P073

Pressurized Water Extraction and Supercritical Water Hydrolysis as means to obtain Ferulic Acid from Wheat Bran

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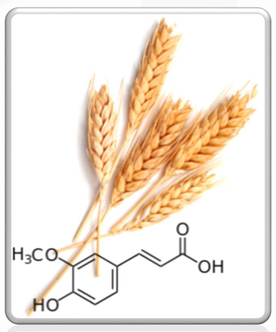
1 - .Introduction and Aims

Phenolic compounds are of considerable interest due mainly to their antioxidant activity. They constitute a large family of compounds that can be found in cereals, fruits or vegetables, and present potential commercial applications in the food, health and cosmetic industries. By extracting them from waste materials, we are contributing not only to reduce but also to revalorize the residues.

Wheat bran is an abundant and underused byproduct from the milling industry, whose main phenolic compound is ferulic acid (FA). It can be found in three forms: soluble free (F-FA), soluble-bound (C-FA) and insoluble bound form (B-FA), being the major part in the insoluble bound form, esterified to the arabinoxylans (AX) and other cell wall structural components (≈92%). Hence, new processes are required to break these bonds in order to recover it in high quantities.

In this study, two different techniques have been applied and compared with the aim of maximizing the extraction of FA from wheat bran: in the first one **PRESSURIZED WATER** (PW) is used as solvent and in the second one **SUPERCRITICAL WATER** (SCW) has been tested.

2 - .Experimental section

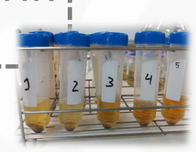


Wheat bran

Provided by a milling factory from Valladolid, Spain: Emilio Esteban SA

- **MEAN PARTICLE SIZE: 1140 µm**
- **MOISTURE: 12.1%**

Characterization of FA in the matrix



Free FA
Soluble -bound FA
Bound FA

→ Soluble-bound FA and bound FA are obtained after an alkaline hydrolysis followed by a liquid-liquid extraction using diethyl ether

Pressurized water extraction (PWE)

Stainless steel batch reactor V_{tot}= 160 mL
P_{max} = 15 bar

Supercritical water hydrolysis (SCWH)

Continuous supercritical water plant
- Operation conditions: 400ºC, 250 bar
- Time: 0,15-0,5 s

Ferulic Acid quantification by HPLC. In the optimum, FA was also quantified after an alkaline hydrolysis (soluble-bound FA)

3 - .Results and Discussion

1. **CHARACTERIZATION.** The total amount of FA in the wheat bran matrix is 3151,6±1,8 µg/g, being 3,8±0,2 and 31,6±0,1 µg/g the amount of free and soluble-bound FA, respectively.

2. **PRESSURIZED WATER EXTRACTION.** Temperature has been tested in the range 130-160 °C. The maximum amount of extracted FA was obtained at 160 °C during 75 min. Results are shown in table 1. The trends observed in these tests showed that higher temperatures could be applied.

Table 1. Extracted ferulic acid expressed as mg of FA per Kg of dry matter used in each procedure

Technology applied	Conditions	Ferulic acid in the liquid extract (mg/Kg)	
		Free	After alkaline hydrolysis
Pressurized water extraction (PWE)	160°C 75 min	381,7	1342,7
Supercritical water hydrolysis (SCWH) (400°C, 250 bar)	0,22 s	848,7	1220,4
	0,33 s	1400,4	1904,7
	0,46 s	1176,0	1704,4

3. **HYDROLYSIS LIMITING STEP**. After the alkaline hydrolysis of the extract around 40% more of FA was quantified, indicating that FA was released but it is still attached to soluble sugars. Analyzing the solid, other 40% was found still unreleased.

4. **SUPERCRITICAL WATER HYDROLYSIS.** In order to release the FA that is still bounded, either to the soluble or insoluble sugars, ultrafast supercritical water hydrolysis (SCWH) was tested. Results using SCWH at 250 bar and 400ºC with times in the range 0,22-0,46 s are presented in table 1, and revealed that around **45% of the initial FA could be obtained** by this technique, which is more than 3-fold higher the amount obtained with PWE. The other 50% was also released but still bounded to the soluble sugars. Comparison of the yields of both techniques are shown in figure 1. Further experiments to find the optimum conditions are going to be performed.

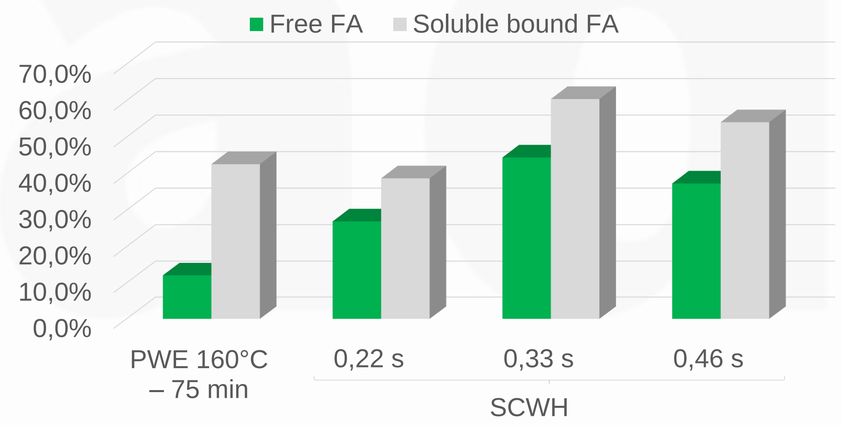


Figure 1. Comparison of the yields of the free and soluble-bound FA obtained in both techniques

4 - .Conclusions

- More than 92% of FA in wheat bran is in an insoluble bound form and its release depends on breaking the ester links between it and the polysaccharide chains.
- The optimum conditions found for the extraction of FA with pressurized water under the condition tested, are 160°C and 75 minutes, which led to a extraction yield of 12.1%.
- After alkaline hydrolysis a higher amount of FA is obtained, revealing that the hydrolysis is the limiting step. Harder conditions could be tested.
- Hydrolysis was enhanced by using ultrafast SCWH. The extracted FA was around 3.5-fold higher than the obtained with PWE and required an extremely low reaction time. Further studies are needed to optimize this technique.

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