IC-6

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Recovery of Ferulic Acid from Wheat Bran by using Calcium Hydroxide

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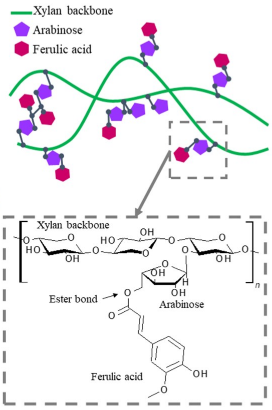
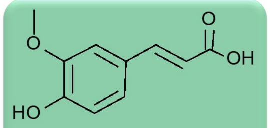
Introduction

1. Wheat bran and ferulic acid

Wheat Kernel → Wheat Milling Process → Byproducts →

Wheat Bran
150 Mtons/years





Ferulic acid

Price: $ 184/kg.
Market size: $ 35 Million.

- Pharmaceutical
- Cosmetics
- Precursors of flavor
- Aromatic chemical feedstock

2. The progress of ferulic acid recovery from wheat bran

Table 1. The progress of ferulic acid recovery from wheat bran

Method	Medium & Conditions	Yield of Ferulic acid [b] (mg/g)	Purity of Ferulic acid [c] (%)
Base-hydrolysis	1M NaOH; 30 °C; 24h	3.75	8.7
Steam explosion[1]	High pressure steam; 180 °C; 5.2 MPa; 0.95 h	0.97	-
Pressurized ethanol[2]	20% ethanol; 160 °C; 3 h	0.26	-
Enzymatic digestion (Aspergillus niger)[3]	EL[a] = 662 U/g; 45°C; 9 h	2.16	-

[a]Enzymatic loading; [b] sum of isomer; [c] based on GC area of product.

[1]*Food Chem.*, 2009, **115**, 1542; [2]*Waste and Biomass Valorization*, 2020, **11**, 4701; [3]*Biocatal. Agric. Biotechnol.*, 2018, **15**, 304;

Problem:
1. **Hard to handle and not environmentally friendly.**
2. **High energy consumption and high cost.**

Experimental

1. Hydrolysis of wheat bran

Wheat bran (225 mg)
$Ca(OH)_2$ (22.5 mg)*
DI water (5 ml)*

*Substituted by 5 mL of 1M NaOH and stir vigorously at 30 °C for 24 h

← Stir vigorously for 3 h at 90 °C*

Products

(Liquid) Water soluble products (±5 ml)
(Solid) Residue & Catalysts

Take for 1 ml

← HCl conc. (5-80 µl; until pH < 3)

Neutralized products

← Ethyl acetate (2 ml ×3)

Ethyl acetate phase 1 / Water phase

→ Solvent Evaporation
← Add 100 µl of dehydrated pyridine

Take for 50 µl of liquid

← Silylated by 50 µl of BTSFA in 60 °C for 30 min.
← Add 500 µl of internal standard 800 ppm
← GC-FID analysis

Selectivity & Yield of ferulic acid

2. Washing treatment of hydrolysis product.

Ethyl acetate phase 1

← DI water (5 ml × 2)

Ethyl acetate phase 2 / Water phase

To solvent evaporation

Results and Discussion

1. Catalytic hydrolysis of wheat bran to recover ferulic acid

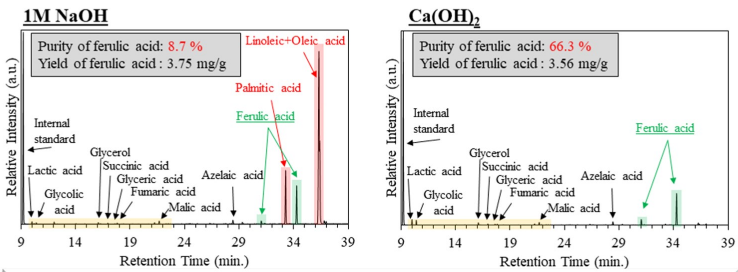


Figure 1. Chromatogram profile of the extracted oils that produced by 1M NaOH and $Ca(OH)_2$

- Recovery purity of ferulic acid over $Ca(OH)_2$-treated (66.3 %) was more selective than the usage of 1M NaOH (8.7%).
- NaOH was also hydrolyzed the fat that caused contamination of fatty acid derivatives into the product.

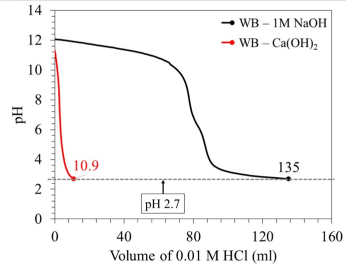


Figure 2. Neutralization curve of supernatant aliquot (1 ml) by 0.01 M HCl.

- Much lower solubility of $Ca(OH)_2$ shows the benefit to reduce the consumption of acid for neutralization in the post-treatment process.

Table 2. Liquid properties of the wheat bran hydrolysis product

Properties	Catalysts	
	1M NaOH	$Ca(OH)_2$
Color	Light Brown	Light Yellow
Viscosity (mm^2/s) [a]	302	4

[a] Determined by using viscometer Ubbelohde at 25 °C.

- Fatty acid salt content in the 1M NaOH-treated aliquot lead the increase of viscosity to 302 mm^2/s.
- Conversely, the low viscosity of $Ca(OH)_2$-treated aliquot (4 mm^2/s) was due to low fatty acid salt content in the product.

2. Washing treatment of hydrolysis product.

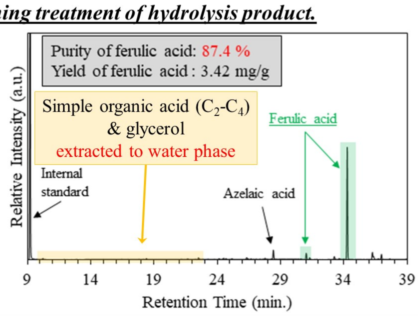


Figure 3. Chromatogram of the extracted oil that produced by $Ca(OH)_2$ after the washing treatment.

- The purity of ferulic acid was successfully increased from 66.3 % to 87.4 % by the washing treatment without significant loss of ferulic acid.

Conclusions

- $Ca(OH)_2$ selectively hydrolyzed the ester bond between ferulic acid and polysaccharides.
- $Ca(OH)_2$-treated process is more environmentally friendly due to the low consumption of acid during neutralization.
- The purity of ferulic acid in the extracted oil produced by $Ca(OH)_2$ increased without a significant loss of the yield of ferulic acid after the simple washing treatment.

Acknowledgement

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