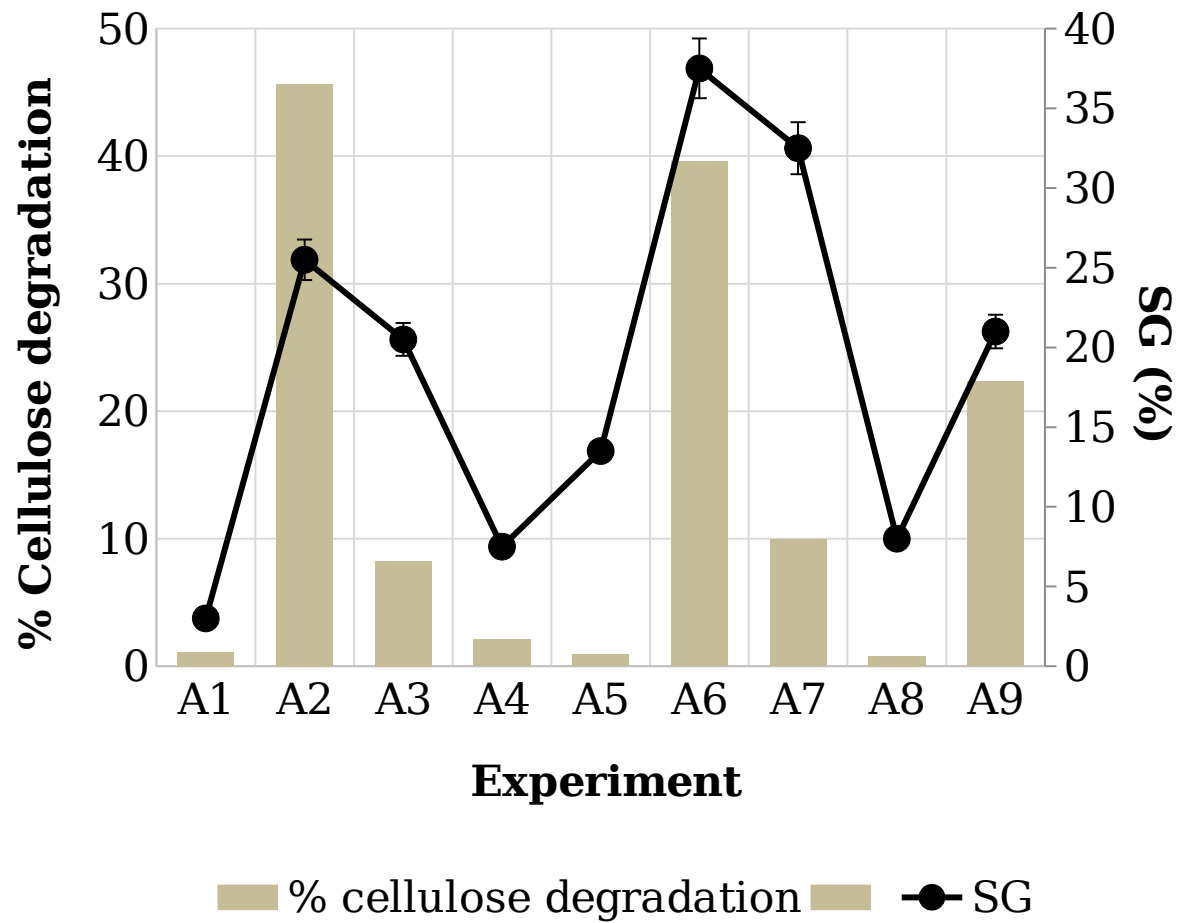
$t_{\text{auto}}$ (h)	$\text{H}_2\text{SO}_4$ (%)	%TS hydrolysis		%cellulose degradation		%AIL degradation		%ASL degradation		%hemicellulose degradation	
1	1	20.37	± 0.2 2	23.79	± 6.25 9	9.73	± 0.4 9	11.11	± 1.97 9	46.47	± 3.45 7
2	3	32.02	± 2.3 2	15.32	± 14.0 4	5.38	± 0.3 8	40.68	± 1.81 8	66.85	± 9.56 5
1.5	2	25.12	± 1.1 5	19.85	± 5.32 5	7.52	± 0.2 9	29.98	± 2.01 9	59.17	± 5.21 7
1	3	20.88	± 0.8 9	35.58	± 7.29 8	5.61	± 5.1 2	28.83	± 3.87 2	65.62	± 1.45 2
2	1	13.18	± 0.8 7	43.75	± 16.5 7	0.69	± 1.2 1	6.64	± 3.63 1	49.57	± 8.85 7

- Insoluble lignin remained almost unaffected
- Volatile Fatty acids 9.92-25.89 mg/g digestate
- Phenolic compounds 0.3-0.54 mg/g digestate



# Acidic pretreatment

Exp	Time (h)	H <sub>2</sub> SO <sub>4</sub> (%)	Cellic C Tec 2 (μL/g)
A1	1	1	100
A2	1.5	3	100
A3	1	3	100
A4	1.5	1	100
A5	1	1	400
A6	1.5	3	400
A7	1	3	400
A8	1.5	1	400
A9	1.25	2	250



# Acidic pretreatment

$$SG_{H_2SO_4} = 11.35 + 0.03 * C_{enz}$$

↑ enzyme loading → ↑ saccharification yield

Max  $SG_{H_2SO_4} = 39\%$

for

**1.5 h** autoclaving time

**3%**  $H_2SO_4$  and

**400  $\mu$ L** CellicCTec2/ g cellulose

Harshest pretreatment condition



# *Conclusions*



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# Conclusions

- Acid pretreatment along with enzymatic hydrolysis was found to yield low sugars recoveries (2-39%).
- Alkaline pretreatment and enzymatic hydrolysis is a better approach with elevated saccharification yields reaching up to 76%.
- NaOH pretreatment presented in all cases much better performance on saccharification yields than acidic pretreatment.



New integrated system that combines ethanol production with anaerobic digestion simultaneously producing energy in the form of methane and ethanol and improving the overall energy balance

# Acknowledgements



The authors acknowledge funding through European Horizon 2020 NoAW (No Agro Waste, Grant no. 688338) project for supporting this work.

**THANK YOU  
FOR YOUR  
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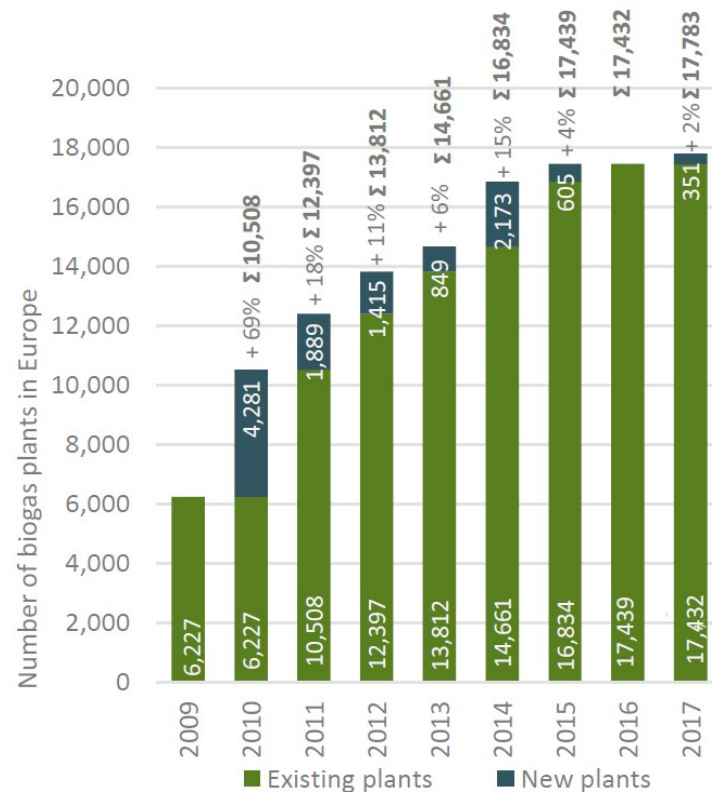
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# Anaerobic Plants in Europe

The number of biogas plants in Europe has greatly increased. Between 2009 (earliest EBA data) and 2017, the total number of biogas plants rose from 6,227 to 17,432 installations (+11,205 units).

Most of that growth derives from the increase in plants running on **agricultural substrates**: these went from 4,797 units in 2009 to 12,721 installations in 2017 (+7,924 units, **67%** of the total increase).

Agricultural plants are then followed by biogas plants running on sewage sludge (2,854 plants), landfill waste (1,374 units) and various other types of waste (688 plants).



[http://biogas.org.rs/wp-content/uploads/2018/12/EBA\\_Statistical-Report-2018\\_European-Overview-Chapter.pdf](http://biogas.org.rs/wp-content/uploads/2018/12/EBA_Statistical-Report-2018_European-Overview-Chapter.pdf)





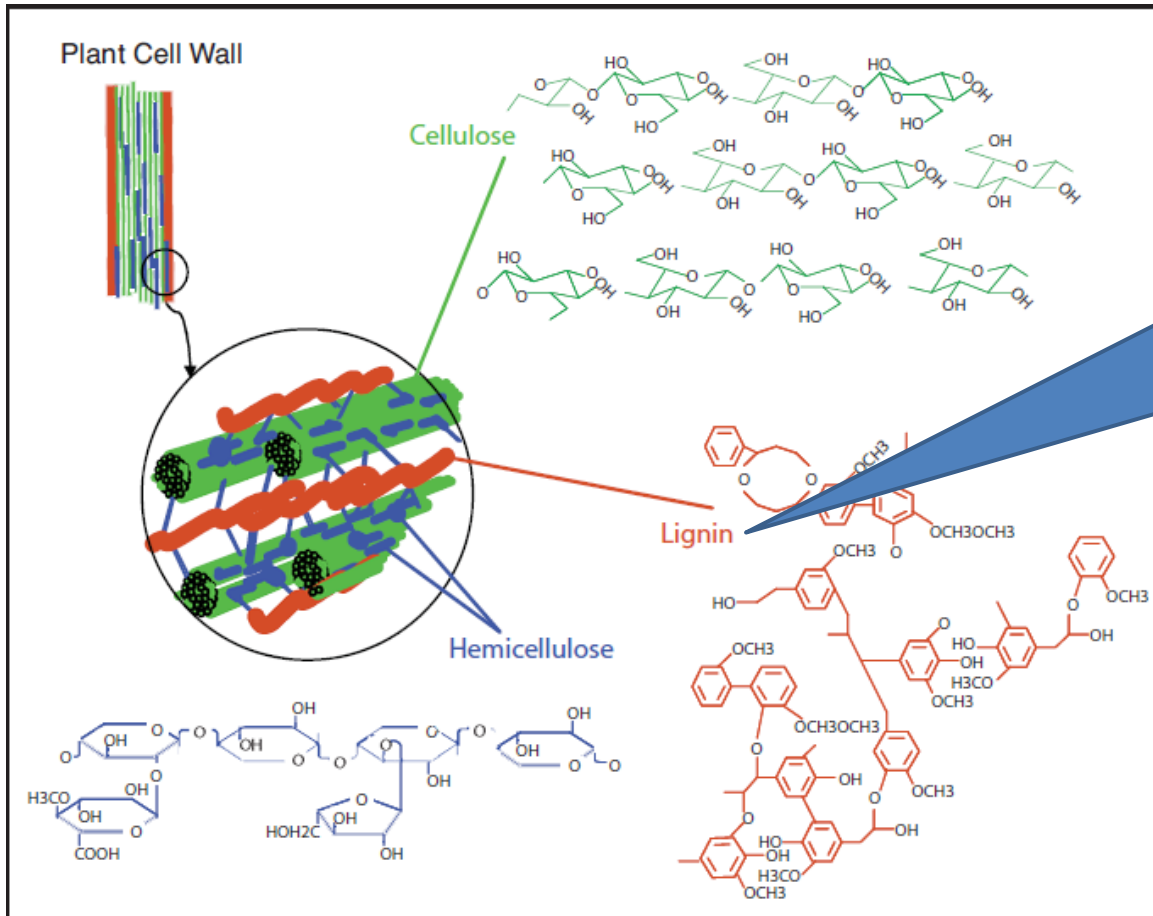
# Challenges of digestate

It is vital that digestate is seen in a holistic way as part of an overall materials processing and re-use system.

- The use of downstream processing to treat digestate may ***consume more energy*** than is likely to be generated by the AD facility.
- Digestate is ***difficult to manage*** due to its fertilising properties, format and high water content.
- There is ***lack of a legal framework***. Many European countries do not have appropriate (if any) legislation concerning digestate, resulting in legal barriers to the use of waste material, its conversion into products or its export abroad.
- There is ***lack of information***. Most farmers are poorly informed (or even misinformed) about the benefits of digestate and other organic fertilisers, often making them hesitant about spreading them on their land. Public authorities should make a conscious effort to explain the advantages of digestate and the adequate management of local resources to build confidence on its use.



# Agricultural waste -Lignocellulosic material



The major barrier to the enzymatic hydrolysis of cellulose towards the production of fermentable



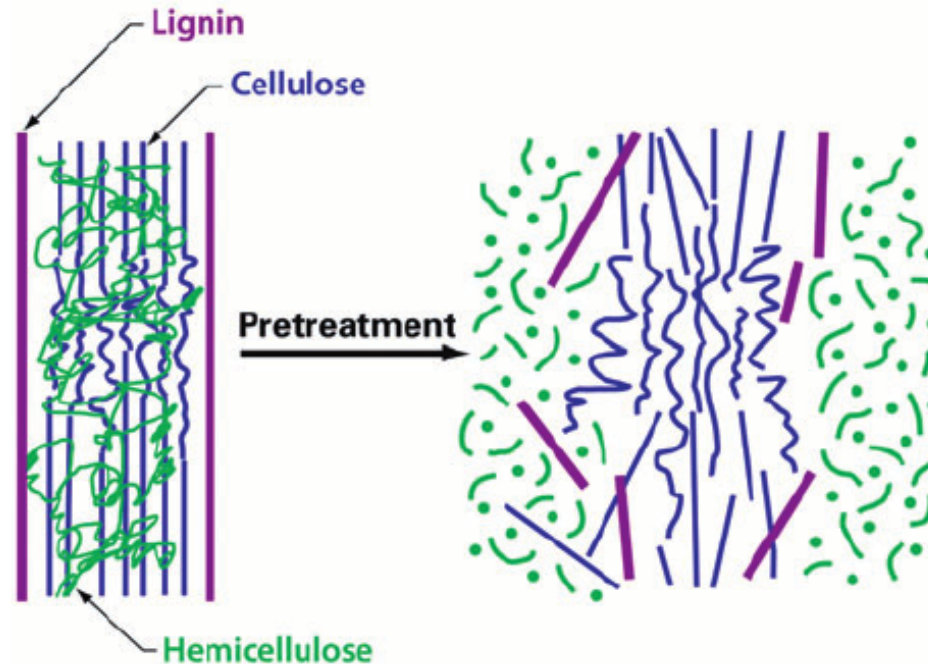
<https://www.e-education.psu.edu/egee439/node/606>

corn stover on a field credit: USDOE-NRE  
<https://www.greenoptimistic.com>



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# Lignocellulosic material



**Removal of lignin** is favorable to reducing the recalcitrance of lignocellulose for enzymatic attack.