Jaroslava Švarc-Gajić
University of Novi Sad

Jointly elaborated by: Université D'Aix Marseille

AGRIMA

Superheated water – a powerful tool for biomasss valorization

Erasmus+
Enriching lives, opening minds.
Higher education

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Agri-food Waste Management for Sustainable bio-economy through Higher Education curricula and upskilling

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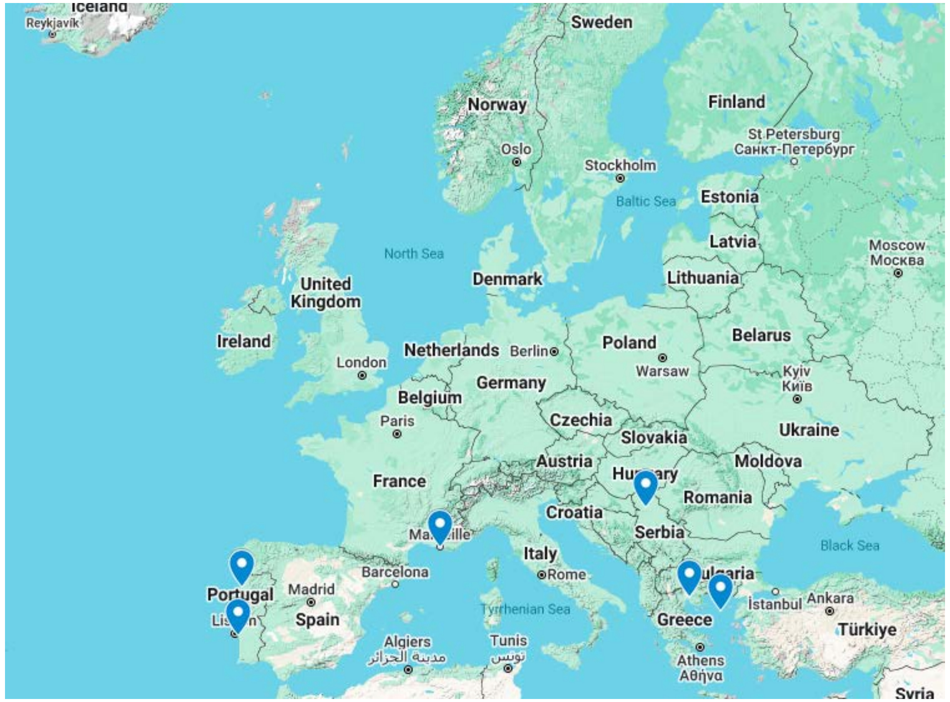
Goals

AGRIMA aims to foster universities' **capacity building** for the **green transition** through **innovative practices and higher education curricula updating in agri-food waste management** for the **circular bioeconomy**.

AGRIMA addresses:

1. **Advancing pedagogical methods** for industrial agri-food waste valorisation **based on business-academia synergies**.
2. **Integrating citizen science** in bio-economy-enhanced waste valorisation as a means of **civic engagement and environmental advocacy**.

Partners



Aix Marseille université
Socialement engagée

AVIPE
Associação de Viticultores do Concelho de Palmela

P.PORTO

UNIVERSITY OF THE AEGEAN

UNIVERSITY OF NOVI SAD

Web2Learn
Open, social learning

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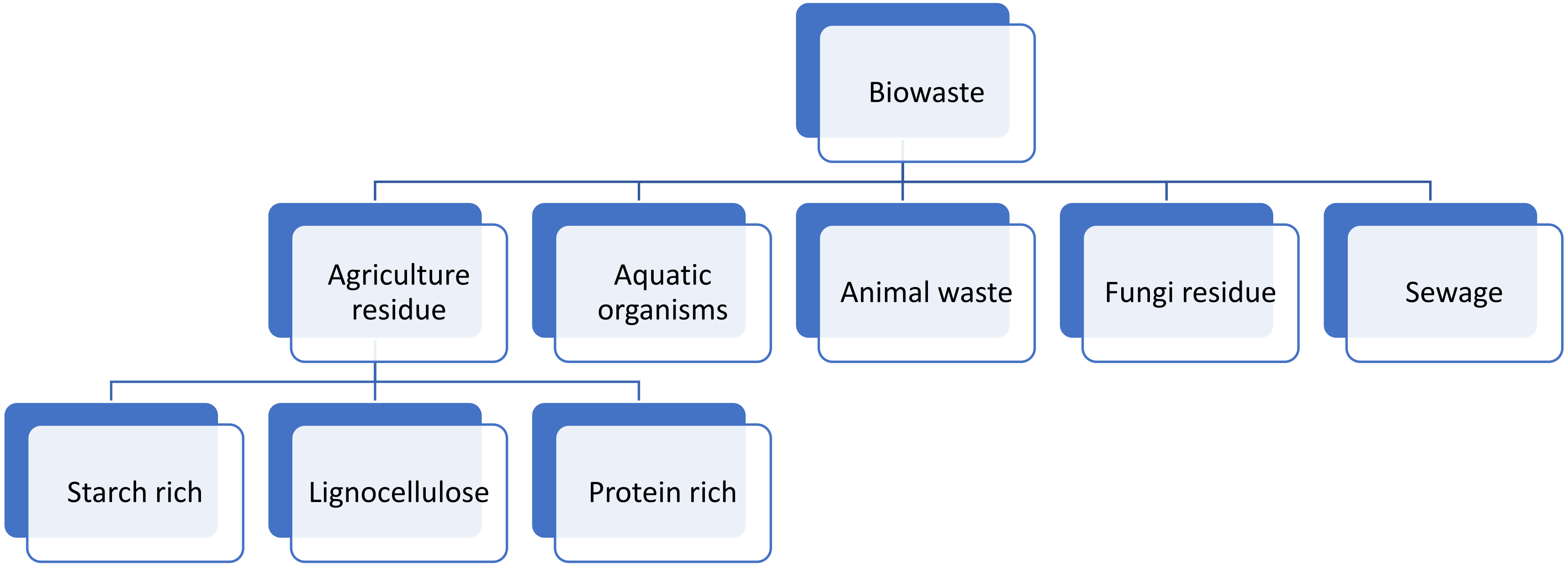

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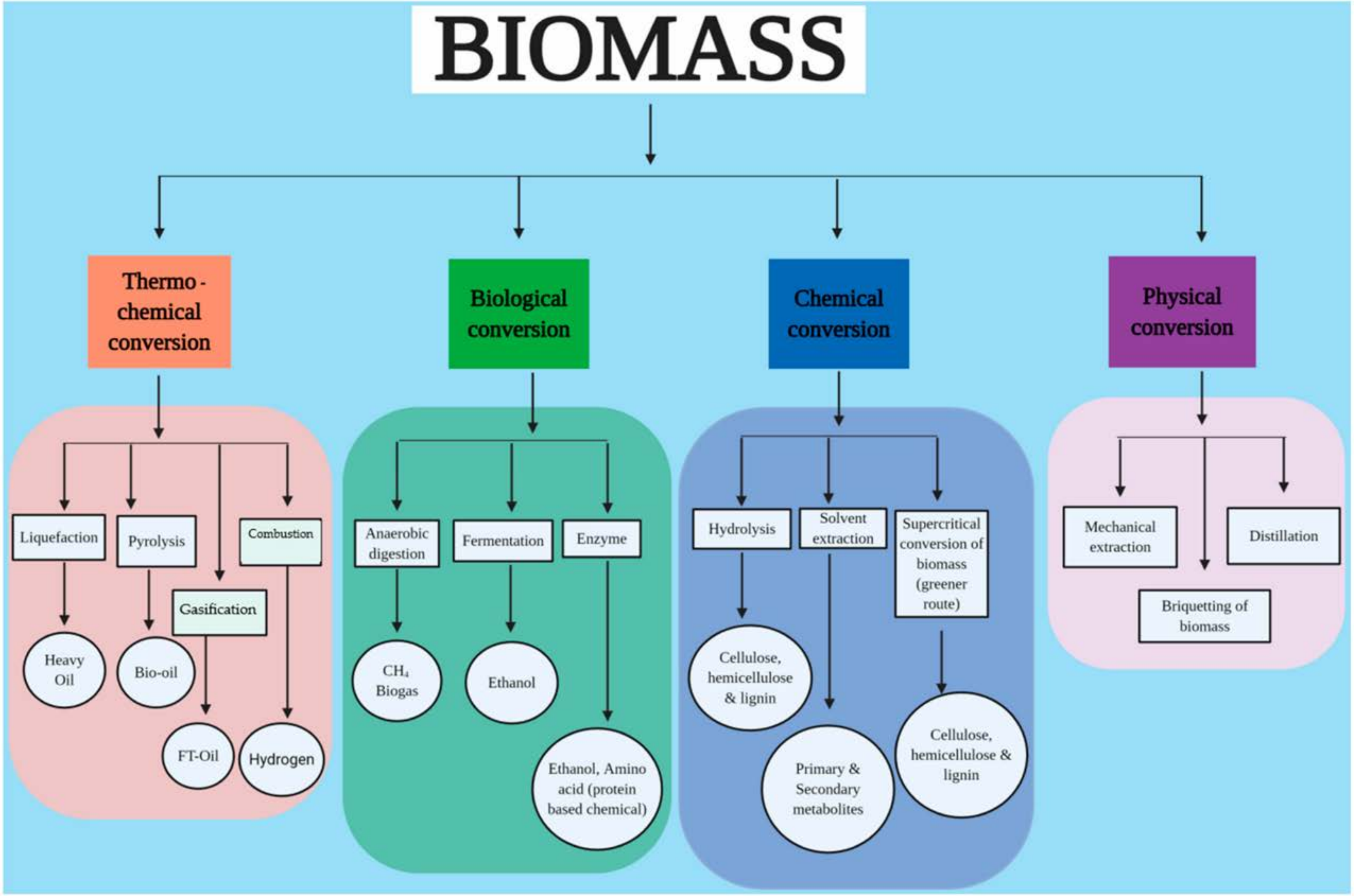

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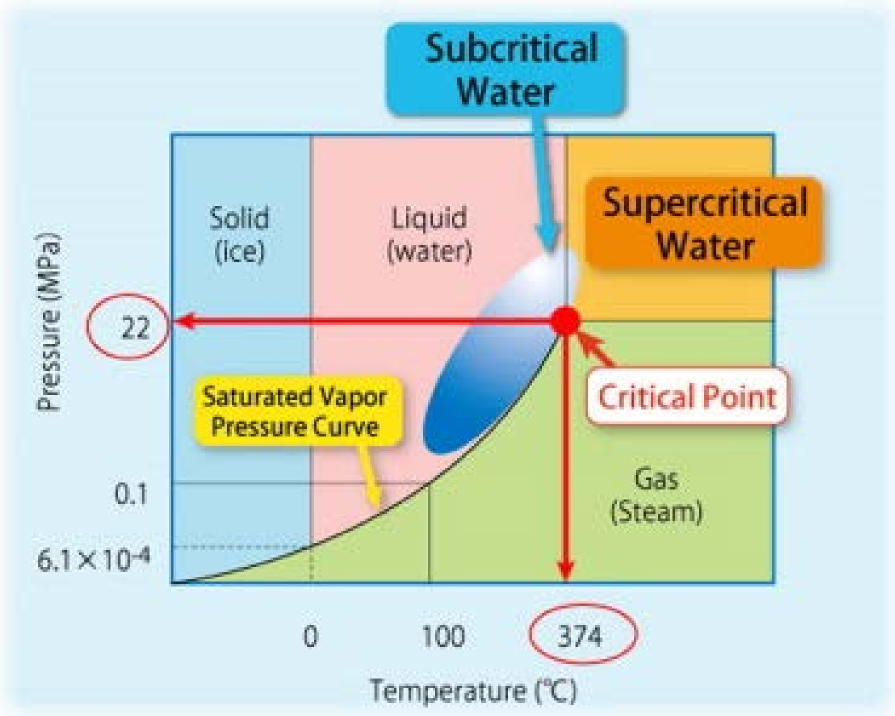
Sub- and supercritical water



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Hot pressurized water

- Solvent
- Reactive medium
- Extraction&Decomposition

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Added-value compounds by sub-/supercritical water

- Organic acids: acetic, lactic, formic etc.
- Amino acids
- Pectin, oligosaccharides, sugars, erythrose
- Fatty acids
- Essential oils
- Compounds of pharmacological interest:
  - Chitin
  - Chitosan
  - Glucosamine
  - Caffeine
  - Saponins
  - Anti-oxidant, anti-inflammatory, anti-microbial, neuroprotective, antidiabetic compounds, etc.

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Subcritical water as a solvent

- Water at room temperature - good solvent for polar compounds
- Polarity decreases drastically with heating
- Solubility of non-polar compounds increases for one order of magnitude with temperature increase of 50 °C
- At 350 °C solubility of benzo(e)pyrene increases 25 000 000 fold
- Solubility of nerol: 0.02 % (25°C); 0.5 % (200°C)

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The reactivity of sub- and supercritical water

- Alkylation reaction (Friedel-Crafts reaction)
- Condensation reactions (Claisen-Schmidt condensation)
- Coupling reactions
- Cyclization reactions
- Decarboxylation reactions
- Diels-Alder reactions
- Disproportination reactions
- Elimination reactions
- Ene reactions
- Izomerization reactions
- Hydrogenation/Dehydrogenation reactions
- Hydrolysis
- Oxidation
- Rearrangement reactions
- Transformation reactions
- Maillard reactions
- Caramelization reactions

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Bioactivity of the chokeberry stems extracts obtained by subcritical water (130 °C, 20 min, 35 bars)

Antioxidant activity (IC_{50} values)	
DPPH assay (mg/mL)	0.10±0.02*
Reducing Power assay (mg/mL)	1.25±0.03
Enzyme-inhibitory activity	
α-Amylase inhibition (mmol ACAE/g)[a]	0.59±0.01
α-Glucosidase inhibition (mmol ACAE/g)[a]	7.50±0.32
Tyrosinase-inhibition (mg KAE/g)[b]	15.87±3.82
Elastase inhibition (mmol/ CAE g)[c]	3.12±0.39
AchE Inhibition (mg GALAE/g)[d]	0.71±0.03

*mean ± 2SD; [a]ACAE - Acarbose equivalents; [b]KAE - kojic acid equivalents.; [c]CAE – Catechin equivalent; [d]GALAE – Galantamine equivalent

Cvetanović, A., Švarc-Gajić, J., Zeković Z., Mašković, P., Djurović, S., Zengin, G., Delerue-Matos, C., Lozano-Sanchez, J., Jakisić, A. (2017): Chemical and biological insights on aronia stems extracts obtained by different extraction techniques: From wastes to functional products. Journal of Supercritical Fluids, 128, 173–181.

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Subcritical water as a reactive medium

- Pesticides (tembotrione, clomazone): 200 ◦C, 0.1% H_2O_2, 30 min
- Pharmaceuticals (ciprofloxacin, 17α-ethinyl estradiol): 200 ◦C, 0.001 mol/l of $KMnO_4$, 30 min
- Mycotoxins (zearalenone, deoxynivalenol, fumonisin B1): 200 ◦C, 100 min

Švarc-Gajić, J.; Brezo-Borjan, T.; Jakšić, S.; Despotović, V.; Finčur, N.; Bognár, S.; Jovanović, D.; Šojić Merkulov, D. Decomposition of Organic Pollutants in Subcritical Water under Moderate Conditions. Processes 2024, 12, 1293. https://doi.org/10.3390/pr12071293

Oil-seed cakes

- Pumpkin seed cake
- Flax seed cake
- Hemp seed cake

160°C
1 h
20 bars N_2, CO_2
water:sample = 1: 30
3 Hz

Amino-acids
Polyphenolics
Minerals

Švarc-Gajić, J., Morais, S., Delerue-Matos, C., Vieira, E., Spigno, G. Valorization potential of oilseed cakes by subcritical water extraction (2020): Applied Sciences, 10, 8815; doi:10.3390/app10248815

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Sample	Atmosphere	EY [%]	Total lipid content [g/100 g dry extract]	Total protein content [g/100 g dry extract]
Pumpkin	N_2	57.51±0.19	28.54±0.18	1.94±0.04
	N_2 + 0.05 mol/l HCl	60.10±0.18	**48.55±0.23**	3.14±0.06
	CO_2	57.13±0.19	38.87±0.22	**4.87±0.08**
Hemp	N_2	42.07±0.18	10.10±0.21	4.83±0.06
	N_2 + 0.05 mol/l HCl	40.45±0.18	**50.75±0.21**	6.59±0.09
	CO_2	44.23±0.17	32.04±0.18	**6.83±0.09**
Flax	N_2	50.33±0.18	6.97±0.10	1.30±0.01
	N_2 + 0.05 mol/l HCl	53.02±0.15	**32.87±0.18**	2.52±0.02
	CO_2	51.73±0.17	23.49±0.17	**2.84±0.03**

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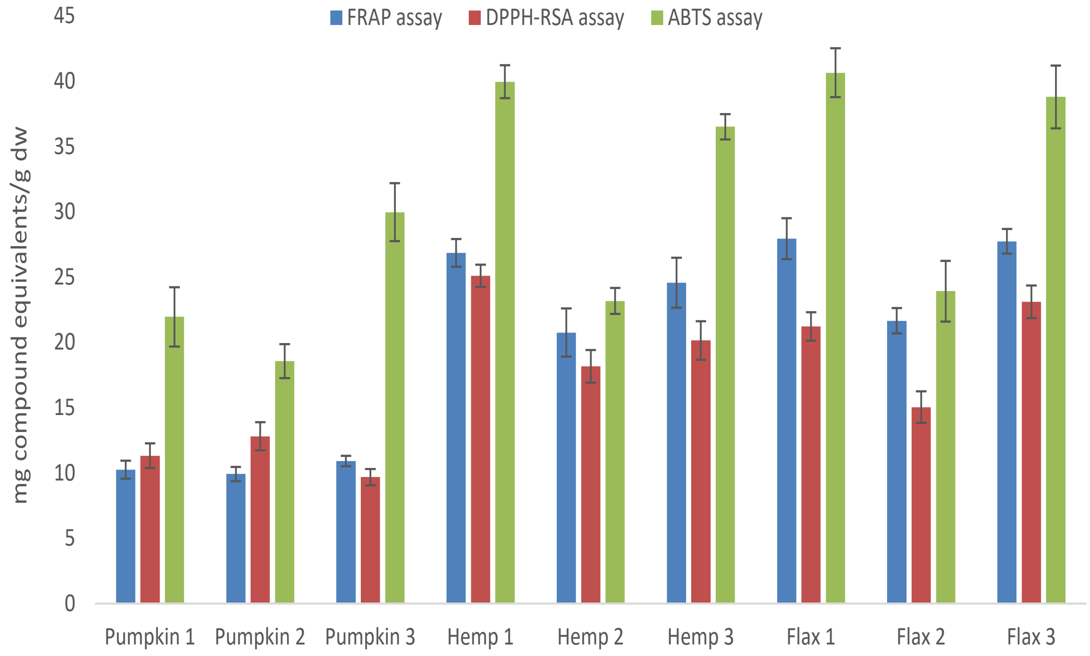
Sample	TPC [g GAE/100 g]	TFC [g RU/100 g]
Pumpkin 1	3.26±0.10	0.45±0.0001
Pumpkin 2	3.79±0.02	0.36±0.0023
Pumpkin 3	3.19±0.01	0.39±0.0129
Hemp 1	3.68±0.04	**1.03±0.0038**
Hemp 2	2.78±0.07	0.41±0.0023
Hemp 3	3.62±0.02	0.58±0.0099
Flax 1	3.56±0.08	0.93±0.0099
Flax 2	2.93±0.02	0.54±0.0008
Flax 3	**3.98±0.04**	0.70±0.0973

TPC – Total phenols content; TFC – Total flavonoids content

1-SWE: N_2, 1:30, 160°C, 20 bar, 1 h; 2-SWE: N_2, 0.05 mol/l HCl, 1:30, 160°C, 20 bar, 1 h; 3-CO_2, 1:30, 160°C, 20 bar, 1 h.



1-SWE: N_2, 1:30, 160°C, 20 bar, 1 h; 2-SWE: N_2, 0.05 mol/l HCl, 1:30, 160ºC, 20 bar, 1 h; 3-CO_2, 1:30, 160ºC, 20 bar, 1 h.

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Sample	K [mg/100 g]	Na [mg/100 g]	Ca [mg/100 g]	P [mg/100 g]
Pumpkin 1	24.89±0.13	0.46±0.03	3.72±0.06	2.29±0.03
Pumpkin 2	24.59±0.13	0.65±0.03	5.26±0.03	2.45±0.02
Pumpkin 3	27.90±0.11	1.53±0.06	6.45±0.05	2.36±0.02
Average ± 2 SD	25.79±0.12	0.88±0.04	5.14±0.05	2.37±0.02
Hemp 1	49.01±0.11	2.14±0.09	6.03±0.04	4.05±0.02
Hemp 2	52.50±0.11	2.87±0.08	7.86±0.08	4.21±0.02
Hemp 3	48.73±0.12	3.35±0.04	7.97±0.09	**4.53±0.03**
Average ± 2 SD	50.08±0.11	2.78±0.07	7.29±0.07	4.27±0.02
Flax 1	49.01±0.10	**6.16±0.05**	5.05±0.07	1.81±0.01
Flax 2	**57.09±0.10**	3.72±0.08	**10.19±0.11**	1.99±0.01
Flax 3	38.05±0.12	4.71±0.07	8.81±0.09	2.05±0.02
Average ± 2 SD	48.05±0.11	4.86±0.07	8.02±0.09	1.95±0.01

1-SWE: N_2, 1:30, 160ªC, 20 bar, 1 h; 2-SWE: N_2, 0.05 mol/l HCl, 1:30, 160ªC, 20 bar, 1 h; 3-CO_2, 1:30, 160ªC, 20 bar, 1 h.

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AA	Pumpkin 1[1]	Pumpkin 2	Pumpkin 3	Hemp 1	Hemp 2	Hemp 3	Flax 1	Flax 2	Flax 3
Asp#	0.06 ± <0.01	0.53 ± 0.02	4.43 ± 0.12	49.17 ± 1.26	70.45 ± 0.87	72.67 ± 0.14	3.81 ± 0.27	6.12 ± 0.45	5.00 ± 0.34
Glu#	2.50 ± 0.14	5.40 ± 0.08	7.34 ± 0.15	44.58 ± 0.14	41.98 ± 0.05	77.86 ± 0.19	3.03 ± 0.08	6.53 ± 0.14	5.88 ± 0.15
Ser	0.10 ± <0.01	0.19 ± <0.01	0.63 ± <0.01	15.84 ± 0.46	19.70 ± 0.02	26.50 ± 0.02	0.96 ± 0.03	2.14 ± 0.10	3.83 ± 0.05
Thr•	1.01 ± 0.03	1.54 ± 0.05	5.11 ± 0.01	8.94 ± 0.02	28.34 ± 0.01	34.70 ± 0.12	1.18 ± 0.06	2.32 ± 0.05	4.09 ± 0.08
His•	0.81 ± 0.02	1.44 ± 0.06	5.55 ± 0.03	6.09 ± 0.08	13.38 ± 0.01	20.94 ± 0.01	0.77 ± 0.04	1.79 ± 0.02	2.63 ± 0.05
Gly#	0.50 ± <0.01	1.00 ± 0.02	4.82 ± 0.02	0.02 ± <0.01	0.07 ± <0.01	0.20 ± <0.01	0.73 ± 0.02	4.71 ± 0.06	6.08 ± 0.05
Gln	0.74 ± 0.01	1.19 ± 0.01	5.31 ± 0.04	0.05 ± <0.01	0.99 ± <0.01	2.60 ± 0.01	0.70 ± 0.01	1.69 ± 0.01	2.87 ± 0.01
Asn	0.01 ± <0.01	0.03 ± <0.01	0.15 ± <0.01	1.35 ± <0.01	0.53 ± <0.01	0.55 ± <0.01	0.01 ± <0.01	0.08 ± <0.01	0.71 ± 0.01
Arg•	0.99 ± 0.03	1.73 ± 0.03	3.69 ± 0.04	39.27 ± 0.26	85.14 ± 1.03	89.01 ± 0.05	0.92 ± 0.08	3.58 ± 0.11	5.17 ± 0.07
Ala#	0.31 ± 0.03	0.97 ± 0.02	2.09 ± 0.01	10.77 ± 0.08	15.82 ± 0.03	17.83 ± 0.01	0.20 ± 0.02	0.76 ± 0.02	1.28 ± 0.02
Tyr	0.81 ± 0.04	1.38 ± 0.02	3.18 ± 0.01	1.50 ± 0.01	1.05 ± <0.01	2.73 ± 0.01	0.82 ± 0.03	1.92 ± 0.01	4.86 ± 0.01
Lys•	4.81 ± 0.04	7.66 ± 0.05	12.37 ± 0.10	10.68 ± 0.14	15.88 ± 0.04	16.46 ± 0.01	0.94 ± 0.02	2.12 ± 0.04	2.78 ± 0.03
Val•	1.80 ± 0.01	0.47 ± 0.01	1.54 ± 0.02	2.85 ± 0.04	5.54 ± 0.02	4.10 ± 0.01	0.26 ± 0.01	0.35 ± <0.01	1.79 ± 0.02
Met•	0.76 ± 0.01	0.32 ± 0.02	1.87 ± <0.01	1.35 ± 0.05	0.99 ± 0.01	0.20 ± <0.01	0.74 ± 0.05	1.76 ± 0.02	3.52 ± 0.01
Trp•	0.17 ± <0.01	0.41 ± <0.01	1.02 ± <0.01	0.29 ± <0.01	0.46 ± <0.01	0.48 ± <0.01	0.23 ± <0.01	0.86 ± <0.01	1.25 ± 0.03
Phe•	1.20 ± 0.01	2.14 ± 0.03	3.01 ± 0.01	1.93 ± 0.01	1.85 ± 0.01	3.35 ± 0.01	1.21 ± 0.01	2.49 ± 0.03	2.95 ± 0.01
Ile•	0.64 ± 0.04	1.07 ± 0.04	2.78 ± 0.05	4.30 ± <0.01	6.66 ± 0.02	7.31 ± 0.02	0.85 ± 0.04	1.97 ± 0.01	2.19 ± 0.02
Leu•	0.74 ± 0.05	1.26 ± 0.08	5.41 ± 0.06	13.19 ± 0.08	9.62 ± 0.05	24.18 ± 0.03	1.18 ± 0.03	2.87 ± 0.05	5.31 ± 0.07
Hyp	0.37 ± 0.02	0.94 ± 0.01	2.82 ± 0.03	0.63 ± <0.01	0.46 ± <0.01	1.37 ± 0.02	0.20 ± 0.01	0.73 ± 0.02	1.48 ± 0.01
Pro	2.07 ± 0.02	1.89 ± 0.02	1.96 ± 0.05	6.71 ± 0.01	3.10 ± <0.01	6.49 ± 0.05	0.10 ± <0.01	1.26 ± 0.03	1.56 ± 0.02
Σ AA	20.40 ± 0.58	32.56 ± 0.77	75.09 ± 2.91	219.50 ± 1.55	321.99 ± 1.05	409.51 ± 1.12	18.84 ± 0.59	46.04 ± 1.01	65.23 ± 0.65
% EAA•	58.58 ± 0.45	53.13 ± 1.02	51.49 ± 0.47	22.60 ± 0.58	25.69 ± 0.89	27.28 ± 0.54	39.06 ± 0.20	35.91 ± 0.79	40.66 ± 0.74
% FAA#	16.56 ± 0.75	24.30 ± 0.52	24.88 ± 0.42	47.63 ± 0.50	39.85 ± 0.89	41.16 ± 0.74	41.20 ± 0.38	39.35 ± 0.40	27.95 ± 0.52

[1]1-SWE: N2, 1:30, 160°C, 20 bar, 1 h; 2-SWE: N2, 0.05 mol/l HCl, 1:30, 160°C, 20 bar, 1 h; 3-CO2, 1:30, 160°C, 20 bar, 1 h.

Values are mean ± SD (n = 4)

EAA •, essential amino acids; FAA #, flavor amino acids; ΣAA, sum of amino acids.

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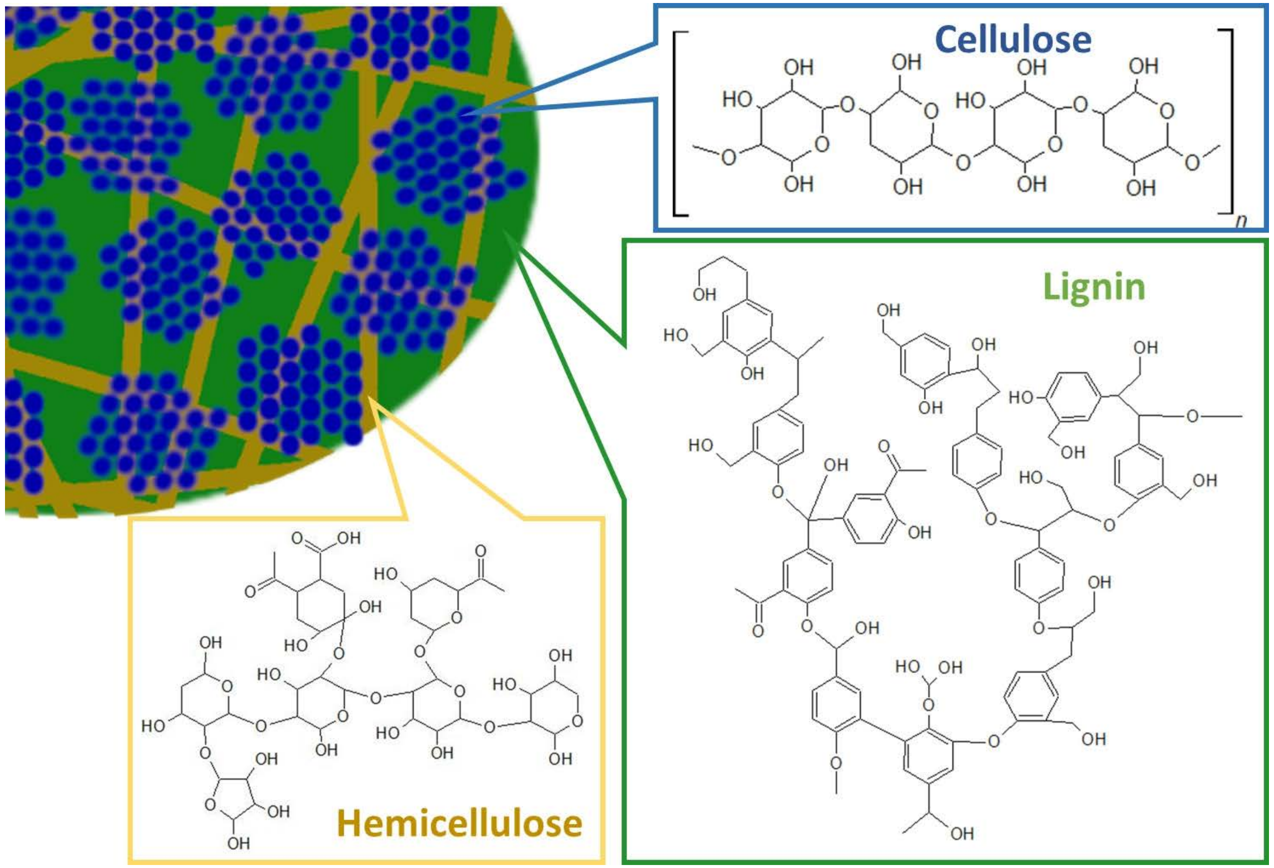
Lignocellulose Biomass



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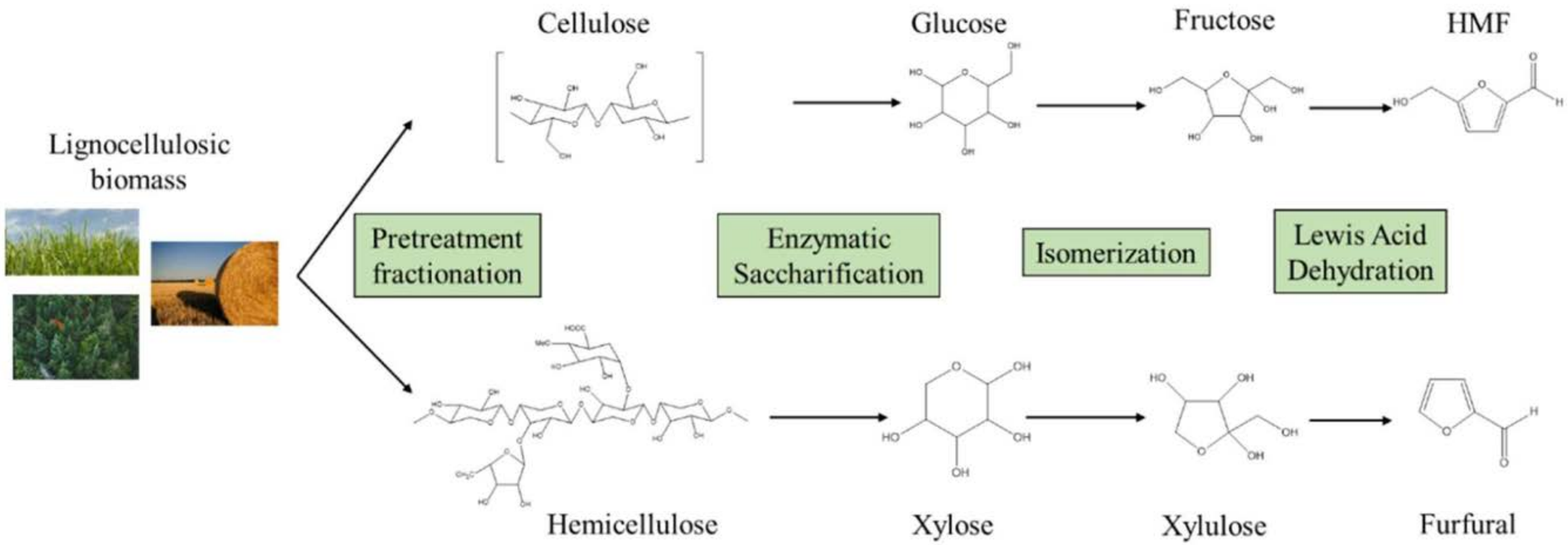
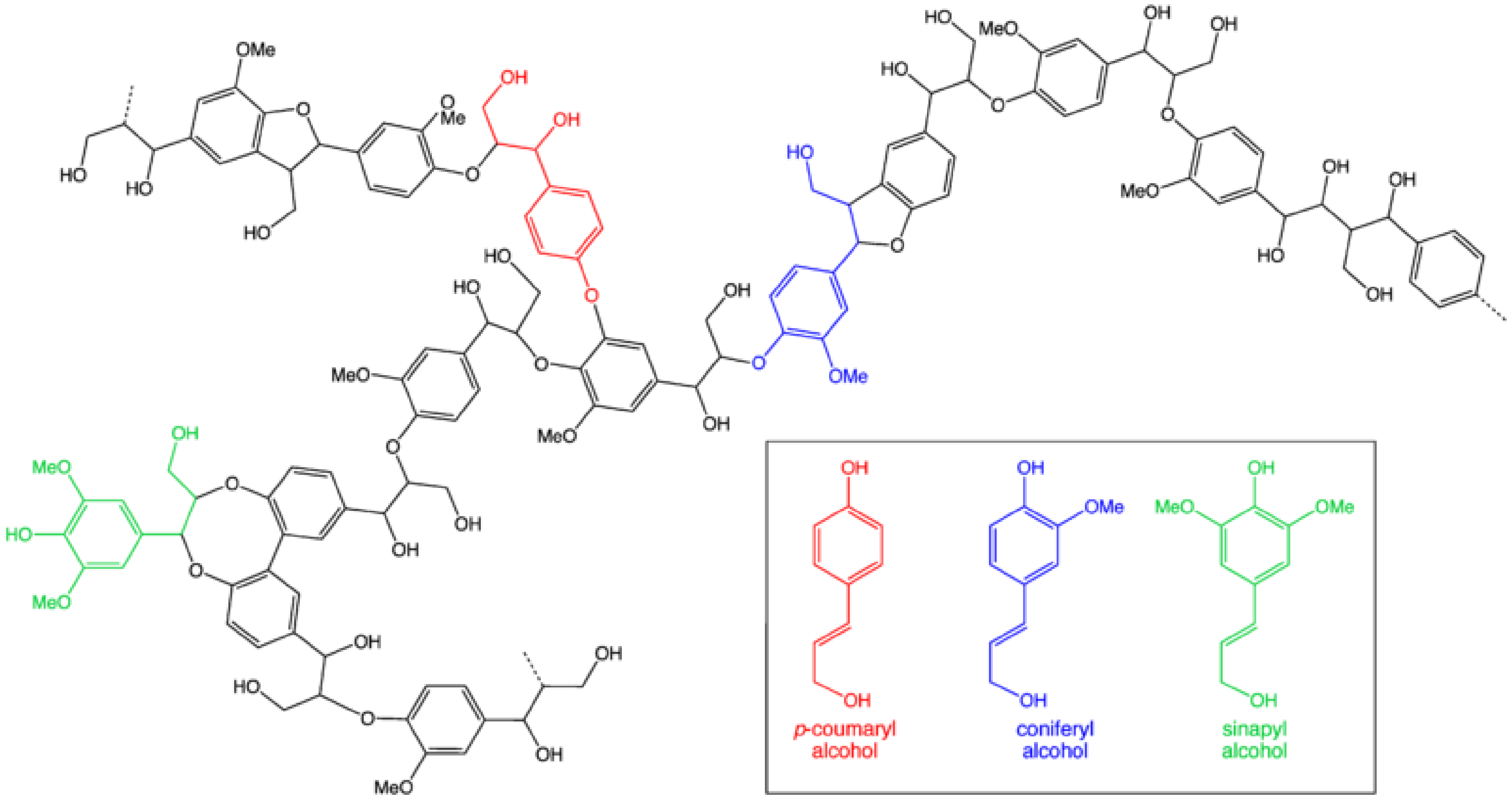

Lignocellulosic biomass
Cellulose
Glucose
Fructose
HMF
Pretreatment fractionation
Enzymatic Saccharification
Isomerization
Lewis Acid Dehydration
Hemicellulose
Xylose
Xylulose
Furfural

Lignin structure



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Lignin uses

- Binder in laminated and composite wood products
- Asphalt binder
- Oil
- Thermoplastics

Lignin for platform molecules

- Hydrothermal liquefaction
- Enzymatic conversion
- Pyrolysis
- Gasification
- Solvolysis
- Catalytic oxidative depolymerisation
- Hydrocracking (catalytic reduction)

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Lignin conversion by supercritical water

- Mechanism:

  1. Hydrolysis
  2. Dealkylation
  3. Cross-linking reactions

- Catalysts: zeolites, amorphous silica-alumina, KOH, NaOH

- Hydrogenation catalysts: cobalt, tungsten, palladium, nickel, molybdenum, Ni-W, carbon-supported Pd, Ru, $Ru(PPh_3)_3Cl_2$

- Oxidation catalysts: Pd/Al_2O_3, $Cu\text{-}Ni/Al_2O_3$, CuO, $CuSO_4$, $FeCl_3$, $MnSO_4$

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Kraft Lignin

- 180˚C
- 1h
- Lignin:water = 1:30
- 3 Hz

	Total phenols (mg GAE/g ± SD)	
Catalyst	N_2	CO_2
Without catalyst	35.30 ± 0.31	42.25 ± 0.63
0.5% Phenol	153.09 ± 1.29	168.48 ±0.34
1% HCl	112.85 ± 0.27	122.82 ± 0.28
1% NaOH	**336.07 ± 1.05**	**240.81 ± 2.95**
0.2% Zeolite	34.52 ± 0.03	35.30 ± 0.41
0.2% TiO_2	35.55 ± 0.51	37.52 ± 0.69
0.2% ZrO_2	34.72 ± 0.70	45.01 ± 0.73
0.2% CeO_2	31.20 ± 0.25	35.44 ± 0.13
0.2% (20% ZrO_2 + 80% CeO_2)	33.59 ± 0.88	34.30 ± 0.30

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Other Aromatics (mg RE/g ± SD)		
Catalyst	N_2	CO_2
Without catalyst	0.94 ± 0.01	5.11 ± 0.11
0.5% Phenol	1.26 ± 0.00	1.21 ± 0.12
1% HCl	3.71 ± 0.12	6.29 ± 0.12
1% NaOH	**173.43 ± 0.80**	**103.06 ± 0.91**
0.2% Zeolite	1.15 ± 0.01	0.97 ± 0.02
0.2% TiO_2	1.96 ± 0.01	1.21 ± 0.03
0.2% ZrO_2	1.16 ± 0.01	1.01 ± 0.02
0.2% CeO_2	0.97 ± 0.01	0.92 ± 0.02
0.2%(20% ZrO_2 + 80% CeO_2)	1.24 ± 0.01	1.00 ± 0.01

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T (°C)	Total Phenols (mg GAE/g)		Other Aromatics (mg RE/g)	
	N_2	CO_2	N_2	CO_2
120	341.88 ± 0.71	164.88 ± 1.40	114.29 ± 0.54	58.48 ± 1.15
150	**426.01 ± 0.35**	249.00 ± 0.61	**209.43 ± 4.41**	125.21 ± 0.32
180	336.07 ± 1.05	240.81 ± 2.95	173.43 ± 0.80	103.06 ± 0.91
200	387.70 ± 0.36	**317.54 ± 1.31**	160.41 ± 0.00	**205.67 ± 3.03**
220	399.95 ± 2.49	289.31 ± 3.06	160.06 ± 0.37	193.65 ± 2.18

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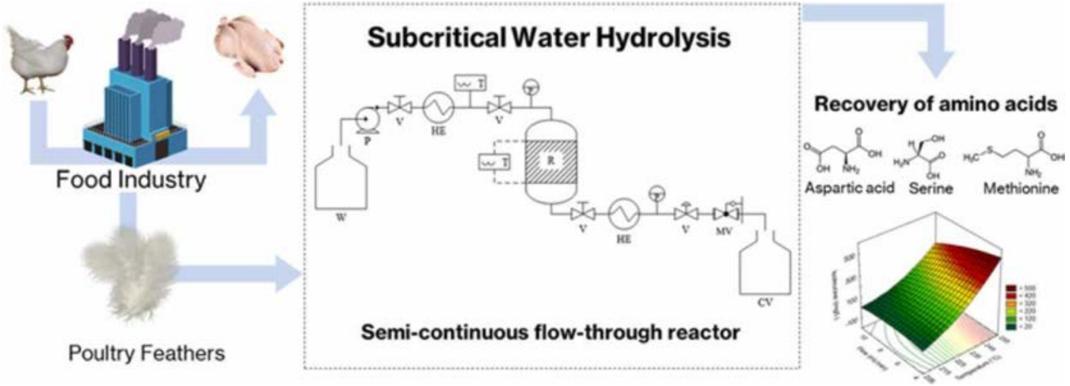
Feathers to amino-acids

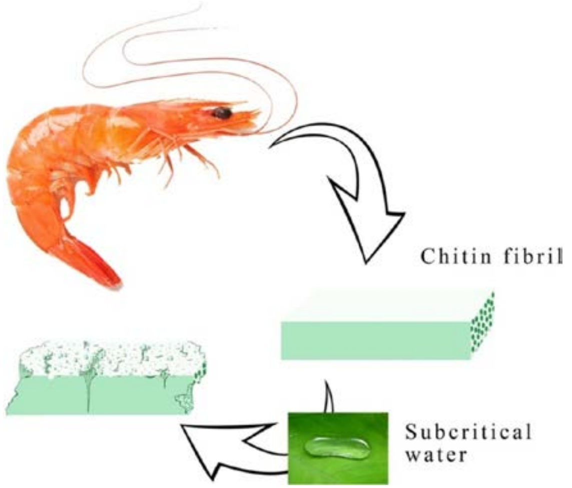
- Chicken feathers: 210–250 °C, 150 bars, flow rates 5–15 ml/min
- The highest hydrolysis temperature resulted in the highest concentrations of valine, methionine, tryptophan, phenylalanine, isoleucine, leucine, and lysine
- Threonine, histidine, and arginine were obtained more effectively at lower temperatures



Henrique Di Domenico Ziero, Larissa Castro Ampese, William G. Sganzerla, Paulo C. Torres-Mayanga, Michael T. Timko, Solange I. Mussatto, T. Forster-Carneiro, Subcritical water hydrolysis of poultry feathers for amino acids production, The Journal of Supercritical Fluids, Volume 181, 2022, 105492, ISSN 0896-8446, https://doi.org/10.1016/j.supflu.2021.105492.

Chitin

- Component of cell walls in fungi
- Exoskeletons of arthropods (crustaceans and insects), molluscs
- Polysaccharide - polymer of N-acetylglucosamine
- Uses: fertilizer, edible films, coatings



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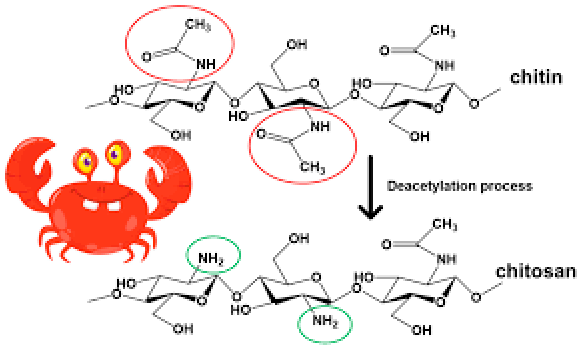
Chitosan

- Chemical or enzymatic deacetylation of chitin
- Uses: biodegradable plastic, biopesticide, antimicrobial, antioxidant activities, drug delivery, biotechnology, bio-nanotechnology, food technology, regenerative medicine, cancer therapy, agriculture, environmental protection



Crab shells to useful resources

- Subcritical water at 260-320 °C:
  - Decomposition of proteins, pigments, extraction of lipids
  - $CaCO_3$ removal by 2 M HCl, 48h
  - Extracts high in organic and amino-acids
- 180 – 220 °C in acetic acid – one stage process for 95-100% chitin purity:
  - 320 – 350 °C : chitin decomposition to glucosamine and erythrose
  - Subcritical 10% NaOH at 220 °C for 60 min 74% deacetylation to chitosan

→ **Chitin** → **Chitosan**

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Nakamura H. et al. Conversion of crab shell to useful resources using subcritical water treatment.

Chicken manure

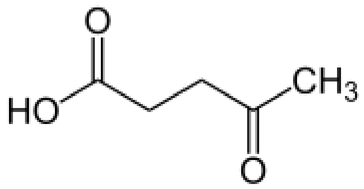
- Humic acid and fulvic acid: highest (3647.2×10^{-6} g/kg) content at 250 °C, at a pressure of 50-60 bars

- Exhausted solid phase: high content of organic carbon, phosphorus, potassium, and microelements

Sushkova S, Minkina T, Chaplygin V, Nevidomskaya D, Rajput V, Bauer T, Mazarji M, Bren AB, Popov I, Mazanko M. Subcritical water extraction of organic acids from chicken manure. J Sci Food Agric. 2021 Mar 15;101(4):1523-1529. doi: 10.1002/jsfa.10768. Epub 2020 Sep 19. PMID: 32869301.

Hazelnut shell

- Levulinic acid:
  - Cosmetics
  - Resins
  - Plasticizer
  - Textile
  - Feed
- Maximum cellulose conversion (82%) was achieved at 230°C and 180 min of reaction time in 25 mM of H_2SO_4



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Aslı Yüksel Özşen. Front. Chem., 29 January 2020 Sec. Green and Sustainable Chemistry. Volume 8 - 2020 | https://doi.org/10.3389/fchem.2020.00024

Biofuel production by hydrothermal liquefaction

Biomass sources:

- Wood and wood waste
- Aquatic plants
- Algae
- Weeds
- Agricultural by-products and waste
- Marine (fish, crab shell, bones, scallop viscera, squid entrails)
- Food processing waste (peel, stems, leaves, seed)

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Biofuel production by superheated water

- Hydrothermal carbonization: 170-260 °C, 20–70 bars
- Hydrothermal liquefaction: 200–370 °C, 15–190 bars
- Hydrothermal gasification: production of hydrogen-rich syngas (H_2 + CO): >250 °C, 100–200 bars

Selvakumar Thiruvenkadam, Shamsul Izhar, Hiroyuki Yoshida, Michael K. Danquah, Razif Harun, Process application of Subcritical Water Extraction (SWE) for algal bio-products and biofuels production, Applied Energy, Volume 154, 2015, Pages 815-828, ISSN 0306-2619, https://doi.org/10.1016/j.apenergy.2015.05.076.

Biocrude from algae

- Organic, liquid product produced from liquefication

- Biocrude production is dependent on the organic fraction of the algae

- Low heating value, high viscosity, corrosive properties, high solid content, chemical instability

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Catalysts increase the yield in biocrude production from algae:

- Na_2CO_3
- HCOOH
- CH_3COOH
- KOH
- Pt/Al_2O_3
- Co/Al_2O_3
- Ni/Al_2O_3

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Biocrude from different algae

- *Dunaliella tertiolecta:* 360 °C
- *Chlorella pyrenoidosa* and planktonic algae: 260 °C - 280 °C
- *Spirulina platensis* (microalgae) and *Entermorpha prolifera* (macroalgae): 340 °C
- *Nannochloropsis* sp.: biocrude yields at subcritical temperatures (300 °C) were higher compared to the yields at 400 °C

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Selvakumar Thiruvenkadam, Shamsul Izhar, Hiroyuki Yoshida, Michael K. Danquah, Razif Harun, Process application of Subcritical Water Extraction (SWE) for algal bio-products and biofuels production, Applied Energy, Volume 154, 2015, Pages 815-828, ISSN 0306-2619, https://doi.org/10.1016/j.apenergy.2015.05.076.

Combined processes with subcritical water

- Pretreatment of jabuticaba (*Myrciaria cauliflora*) biowaste (at 180 °C, 150 bars, flow rate of 10 ml/min, 45 minutes):
  - glucose (5.78 g/l), fructose (3.63 g/l), arabinose (1.82 g/l), cellobiose (1.28 g/l)
- Semi-continuous anaerobic digestion:
  - methane yield 239.04 l/kg *vs*. 42.31 l/kg
  - 543 kWh/t of electricity, 2,243.17 MJ/t of heat

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Rafael Gabriel da Rosa, William Gustavo Sganzerla, Tiago Linhares Cruz Tabosa Barroso, Luiz Eduardo Nochi Castro, Mauro Donizetti Berni, Tânia Forster-Carneiro, Sustainable bioprocess combining subcritical wa pretreatment followed by anaerobic digestion for the valorization of jabuticaba (*Myrciaria cauliflora*) agro-industrial by-product in bioenergy and biofertilizer, Fuel, Volume 334, Part 1, 2023, 126698, ISSN 0016-2361, https://doi.org/10.1016/j.fuel.2022.126698.

Steam explosion and subcritical water treatment

- Brewer`s spent grains: steam explosion with CO_2 (150-250 bars, 120-180 °C)
- Subcritical water hydrolysis – 210 °C, 150 bars, 20 ml/min:
  - xylose, arabinose, glucose, cellobiose, HMF, furfural

→ **Sugars**

Nunes, L.F.; Ugalde, G.A.; Anschau, K.F.; Müller, E.I.; Tres, M.V.; Zabot, G.L.; Kuhn, R.C. Clean Production of Sugars from Brewer's Spent Grains Using Subcritical Water Hydrolysis and Steam Explosion. Sustain. Chem. 2024, 5, 308-323. https://doi.org/10.3390/suschem5040021