Industrial Crops and Products 52 (2014) 347–353

Contents lists available at ScienceDirect

Industrial Crops and Products

journal homepage: www.elsevier.com/locate/indcrop

Valorization of oilseed residues: Extraction of polyphenols from flaxseed hulls by pulsed electric fields

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ARTICLE INFO

Article history:
Received 15 July 2013
Received in revised form 23 October 2013
Accepted 29 October 2013

Keywords:
Polyphenols
Acidic hydrolysis
Alkaline hydrolysis
Ethanol
Extraction

ABSTRACT

This work aims at obtaining extracts with high level of polyphenols from flaxseed hulls treated by pulsed electric fields (PEF). The effect of the different operating parameters was studied on the extraction of polyphenols such as the PEF treatment duration, the PEF electric field strength, the solvent composition (ethanol, acid or base content) and the rehydration duration of the product. Results have shown that a PEF treatment allowed the extraction of up to 80% of polyphenols when applied at 20 kV/cm for 10 ms. For lower PEF electric field strength, the extraction efficiency was smaller. The rehydration of the product before PEF application improved the treatment efficiency. The highest polyphenols increase (≈37%) was observed when the product was rehydrated for 40 min before PEF application. The addition of ethanol, citric acid and sodium hydroxide has increased the extraction of polyphenols. The highest polyphenols yield was reached with a solvent containing 20% of ethanol and 0.3 mol/L hydroxide sodium. The alkaline hydrolysis was more effective than the acidic hydrolysis.

1. Introduction

The re-use of agricultural residues have been received much attention over the last few years due to the increasingly shortage of natural resources and the need for environmental protection. Many investigations have been aimed at converting the waste materials into food ingredients, bio-fuels, and other value-added applications (Makris et al., 2007; Pan et al., 2012; Piwowarska and Gonzalez-Alvarez, 2012). In particular, flax, *Linum usitatissimum*, is mainly produced for fiber and oil. Recently, flaxseed has attracted attention from the scientific community due to its favorable chemical composition (Muir and Westcott, 2003). Flaxseed appears to be a key raw material in the nutraceuticals and functional foods industry, as it is an important source of omega-3 fatty acids, soluble fiber (mucilage) and polyphenols (lignans) (Oomah, 2001). The polyphenols of flaxseed have been shown to reduce the levels of LDL-cholesterol in blood, the risk of diabetes, and hormone related cancer. They have antioxidant activity, cardioprotective effect, and improve renal function in lupus nephritis patients (Muir and Westcott, 2003; Oomah, 2001; Prasad, 2000).

The flaxseed polyphenols are mainly found in the seed coat of seed, where they are ester-linked to 3-hydroxy-3-methyl glutaryl (HMG) residues, and possibly bound to other compounds. These polyphenols can be released from such structures by hydrolytic cleavage of the ester bonds (Ford et al., 2001). The traditional polyphenols extraction methods involve a grinding pretreatment and a sequential or a simultaneous alcoholic solid–liquid extraction and alkaline treatment (Eliasson et al., 2003). These methods are time and energy consuming; they can take a few hours. Moreover, they involve extensive subsequent solid–liquid separation and purification steps.

Recently there has been an increasing demand for new extraction techniques that are environmentally friendly, faster, and more efficient than the traditional extraction methods. Among these techniques, microwaves (Bagherian et al., 2011), high pressure (Corrales et al., 2008), and pulsed electric field (PEF) (Grimi et al., 2011; Loginova et al., 2011; Boussetta et al., 2012; Shynkaryk et al., 2009) have shown their efficiencies for the extraction of biomolecules from different plants. In particular, PEF is a non-thermal technique providing electrical pulses of a few microseconds. The PEF action is mainly localized on a microscopic scale. Pores are formed in cell membranes thus accelerating the release of intracellular compounds. For example, the impact of PEF

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http://dx.doi.org/10.1016/j.indcrop.2013.10.048

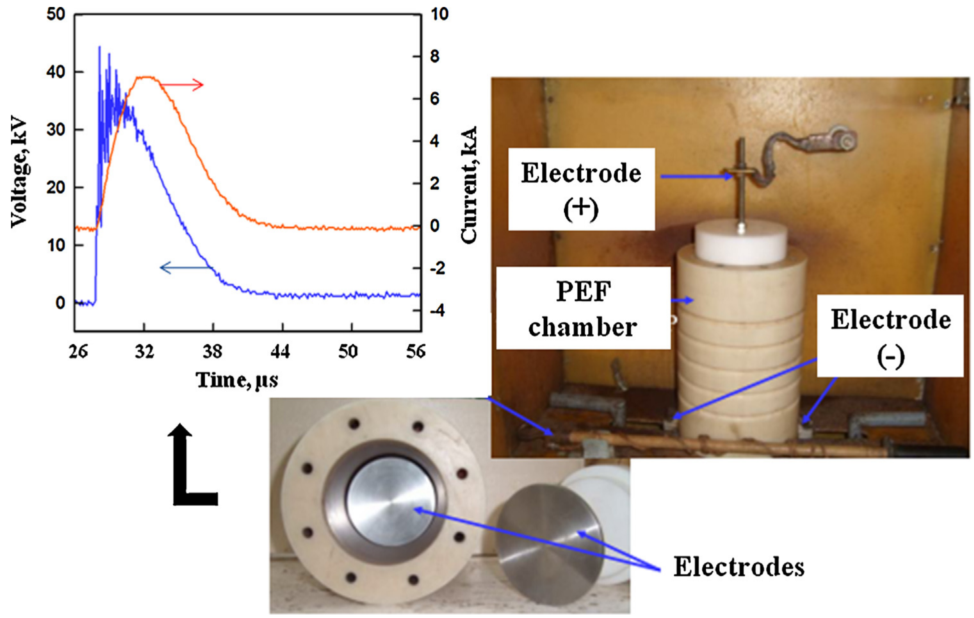

Fig. 1. Experimental set-up.

on the recovery of oils from oilseeds and the impact on high fatty plant cells were studied (Guderjan et al., 2007). In this work the application of pulsed electric fields on the recovery of oil and functional food ingredients as antioxidants, tocopherols, polyphenols and phytosterols as well as oil quality parameters from hulled and non-hulled rapeseed were investigated. In summary, PEF have been applied on various fresh fruits and vegetables. However, there are only a few studies concerning the treatment of oilseed but nothing concerning the seed hulls.

The main objective of this study is to optimize the PEF-assisted extraction of polyphenols from flaxseed hulls. The effect of pretreatment (electric field intensity, input treatment energy) and diffusion (solvent, pH) operating parameters will be investigated. This study aimed at optimizing the combination of factors in order to reach the highest extraction yield of polyphenols.

2. Materials and methods

2.1. Biological material

Flaxseed (*L. usitatissimum*, cultivar Baladin) hulls (seed teguments) were provided by Lasalle Beauvais (Beauvais, France). The dry matter content of dried seeds is 96.3%.

2.2. Polyphenols extraction

2.2.1. Rehydration of flaxseeds hulls

The flaxseeds hulls (10 g) were mixed with a mixture of water and ethanol (250 mL). Various concentrations of ethanol (from 0% to 50%, v/v) in water were supplemented with 0.05–0.3 mol/L sodium hydroxide for alkaline extraction or with 0.05–0.3 mol/L citric acid for acidic extraction. The suspension was introduced in a beaker under agitation at 150 rpm for up to 60 min. The temperature of the mixture was 20 °C. After rehydration, the suspension of flaxseeds hulls was treated by PEF.

2.2.2. Pulsed electric fields treatment

The PEF apparatus consisted of a pulsed high voltage power supply (Tomsk Polytechnic University, Tomsk, Russia) and a batch 1-L treatment chamber with stainless electrodes as previously described (Boussetta et al., 2012). The electrodes of the treatment chamber were two parallel disks. As two plane electrodes are used for the treatment, the electric field should be homogeneous. The electrode area was 95 cm^2. The distance between the electrodes can be varied from 1 to 10 cm. The circuit configuration and the electrodes shape generated exponential decay pulses. The PEF pulse length was about t_i = 10 µs. The test set-up and the pulse shape are described in Fig. 1.

Flaxseed hulls (10 g) suspended in the extraction solvent (250 mL) at 20 °C were introduced between the electrodes. The extraction solvent was the same as that used during the rehydration step. The high voltage pulse generator provided 40 kV to 10 kA pulses. These data are the maximum capability of the generator. The distance between electrodes was varied so that the corresponding electric field strengths E were 10, 15 and 20 kV/cm. Note that the change in distance between electrodes has no effect on the pulse shape. The PEF treatment consisted of applying up to n_{PEF} = 1000 pulses with a frequency f of 0.33 Hz. This pulse frequency was imposed by the generator. Thus the time of PEF application $t_{PEF} = n_{PEF} \cdot t_i$ was varied from 1 to 10 ms. In this study, the number of pulse was counted and the pulse length was defined as the time required for a given pulse to decay from its peak voltage to 37% of the peak voltage. The voltage (Ross VD45-8.3-A-K-A, Ross Engineering Corp., Campbell, USA) and current (Pearson 3972, Pearson Electronics Inc., Palo Alto, USA) sensors were connected with a 108 MHz sampling system via an oscilloscope (Tektronix TDS1002, Oregon, USA). The software HPVEE 4.01 (Hewlett-Packard, Palo Alto, USA) was used for acquisition of data. From the measured voltage $V(t)$ and current $I(t)$, the instantaneous power $P(t) = VI$ is calculated numerically, and the total dissipated energy per pulse W_p is obtained by integration of the power P for the whole duration T of the pulse (Eq. (1)).

$$W_p = \int_0^T V(t)I(t)dt \quad (1)$$

2.2.3. Grinding

Flaxseeds hulls were crushed for 40 s in a laboratory coffee grinder (SEB, Ecully, France) (180 W). The total energy input