HYDROTHERMAL CARBONIZATION OF FOOD WASTE: INFLUENCE OF FOOD WASTE COMPOSITION AND CARBONIZATION CONDITIONS ON HYDROCHAR FOR APPLICATION IN SOILS

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Technology development → Growth of population → Social problems

Waste management:
- Landfilling
- Incineration
- Compost

Circular Economy:
- Biomass waste
- Productive sector
- New products
- Transformation processes
- CO₂
- Material
- Energy

Biorrefinery

Bioeconomy

Feedstock

CHANIA 2023
HYDROThERMAL CARBONIZATION (HTC) OF BIOMASS WASTE

EVOLUTION OF PUBLICATIONS ON THE TOPIC

- Hydrothermal Carbonization TOTAL: 4,870
- Hydrothermal Carbonization and Wastes TOTAL: 1,578

Source: Scopus (08/06/2023)
HYDROTHERMAL CARBONIZATION (HTC) OF BIOMASS WASTE

HTC PROCESS

TEMPERATURE
170 – 250 ºC

RESIDENCE TIME
min – 24 h

HIGH-MOISTURE WASTE
(> 40 %)

Hydrochar

Biofuel

Catalyst support

Soil amendment

Activated carbon

Energy storage

Methane

Hydrogen

Irrigation water

Chemical recovery

Process water

Material

Energy
Project Motivation

- What feedstock properties are needed to achieve desirable products?

Carbonization product market and usability is critical for HTC commercialization and/or routine HTC use.
Goals and Objectives

• **Overall Goal**
  Identify the feedstock properties and carbonization conditions that are critical in determining appropriate hydrochar use

• **Objectives**
  1. To conduct carbonization experiments on food waste components over different carbonization conditions.
  2. To build statistical models using laboratory data.
  3. To identify the feedstock properties and carbonization conditions that most significantly impact hydrochar use.
HTC EXPERIMENTS

Food waste

160 mL HTC reactor

Slurry

Vacuum filtration

Hydrochar

- Elemental composition
- Energy content

Process water

- Typical water quality parameters: pH, conductivity, TOC, COD
- Primary nutrients: Total nitrogen, ammonia, phosphorus, potassium

Operating conditions
- Reaction times: 4, 8, 16, 24 h
- Reaction temperatures: 200°C, 225°C, 250°C
Individual Components Comprising Typical Food Waste

- Meat (MT)
- Vegetables (VG)
- Fruits (FT)
- Grain derived (GD) foods
- Daily commodities (DC)
## Properties of the Individual Components Comprising Typical Food Waste

<table>
<thead>
<tr>
<th>Food Waste</th>
<th>Moisture (%)</th>
<th>Component</th>
<th>%</th>
<th>Food Waste</th>
<th>Moisture (%)</th>
<th>Component</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Meat</strong></td>
<td>72.6</td>
<td>Poultry</td>
<td>50</td>
<td><strong>Fruit</strong></td>
<td>81.3</td>
<td>Banana</td>
<td>31</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Beef</td>
<td>25</td>
<td></td>
<td></td>
<td>Apple</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pork</td>
<td>25</td>
<td></td>
<td></td>
<td>Orange</td>
<td>15</td>
</tr>
<tr>
<td><strong>Vegetables</strong></td>
<td>90.4</td>
<td>Potato</td>
<td>19</td>
<td></td>
<td></td>
<td>Grape</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lettuce</td>
<td>17</td>
<td></td>
<td></td>
<td>Avocado</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Onion</td>
<td>14</td>
<td></td>
<td></td>
<td>Pineapple</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Tomato</td>
<td>14</td>
<td></td>
<td></td>
<td>Strawberry</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pumpkin</td>
<td>12</td>
<td>Daily commodities</td>
<td>27.7</td>
<td>Cheese</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cole</td>
<td>9</td>
<td></td>
<td></td>
<td>Chocolate</td>
<td>19</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Carrot</td>
<td>8</td>
<td></td>
<td></td>
<td>Butter</td>
<td>11</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Pepper</td>
<td>7</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Grain derived</strong></td>
<td>34.8</td>
<td>Bread/Flour</td>
<td>88</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Rice</td>
<td>9</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Oat</td>
<td>3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
More than 120 products obtained.

General trends associated with products generated for each food waste component are as expected:

* Drainable process water was not always obtained with the grain derived foods and daily commodities
### Comparison of Hydrochar Yields

<table>
<thead>
<tr>
<th>Commodities</th>
<th>Hydrochar Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain</td>
<td>24.6</td>
</tr>
<tr>
<td>Meat</td>
<td>40.1</td>
</tr>
<tr>
<td>Fruit</td>
<td>50.8</td>
</tr>
<tr>
<td>Vegetables</td>
<td>53.9</td>
</tr>
<tr>
<td></td>
<td>65.7</td>
</tr>
<tr>
<td></td>
<td>83.8</td>
</tr>
<tr>
<td></td>
<td>96.6</td>
</tr>
</tbody>
</table>

*Hydrochar yields decrease as initial solids content decreases*

- Yields influences hydrochar use
- Interesting hydrochar characteristics:
  - Daily commodities & Meat: more of a tar
Hydrochar yields greatly influence recoverable energy.
• **Carbon densification** increases with temperature and time:

  ![Graph showing carbon densification vs reaction time](image)

  **Example: Vegetable waste**

  Wastes with high carbohydrate content show the greatest carbon densification.

• **Carbon content** is relevant for energy-related applications

• **Carbon, Nitrogen, and Phosphorus** are relevant when using as a fertilizer
Nitrogen densification trends vary based on food waste component.

Example at 225°C:
- Meat slightly decreases
- Daily commodities remain constant
- Vegetables, fruit, and grain derived foods slightly increase
- Trends depend on waste chemical composition

- Carbon content is relevant for energy-related applications
- Carbon, Nitrogen, and Phosphorus are relevant when using as a fertilizer
• **Phosphorus densification** usually increases with temperature and time

Example at 225°C:
- Meat, vegetables, fruit, and grain derived foods slightly increase
- Daily commodities slightly decrease
- Trends depend on waste chemical composition

**Hydrochar Nutrients**

- Carbon content is relevant for energy-related applications
- Carbon, Nitrogen, and Phosphorus are relevant when using as a fertilizer
Potential Implications of Using as a Soil Amendment

Hydrochar shows adequate characteristics to be used as a fertilizer/soil amendment.

Different trends were found, with no clear understanding of what causes the differences.
Sequential Water Washing of Hydrochar

**Washing Process:**
- 5 washing cycles
- 24 h/cycle
- Ratio water:HC = 6:1 (equivalent to a rainfall/irrigation event for soils with 1% hydrochar)

**Analysis:**
- pH
- COD
- Toxicity
- $^1$H NMR
- N, P, K

**Toxicity testing:**
- Acute toxicity to E. coli
  - Toxi-Chromo Test (Environmental Bio-detection products, Inc.)
- Acute toxicity to V. fischeri
  - Microtox test (ISO 11348-3)

**Elemental analysis**
- Proximate analysis
- ICP
• Acute ecotoxicity to *E. coli*

- Trends depend on feedstock and carbonization conditions
- Overall, toxicity decreases with washing
• Acute toxicity to E. coli – Day 1 washing

**Specific trends differ for each feedstock**
Wash Water Toxicity

- Acute toxicity to *V. fischeri* – Day 1 washing
  - As EC50 increases, toxicity decreases
  - Trends depend on feedstock and carbonization conditions
  - Overall, toxicity decreases with washing
Wash Water Toxicity

- Acute toxicity to *V. fischeri* – Day 1 washing
Wash Water Composition

• Measured:
  • Nutrients (N, P, K)
  • pH
  • COD
  • TOC
  • $^{1}$H-NMR to get general composition
Wash Water COD

- Decreases with washing, TOC trends are similar
- Example:

200 °C

225 °C

250 °C
Wash Water Composition

- Correlation of these data with toxicity is not clear
Factors that Influence Wash Water Toxicity: Machine Learning Model

- Model predictions: Ensemble of decision trees and gradient boosting
  - XGBRegressor from XGBoost in Python
- Trained model with 75% data

Factors that Influence Wash Water Toxicity: Machine Learning Model

- Permutation feature importance
  - Successively eliminate variables with low feature importance
  - Change in model score indicates importance

Start with a model with all variables

Step #1:
- Remove least significant variable
  - Model with 3 variables
  - X2

Step #2:
- Keep removing least significant variable until reaching the stopping rule or running out of variables
  - Model with 2 variables
  - X4
  - X2

Step #3 - n steps:
Parameters of Importance:
1. Aromatic content
2. pH
3. Ammonia
4. TOC

Mean Square Error = 279.3

High MSE suggests we are not measuring some parameters that are important.
Can we predict aromatic content of the wash water?

Parameters of Importance:
1. Temperature
2. Feedstock H
3. Feedstock C
4. Time

Mean Square Error = 73.0

Decent prediction with parameters that make sense
Important Wash Water Properties

Parameters of Importance

1. Nitrogen
2. Molybdenum
3. Calcium
4. Cobalt
5. Chromium
6. Volatile matter
7. Phosphorus

Mean Square Error = 69.1
Parameters of Importance:

1. Feedstock H
2. Temperature
3. Time
4. Feedstock C
5. Feedstock VM

Mean Square Error = 57.7
What does this mean with respect to choosing a feedstock?

- **Predicted trends**
  - Fixed one variable, randomized others with a uniform distribution

![Graphs showing predicted toxicity changes with varying feedstock composition.](image)
Conclusions

Energy value can be approximated and feedstocks chosen appropriately

Possible acute toxicity in wash water from leaching of hydrochar can be predicted

Link between specific waste properties and hydrochar characteristics can be identified
Future Work

- Leaching model to predict washing requirements
- Refine machine learning model
- Determine parameters important for energy-related hydrochar use
- Investigate different methods for reducing potentially toxic substances on the hydrochar
- Perform an LCA to determine factors that influence the sustainability of hydrochar use
- Evaluate components of the liquid stream to investigate its toxicity