

ADDING VALUE TO CROP BIOREFINERIES: EFFICIENT EXTRACTION OF FERULIC ACID FROM WHEAT BRAN





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- Introduction and Aims

The actual concept of circular economy aims to minimize the amount of residues, having lot of intends focused there attention on biorefineries. The profitability of these biorefineries could be increase if, as a previous step, the minor compounds but with high value added products are extracted.

In this work, wheat bran is used as raw material as it constitutes an abundant and underused byproduct from the milling industry. Ferulic acid (FA) is the main phenolic compound present in wheat bran (its structure is shown in figure 1). It exhibit potential commercial applications not only in health but also in food and cosmetic industries, and can be found in three forms: soluble free, soluble conjugated and insoluble bound form, the latest one mostly related to the ester links between it and the arabinoxylans (AX).

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In this work, the FA content in wheat bran was characterized, then a **PRESSURIZED LIQUID EXTRACTION (PLE)** was performed using different mixtures of ethanol-water, at different temperatures and extraction times in order to evaluate their effect in the amount of extracted FA. Other two techniques: SUPERCRITICAL WATER HYDROLYSIS (SCWH) and MICROWAVE ASSISTED EXTRACTION (MAE) were proposed as they are in accordance with the





- .Results and Discussion

- **CHARACTERIZATION.** The total amount of FA in the wheat bran matrix is 2570.9 µg/g, being 3.8 and 31.6 µg/g the amount of free and soluble conjugated FA, respectively.
- PRESSURIZED LIQUID EXTRACTION-EXPERIMENTAL DESIGN. Extracted FA ranged from 43 to 236 µg/g. The main effect plot for FA is shown in figure 2: it can be seen that higher 2. temperatures, longer extraction times and less ethanol content in the solvent are needed in order to obtain the greater amount of FA. Following the trends, further experiments were done using longer times and also water as solvent. The maximum amounts obtained are shown in table 1.



Table 1. N FA obt	/aximum d tained with	amount of h PSAE	100% 90% 80%	100% 90% 80%				Table 2. SCWH results, obtained with water at 250bar and 400°C				
	Water	20%EtOH	% 70% % % %	· · · · · • · · · · · · · · · · · · · ·				Reaction time (s)	Convertion (%)	log(R0)	FA (µg/g)	Yield (%)
T (°C)	160	160	₫ 40%		· · · •							
t (min)	74.3	101.9	<u>₹</u> 30%		· · · · ·	•••		0,22	0,90	6,32	822,9	32,0%
log(R0)	3.64	3.77	10%			•••••	••••••	0,33	0,87	6,34	1195,8	46,5%
Max [FA]	381.7	253.9	0%	20	40	60	80 100	0,46	0,84	6,29	1069,5	41,6%
Yield (%)	14.8%	9.9%		20	Time (m	nin)	200	0,48	0,93	6,34	1295,6	50,4%
			Figure 3	. Degrado	ation cur	rve of FA	<i>at 160</i> °C		,		,	,

- **DEGRADATION CURVE.** The degradation curve for a solution of 25 ppm of commercial FA was performed at 160°C (figure 3). It was obtained that at 75 min, only 15% of the initial FA is still present. The high degradation indicates that the hydrolysis is the limiting step, being 75 min the time where the degradation becomes higher than the hydrolysis.
- HYDROLYSIS LIMITING STEP. As the hydrolysis is the limiting step, but the exposition of the released FA in its free form conduce to high degradation, with the chosen techniques: 4. ultrafast supercritical water hydrolysis (SCWH) and microwave-assisted extraction, the temperature can be increased fast while the exposure of FA to these hard conditions is reduced. Results found with SCWH at 250 bar and 400°C are presented in table 2, and revealed that around 50% of the initial FA could be obtained by this technique, which is more than 3-fold higher the amount obtained with PLE.



- More than 98% of FA in wheat bran is in an insoluble bound form and its released depends on breaking the ester links between it and the polysaccharides chains.
- The PLE showed that water is a better solvent than mixtures Ethanol-Water, since the limiting step is hydrolysis. 160°C and 75 minutes are the optimum conditions for the extraction with \bullet water in stirred batch reactor, leading to a extraction yield of 14.8%.
- FA is extremely sensitive to high temperatures. The hydrolysis needs to be enhanced in order to reduce the time of exposition of the released FA to the high temperature.
- Ultrafast SCWH and MAE are promising techniques to this purpose. The extracted FA obtained with SCWH was around 50% of the initial one, which was 3.5-fold higher than the obtained with PLE and required an extremely low reaction time. Further studies are needed to decide the best extraction technique.

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