OCL 2020, 27, 65
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https://doi.org/10.1051/ocl/2020060

OCL Oilseeds & fats Crops and Lipids

Available online at:
www.ocl-journal.org

RESEARCH ARTICLE — OPEN ACCESS

Residence time distribution and flow pattern modeling of oilseeds in a pilot screw press☆

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Received 12 May 2020 – Accepted 27 October 2020

Abstract – Mechanical expression is widely applied for oil recovery from oilseeds using continuous screw presses. Despite significant recent advances in the field of press design and automation, it remains difficult to predict the press performances based on the theoretical approaches, and more experimental investigations are needed to clarify and characterize the seeds flow and expression behavior in the press. Residence Time Distribution (RTD) is a frequently used tool in chemical engineering to characterize the material flow by simple tracer tests. In this paper, we explore the feasibility of using RTD for the screw presses, in order to check the flow patterns homogeneity and identify the possible deviations depending on the press geometry and the operating conditions. Both theoretical modeling and experimental investigation are conducted for two different screw press designs (Reinartz and Olexa), and at the different rotation speeds. An original and reliable experimental methodology was developed by using erucic acid as tracer in the form of pulse injection and gas chromatography as detection method. Experimental results coupled with statistical calculations showed the influence of the screw geometry and the rotation speed on the seeds flow inside the press. The matter displacement was much faster and the experimental residence time was very close to the theoretical one indicating more homogeneity and less dispersion in the Olexa arrangement in comparison to the Reinartz arrangement. The higher variance observed at lower rotation speed (2.4 rpm) suggested the presence of flow defects like mixing and axial dispersion in the press. To complete the experimental work, axial dispersion model was applied, and allowed obtaining the valuable information, such as axial dispersion degree and distribution functions. Obtained results can be very useful to predict the performance of existing screw presses and design more efficient industrial equipments.

Keywords: screw press / pressing / residence time distribution / oil seeds / crushing

Résumé – Distribution de temps de séjour et modélisation de l'écoulement des graines oléagineuses dans une presse à vis pilote. Le pressage est largement utilisé pour l'extraction des huiles végétales. Dans les usines de trituration des graines oléagineuses, des presses à vis industrielles sont souvent employées. Malgré les avances récentes dans le domaine de la conception et de l'automatisation des presses, il reste difficile de prédire les performances d'une presse sur la base des approches théoriques et par conséquence des recherches expérimentales supplémentaires sont nécessaires pour clarifier et caractériser l'écoulement dans une presse à vis. La Distribution de Temps de Séjour (DTS) est un outil fréquemment utilisé en génie chimique pour caractériser le flux de matière en écoulement à l'aide d'un traceur. Cette étude est consacrée à l'étude de la DTS dans une presse à vise afin de vérifier l'homogénéité de l'écoulement et identifier les écarts possibles en fonction de la géométrie de la presse et les conditions de fonctionnement. Pour ce faire, une analyse expérimentale couplée à une modélisation a été appliquée pour deux géométries de presse à vis (Reinartz et Olexa), et à différentes vitesses de rotation. Une méthodologie expérimentale originale et fiable a été développée en utilisant l'acide érucique, comme traceur biologique et la chromatographie en phase gazeuse pour la détection. Les résultats expérimentaux et l'analyse statistique ont montré l'influence de la

☆ Contribution to the Topical Issue "Technological challenges in oilseed crushing and refining / Défis technologiques de la trituration et du raffinage des oléagineux".

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géométrie de la vis et de la vitesse de rotation sur l'écoulement des graines à l'intérieur de la presse. Dans l'arrangement Olexa, l'écoulement des graines est plus rapide et le temps de séjour expérimental est très proche de celui théorique, indiquant un écoulement plus homogène et moins dispersé par rapport à l'arrangement de Reinartz. La variance est plus élevée à faible vitesse de rotation (2.4 trs/min) suggérant la présence des défauts d'écoulement comme le mélange et la dispersion axiale dans la presse. Pour compléter l'analyse expérimentale, la modélisation à l'aide du modèle de dispersion axiale a permis d'obtenir des informations précieuses, telles que le degré de dispersion axiale et les fonctions de distribution. Les résultats obtenus peuvent être très utiles pour prédire les performances des presses à vis existantes et concevoir des équipements industriels plus efficaces.

Mots clés : presse à vis / pressage / distribution de temps de séjour / grains oléagineux / trituration

1 Introduction

In the conventional crushing process, vegetable oil is expressed from oilseeds using screw press. Industrial presses, measuring up to 5 m, are basically made up of a rotating screw surrounded by perforated barrel. Solid-liquid expression is achieved, all along the screw, by a progressive compression of the press-cake combined with oil drainage through the barrel perforations. The liquid fraction (oil) is recovered under the press, in a collecting tray, whereas partially deoiled press-cake is released in the discharge section. The pressing efficiency in a screw press depends on many factors, like seeds properties, screw press geometry and operating conditions (rotation speed, compressive pressure and temperature). Pressing time has been identified as a key parameter to optimize oil extractability (Hickox, 1953; Vadke and Sosulski, 1988; Bamgboye and Adejumo, 2007; Akinoso *et al.*, 2010). Indeed, in order to be recovered, the oil stored inside the seed cells must be expressed from the intracellular to the extracellular medium (Lanoisellé, 1996; Bogaert *et al.*, 2018a). Experiments conducted on discontinuous presses, showed that slower compression velocity and larger pressing times improved oil percolation and leakage through the compressed matter and resulted in higher extraction yields (Hickox, 1953; Lanoisellé, 1996; Savoire, 2008; Mhemdi *et al.*, 2016). In the continuous screw press, the compression time is related to the residence time, which represents the time that the oilseeds take to pass through the press, from the feed hopper to the discharge section. It is consequently inversely proportional to the press capacity. In order to optimize screw press-operating performances, the pressing time must be precisely controlled and adjusted to fit a compromise between the oil expression yield and the press capacity (Savoire *et al.*, 2013).

However, it's difficult to ensure a good homogeneity of the solid matter flow in a screw press. Consequently, unpressed virgin seeds are frequently released in the discharge section (Pradhan *et al.*, 2011; Dunford, 2012). This processing defect is due to a heterogeneous distribution of the flow pattern and it represents a huge financial loss. This concern is common for different continuous processes, in which the residence time has a major impact on the final product quality. Residence Time Distribution (RTD) studies are then frequently applied to characterize the flow pattern, identify the flow defects and model the matter flow in order to predict the operating performances of the studied equipment.

RTD determination is based on the use of an inert tracer. The tracer is first injected to the process inlet and its concentration is measured at the output. Literature mentions a large variety of tracers (dyes, magnetic materials, acids, bases, salts, minerals, etc.) but their use may disturb the flow behavior and impact the study accuracy (Chalamet and Taha, 1999). Ideally, the tracer should be miscible and chemically similar to the original material, while possessing identical rheological properties (Poulesquen *et al.*, 2003). Both off-line and in-line detection techniques are available, including γ-ray detection, optical methods, spectrometric techniques', electrical conductivity measurements, etc. (Wolf and White, 1976; Oberlehner *et al.*, 1994; Sun *et al.*, 2003; Zhang *et al.*, 2006; Fang *et al.*, 2011). Once the tracer concentration profile at the outlet is known, RTD is usually represented by a distribution function E (t), describing the vessel response to the tracer introduction and leading to the mean residence time, variance et standard deviation calculations (Levenspiel, 1972).

Up to now, the RTD methodology was very rarely applied to screw presses. The case of extruders, which working principle is very similar to continuous pressing, is however well documented, especially because this process involves both heat sensitive materials and good mixing requirements (Wolf and White, 1976; Puaux *et al.*, 2000; Poulesquen *et al.*, 2003; Nikitine *et al.*, 2009; Gao *et al.*, 2012; Zhang *et al.*, 2014). Studies showed that the feed rate, the rotation speed, the die diameter and the screw temperature have a significant impact on the process performance (Van Zuilichem *et al.*, 1988; Yeh *et al.*, 1992; Pansawat *et al.*, 2008; Nwabueze and Iwe, 2010). Various models have been developed to characterize RTD in extruders. Some of the models are based on the matter velocity profile, the screw geometry and strong assumptions (isothermal, laminar, non-compressible Newtonian and non-slip flow) (Bruin *et al.*, 1978; Harper, 1981; Rauwendaal, 1989; Ganjyal and Hanna, 2002; Poulesquen and Vergnes, 2003). Others use an association of ideal reactors, such as plug flow, tubular, plate or continuous stirred tank reactor to construct a conceptual flow model (Wolf and Resnick, 1963; Gao *et al.*, 1999; Kumar *et al.*, 2008; Xu *et al.*, 2017). They describe every flow pattern combination that can occur along the screw: mixing zone, dead volume, by-pass, recirculation loops or segregated flow.

Unlike extrusion devices, screw presses are characterized by oil output flow, which complicates the RTD study. The tracer concentration evolution must be measured in both oil and press cake produced in order to check the material balance and obtain relevant statistical results. Therefore, an original methodology should be developed for screw press RDT study. Our previous study, conducted on a Reinartz AP08 pilot screw

Table 1. Oil and moisture content in oleic and erucic rapeseeds.

	Oleic rapeseed	Erucic Rapeseed
Oil content	46.38 ± 2.44%	33.52 ± 1.86%
Water content	7.55 ± 0.74%	8.04 ± 0.42%

press, revealed the impact of the screw geometry and the rotation speed on the matter compression (Bogaert *et al.*, 2018b). An alternation of high and low pressure sections was observed along the screw, causing press-cake compression, relaxation or even mixing phenomena. To complete this study, the present work was consecutively devoted to characterize the influence of the screw geometry and the rotation speed on the flow pattern and the residence time distribution of the particles. Experiments were conducted on oleic (canola) rapeseed and erucic rapeseed (gaspard) was used as a tracer. This choice appeared as the optimal solution to minimize the bias induced by the tracer injection. These two varieties, belonging to Brassicaceae family, have very similar phenotypic and textural properties but different fatty acid profiles. The tracer (erucic rapeseed) is rich in erucic acid (EA) while canola seeds do not contain this fatty acid. After the tracer injection, oil and press-cake were regularly sampled and their fatty acid composition was determined by gaseous chromatography. This study, combining experimental and statistical approaches, allowed calculating the mean residence time and the distribution functions for different rotation speeds (2.4–18.2 rpm) and two screw geometries (Reinartz and Olexa). The flow pattern was also modeled using the axial dispersion model and the flow behavior was compared to ideal reactors (plug flow and perfectly stirred reactors).

2 Material and methods

2.1 Plant materials

Rapeseeds were provided by the technical center for oilseeds "OLEAD" (Pessac, France). The press was operated with oleic rapeseed (Canola variety) and erucic rapeseed (Gaspard variety) was used as a tracer. Seeds oil and moisture content were measured by hexane extraction method (ISO 659:2009) and oven desiccation (ISO 665:2000), respectively. The results are presented in Table 1.

Seeds fatty acid profile was determined meaning gaseous chromatography according to the international standards ISO 12966-1:2014 and ISO 12966-2:2011. The details of the operating protocol are available in the Section 2.4 and the results are presented in Table 2. As we can see, erucic rapeseed oil is rich in erucic acid (33.47%) while oleic rapeseed oil doesn't contain this fatty acid, justifying thus the choice of this tracer.

2.2 Experimental setup

Experiments were performed in a Reinartz AP08 screw press (length 1800 mm, width 500 mm and height 800 mm). This equipment is a small capacity pilot screw press accurately reproducing the phenomena observed in industrial scale presses. The pressing section is composed of a 62 cm single rotation screw, surrounded by a perforated barrel. The screw rotation speed can be adjusted in the range 0–18.2 rpm on the control panel. The mass of oil and press cake produced are continuously weighted by two balances connected to a computer. An acquisition software calculates and records press cake Q_{cake} and oil Q_{oil} flow rates.

Table 2. Fatty acid composition for oleic and erucic rapeseeds.

Fatty acid		Oleic rapeseed	Erucic rapeseed
Palmitic acid	C16:0	2.50 ± 0.15%	2.24 ± 0.04%
Stearic acid	C18:0	0.67 ± 0.05%	0.60 ± 0.02%
Oleic acid	C18:1 (ω-9)	61.84 ± 4.23%	25.9 ± 0.56%
Linoeic acid	C18:2 (ω-6)	23.91 ± 3.18%	18.35 ± 0.17%
Alpha-linoleic acid	C18:3 (ω-3)	10.29 ± 0.96%	8.15 ± 0.14%
Gadoleic acid	C20:1	0.79 ± 0.05%	11.28 ± 0.18%
Erucic acid	**C22:1**	**0 ± 0%**	**33.47 ± 0.63%**

During the pressing operation, the motor transmits rotation to a shaft, equipped with a set of annular elements composing the screw arrangement. These elements can be dismounted and replaced. Two different geometries have been studied and compared in this study (Fig. 1). The first geometry, made up of the Reinartz AP08 original screw model (Fig. 1a) and named "Reinartz arrangement", has a constant diameter and reversed screw paths. The second arrangement, provided by Olexa company, called "Olexa arrangement" (Fig. 1b), has an increasing diameter and regular screw paths. For both screw arrangements, former extensive studies based on pressure, density and residual oil content profiles have shown discontinuities in the matter flow. An alternation of high pressure (compression) and low pressure (relaxation + mixing) sections was observed. Defatting occurred exclusively in the compression sections. In the low pressure sections, intensive press cake mixing and oil reflux was identified for the Reinartz arrangement whereas only transport and cake relaxation occurred with the Olexa arrangement (Fig. 1). More details are available in Bogaert *et al.* (2018b).

2.3 Experimental methodology

The residence time distribution was studied, for both Olexa and Reinartz arrangements, at different screw rotation speeds (2.4, 6.1, 12.1 and 18.2 rpm). For each experiment, the steady state was established using oleic rapeseed and the press operating performances were determined.

The press capacity Q_{in} (kg/h) was calculated using equation (1):

$$Q_{in} = Q_{oil} + Q_{cake} \quad (1)$$

The theoretical passage time $t_{passage}$ (min) in the press was calculated using equation (2):

$$t_{passage} = \frac{Mass\ of\ seeds\ in\ the\ press}{Q_{in}} \quad (2)$$

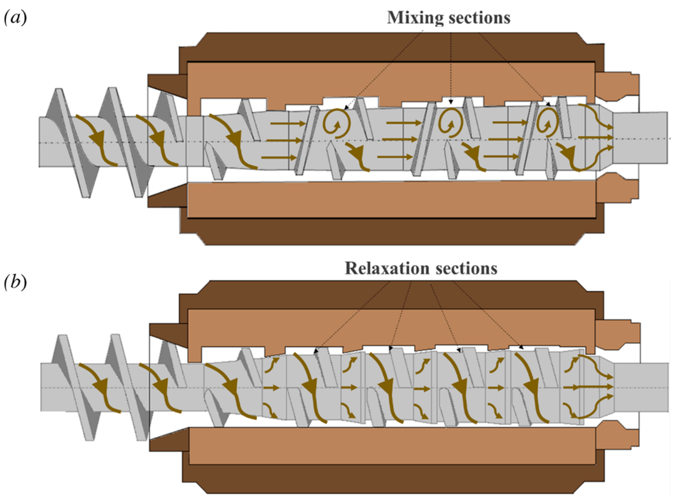

Fig. 1. Influence of the screw geometry on the matter displacement in the barrel for (a) Reinartz and (b) Olexa arrangements.

The mass of seeds in the press was experimentally determined by weighing.

The oil yield Y (%) was calculated using equation (3):

$$Y = \frac{Q_{oil}}{Q_{in}.C_{in}} \quad (3)$$

where C_{in} (g/g) is the oil content in the seeds.

The tracer (erucic rapeseed) was then introduced in the feed hopper in the form of a pulse injection. A preliminary study was conducted to determine the minimal quantity of tracer allowing a good and accurate detection of the tracer in oil and cake outflows. Hence, 300 g (m_{tracer}) of erucic rapeseed containing 33.7 g of erucic acid was injected for each experiment. The corresponding pulse duration (t_{pulse}, min) and the pulse time ratio (τ, %) were calculated using equations (4) and (5) respectively:

$$t_{pulse} = \frac{m_{tracer}}{Q_{in}} \quad (4)$$

$$\tau = \frac{t_{pulse}}{t_{passage}} \times 100 \quad (5)$$

For a satisfying statistical reliability, sampling time ($t_{sampling}$, min) and frequency ($\Delta t_{sampling}$, min) were established as:

$$t_{sampling} = 3t_{passage} \quad (6)$$

$$\Delta t_{sampling} = \frac{t_{passage}}{20} \quad (7)$$

Oil and cake samples were finally analyzed to determine their erucic acid content. Cake samples were used for RTD calculation, because they characterize the time taken by solid particles to be transported from the feed to the discharge section. Oil samples were used to check the material balance, measuring the quantity of erucic acid recovered in the liquid phase.

2.4 Fatty acid composition analysis

Samples fatty acid composition was determined by gaseous chromatography (GC-MS). The residual oil contained in the press cake samples was recovered meaning hexane. After solvent evaporation, the experimental procedure was the same as for oil samples. The fatty acids were first converted into fatty acid methyl ester (FAME) by transesterification. For this purpose, 5 mg of oil was solubilized with 150 µL of toluene and 0.5 mL of methanolic HCL was added. The solution was then vortexed for 5 s and incubated during 2 h at 80 °C, with intermediate vortex operations every 30 min, to maximize the reaction yield. The reaction was subsequently stopped with 250 µL of sodium bisulfite (5% aqueous solution) and 2 mL of hexane was added to extract the FAME. The solution was vortexed for 1 min and centrifuged during 2 min at 10 000 rpm to separate aqueous and organic phases. A vial was finally filled with 1 mL of the organic phase. The vial composition was analyzed with a mass spectrometer (Thermo fisher Scientific, Allemagne) equipped with a BPX-70 column (length 30 m, diameter 0.25 mm and film thickness 0.25 µm). The oven temperature was first fixed at 140 °C and then increased by 19 °C/min until 230 °C. It was maintained at this temperature for 5 min. The carrier gas (helium) was injected at a flow rate of 1 mL/min and the sample injection volume was 1 µL. The FAME were identified from NIST standard reference library. Gaseous chromatography analysis provided the samples fatty acid profiles and allowed the calculation of the erucic acid concentration in the oil $C_{EA,oil}$ and the cake $C_{EA,cake}$.

2.5 RTD calculations

For each experiment, the total mass of erucic acid recovered in the oil $m_{EA,oil}$ and the cake $m_{EA,cake}$ were calculated from equations (8) and (9):

$$m_{EA,oil} = \sum_{i=t_0}^{\infty} \left[C_{EA,oil}\right]_i.\Delta t_{sg}.\ Q_{oil} \quad (8)$$

$$m_{EA,cake} = \sum_{i=t_0}^{\infty} \left[C_{EA,cake}\right]_i.\Delta t_{sampling}.\ Q_{cake} \quad (9)$$

The total mass of erucic acid leaving the press was then obtained by summing $m_{EA,oil}$ and $m_{EA,cake}$ and compared to the corresponding mass of erucic acid introduced during the pulse. An error under 5% on the material balance was considered acceptable.

After material balance checking, the residence time distribution function E(t), the cumulative distribution function F(t), the mean residence time $\bar{t}$ and the associated standard deviation σ were determined using equations (10) to (13) respectively (Levenspiel, 1972). E(t) represents the fraction of the outflow rate which residence time ranges between t and t + Δt (Eq. (10)). F(t) represents the accumulated quantity of tracer at the exit at a given time (Eq. (11)).

$$E(t),\ min^{-1} = \frac{[C_{EA,}]_i}{\int_{t_0}^{\infty} [C_{EA,}]_i .\, dt} = \frac{[C_{EA,}]_i}{\sum_{i=t_0}^{\infty} [C_{EA,}]_i .\, \Delta t} \quad (10)$$

$$F(t), - = \int_{t_0}^{\infty} E(t).dt \quad (11)$$

$$-\bar{t}, min = \int_{t_0}^{\infty} t.E(t).dt \quad (12)$$

$$\sigma^2, min^2 = \int_{t_0}^{\infty} (t - -\bar{t})^2 .E(t).dt \quad (13)$$

2.6 Flow modeling

To provide accurate information concerning the matter displacement along the screw, the axial dispersion model was applied to fit the experimental data. This model was developed to describe non ideal flow patterns and compare a real flow to a plug flow with possible axial dispersion (Fig. 2). It uses normalized and dimensionless distribution functions E(θ) and F(θ) calculated with equations (14) and (15).

$$E(\theta) = \bar{t}.E(t) \quad (14)$$

$$F(\theta) = F(t) \quad (15)$$

$\theta = \frac{t}{-\bar{t}}$ is the normalized and dimensionless time.

This model is based on equation (16), which characterizes the dispersion between two adjacent portions of a plug flow reactor. It is analogue to the Fick's law applied for diffusion phenomenon.

$$\frac{\partial C_{EA}}{\partial \theta} + \frac{\partial C_{EA}}{\partial X} = \frac{D_a}{uL} \frac{\partial^2 C_{EA}}{\partial X^2} \quad (16)$$

Where D_a (m^2/s) is the axial dispersion coefficient, u (m/s) is the displacement velocity, L (m) is the total length of the reactor, x is the axial position and X = x/L is the normalized axial position. When the tracer is introduced in the form of a pulse injection, the solution of this equation provides an expression for the normalized distribution function E(θ) (Eq. (17)):

$$E(\theta) = \frac{1}{\sqrt{4\pi \frac{D_a}{uL}}} \exp \frac{1-\theta^2}{4\pi \frac{D_a}{uL}} \quad (17)$$

The maximum $E(\theta)_{max}$ of this function can be calculated from equations (18):

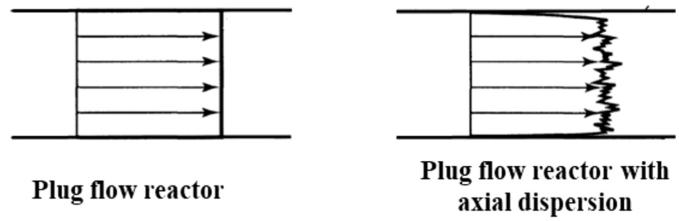

Fig. 2. Illustration of the axial dispersion occurring in a plug flow reactor.

$$E(\theta)_{\max} = \frac{1}{\sqrt{4\pi \frac{D_a}{uL}}} = \frac{1}{\sqrt{4\pi \frac{1}{Pe}}} \quad (18)$$

The Peclet number ($Pe = uL/D_a$, dimensionless) represents the ratio of the mass transportation (convective flow) to the mixing phenomena (dispersive flow) occurring in the reactor. The dispersion index 1/Pe (dimensionless) is used to estimate axial dispersion degree in the studied equipment (Levenspiel, 1972; Stehr, 1984).

If 1/Pe → 0, then the axial dispersion is negligible, and flow is close to the plug flow

If 1/Pe → ∞, then the dispersion is important, and flow is close to the mixed flow.

In practice, the distribution function E(t) was first calculated from the experimental data and then normalized to obtain the E(θ). Its maximum $E(\theta)_{max}$ was determined to calculate the dispersion index 1/Pe.

3 Results and discussion

3.1 Experimental methodology validation

Oleic an erucic rapeseed used in this study had very similar phenotypic properties, composition and equivalent humidity content (Tab. 1). For example, moisture content was around 7.55 ± 0.74% for the oleic rapeseeds and 8.04 ± 0.42% for the erucic rapeseeds, meaning that they underwent similar preparation processes. However, their oil content and fatty acids profiles were significantly different (Tab. 2). The oil content was 46.38 ± 2.44% for the oleic rapeseed *versus* 33.52 ± 1.86% for the erucic rapeseed. The two species can be easily differentiated by their erucic acid content (with values of 33.47% for the erucic rapeseed and 0% for the oleic rapeseed). The singularity of its fatty acid profile made erucic rapeseed a perfect biological tracer for this study. During the pressing operation, a large part of the seeds' fatty acids (up to 94%) flows out from the barrel perforations in the oil fraction. The press cake produced contains only a small part of the initial oil and tracer (erucic acid) content. For this reason, the mass of tracer was fixed so that its concentration in the press cake remained above the limit of detection of gaseous chromatography analysis method

Tables 3 and 4 give the experimental parameters for each experiment conducted with the Reinartz and Olexa arrangements at different rotation speeds (2.4, 6.1, 12.1 and 18.2 rpm). The press capacity, the passage time and the extraction yields were calculated respectively from equations (1) to (3).

Table 3. Experimental parameters for Reinartz arrangement RTD study.

Screw rotation speed	rpm	2.4	6.1	12.1	18.2
Press capacity (Q_{in})	kg/h	4.1	10.0	19.8	29.4
Oil yield (Y)	%	89.5	86.1	85.4	84.0
Passage time ($t_{passage}$)	min	27.7	13.0	5.7	3.7
Mass of pulse tracer	kg	0.300	0.300	0.300	0.300
Mass of erucic acid in the pulse	kg	0.034	0.034	0.034	0.034
Pulse duration (t_{pulse})	min	3.85	1.81	0.79	0.51
Pulse time ratio (τ)	%	13.9	13.9	13.9	13.9
Sampling frequency ($\Delta t_{sampling}$)	min	1.4	0.6	0.3	0.2
Sampling time ($t_{sampling}$, min)	min	83.1	39.0	17.1	11.1

Table 4. Experimental parameters for Olexa arrangement RTD study.

Screw rotation speed	rpm	2.4	6.1	12.1	18.2
Press capacity (Q_{in})	kg/h	6.6	14.3	31.4	46.8
Oil yield (Y)	%	92.5	88.4	83.7	81.0
Passage time ($t_{passage}$)	min	12.1	5.6	2.5	1.7
Mass of pulse tracer	kg	0.300	0.300	0.300	0.300
Mass of erucic acid in the pulse	kg	0.034	0.034	0.034	0.034
Pulse duration (t_{pulse})	min	2.72	1.26	0.56	0.38
Pulse time ratio (τ)	%	22.5	22.5	22.5	22.5
Sampling frequency ($\Delta t_{sampling}$)	min	0.60	0.28	0.12	0.08
Sampling time ($t_{sampling}$, min)	min	36.3	16.8	7.5	5.1

The mass of erucic rapeseed (300 g) injected during the pulse contained 33.7 g of erucic acid. The pulse duration was calculated with equation (4). In residence time distribution studies, the pulse duration determines the reliability of the results: a long pulse reduces the accuracy of the statistical study, whereas a short pulse may cause difficulties for tracer detection in the outflow. In the present work, rather large pulse durations (from 0.38 to 3.58 min) were used to guaranty a satisfying detectability of the tracer in the press cake. The sampling frequency was increased from 0.2 to 1.4 min for Reinartz arrangement and from 0.08 to 0.6 min for Olexa arrangement, with the variation of the screw rotation speed from 18.2 to 2.4 rpm. The sampling time was fixed to cover 3 times the passage time to be sure that the injected tracer was entirely recovered in oil and cake fractions.

To check the accuracy of the experimental methodology developed in this work, the erucic acid EA concentration measurements were used to check the material balance of the tracer injected in the press. Figure 3 shows the temporal evolution of the erucic acid flow rate in the oil and the press cake, for Olexa arrangement at 2.4 rpm. This result is typical response of tracer's pulse injection. Similar results were also obtained for other rotation speeds and for the two screw arrangements investigated in this study. The curves show that the erucic acid was mostly recovered in the liquid phase (oil) with a maximum flow rate of 5 g/min. In the solid phase (the press cake), the EA was only detected after 9 min with a maximum flow rate of 0.7 g/min. These results confirm the efficiency of the solid-liquid separation in the screw press, with a good recovery of the seeds' oil giving low residual oil content in the produced press cake.

Experimental data presented in Figure 3 were correlated with equations (8) and (9) to estimate the total mass of erucic acid recovered in oil and press cake. As expected, this quantity is much higher in the oil than in the press cake (Fig. 3). For example, calculation showed that 31.44 ± 1.40 g of EA were recovered in the oil *versus* 2.99 ± 0.01 g in the press cake (under screw velocity 2.4 rmp with Olexa arrangement). In other words, 91.4% of the EA injected in the press were expressed from the seeds and recovered in the oil fraction. This value is very close to the oil extraction yield (Y = 92.2%) obtained at the same conditions justifying even more the choice of the tracer used in this study. Moreover, the total mass of EA recovered (34.41 ± 1.41 g) matches well the mass injected with an error of 2.2% confirming the reliability of the experimental methodology (injection, sampling and detection). This consistency was checked at different ration speeds for both Olexa and Reinartz screw press arrangements (Fig. 4).

3.2 Residence time distribution

The evolution of the tracer concentration in the press cake allowed calculation of the residence time distribution function E(t) using equation (10). Curves in Figure 5 show the influence of the rotation speed on the values of E(t) for both screw arrangements. It can be observed that curves are bell shaped characteristic of normal function, with a better symmetry at high rotation speeds (12.1 and 18.2 rpm). At low rotation speeds (2.4 and 6.1 rpm) a slight asymmetry can be noted, particularly for the Reinartz arrangement, with a tail on the curve's terminal part. This tail suggests the existence of recirculation loops or dead volumes inside the barrel induced

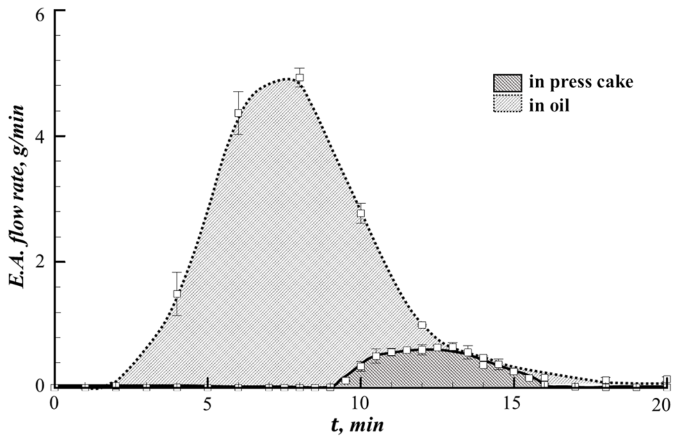

Fig. 3. Temporal evolution of erucic acid (EA) flow rate in oil and press cake at 2.4 rpm for the Olexa arrangement.

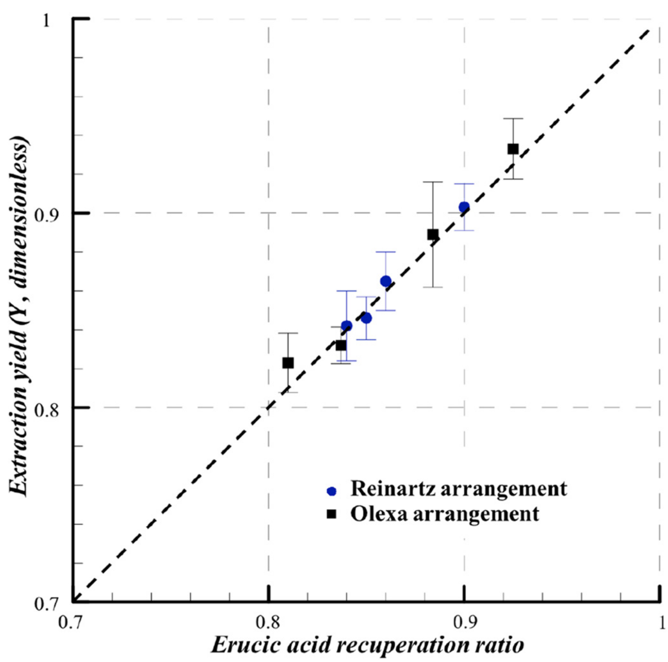

Fig. 4. Oil extraction yield *versus* tracer recuperation ratio in the oil fraction at different rotation speeds for Reinartz and Olexa screw presses.

by axial dispersion. In fact, results show that for both Olexa and Reinartz arrangements, the screw rotation speed has a major influence on the seeds flow. Indeed, at high rotation speeds, E(t) curves form high and narrow peaks, whereas at low rotation speeds the curves are much flatter. Moreover, it can be observed that the maximal value $E(t)_{max}$ is higher for the Olexa arrangement (Fig. 5b) comparatively to the Reinartz arrangement (Fig. 5a) independently on the screw rotation speed. For example, at the screw rotation speed 2.4 rpm, $E(t)_{max}$ was about 0.24 min^{-1} for Reinartz and only 0.11 min^{-1} for Olexa arrangement. This difference may be attributed to the difference in the press geometry. Consequently, the passage time, the flow speed, and the E(t) values were different. For

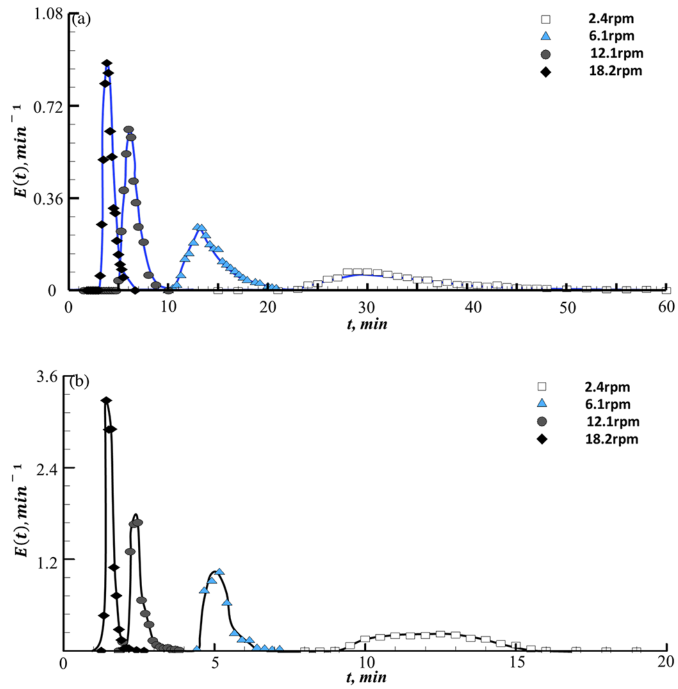

Fig. 5. Residence time distribution function for (a) Reinartz and (b) Olexa screw arrangements at different rotation speeds (2.4, 6.1, 12.1 et 18.2 rpm).

instance, it can be observed that the theoretical passage time at the screw rotation speed 2.4 rpm is about 2.2 times higher for the Reinartz (27.7 min) as compared to the Olexa arrangement (12.1 min). The ratio $\frac{t_{passage,reinartz}}{t_{passage,olexa}}$ is very close to the ratio $\frac{E(t)_{max,reinartz}}{E(t)_{max,olexa}}$ confirming the accuracy and precision of the experimental methodology developed in this study.

The experimental data and equation (12) were then used to calculate the mean residence time in the press. Results show that the experimental mean residence time decreased exponentially with the rotation speed, for both screw arrangements (Fig. 6). It plummeted from 32.2 min to 4.1 min for the Reinartz arrangement and from 12.2 min to 1.5 min for the Olexa arrangement, when the screw rotation speed increased from 2.4 rpm to 18.2 rpm. Similarly, the standard error σ, characterizing the dispersion decreased respectively from 6.1 min to 0.5 min and from 1.7 min to 0.2 min. High dispersion at low rotation speed confirms the accentuation of flow defects and mixing phenomena. On the other hand, experimental results highlight the influence of the screw geometry on the seeds flow inside the press. The matter displacement was faster in Olexa arrangement and the standard errors were significantly lower indicating better homogeneity and less dispersion in comparison with Reinartz arrangement. Consequently, the experimental mean residence time corresponded exactly to the theoretical passage time calculated from the press operating performances (Fig. 6). It can be speculated that the conical elements and the subsequent progressive volume reduction present in the Olexa arrangement homogeneously slow down the matter displacement velocity. On the contrary, in the Reinartz arrangement, it was observed that the experimental mean residence time is larger than the theoretical

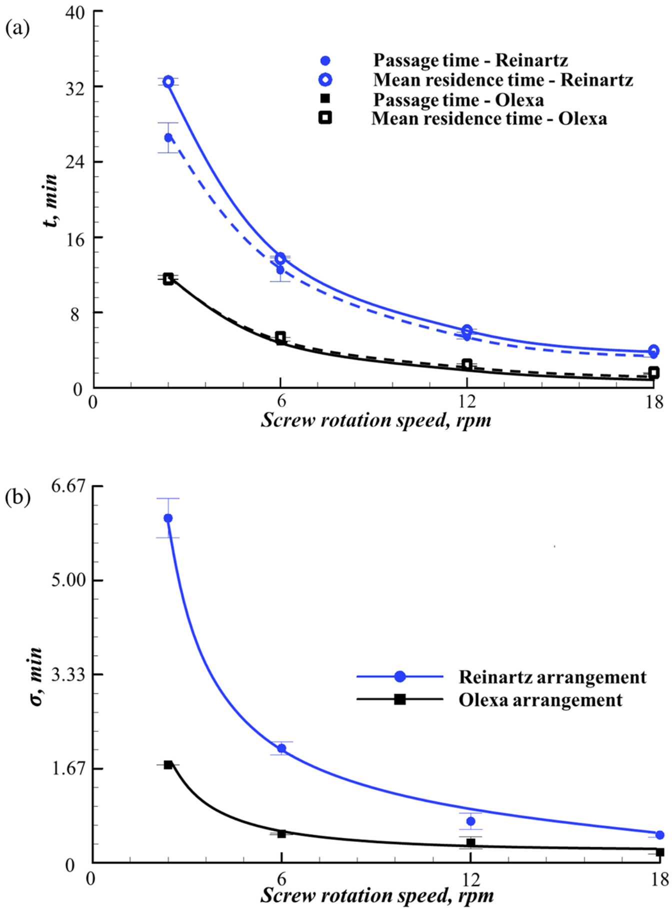

Fig. 6. Evolution of (a) the mean residence time and (b) the standard error *versus* the screw rotation speed.

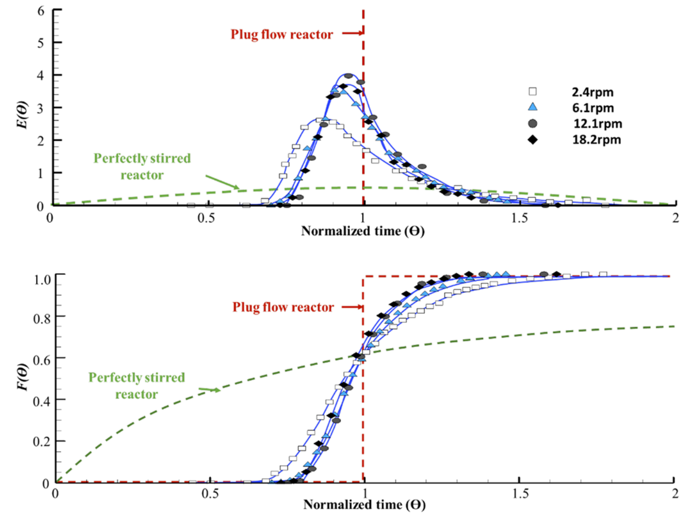

Fig. 7. Experimental and normalized distribution functions for the Reinartz arrangement.

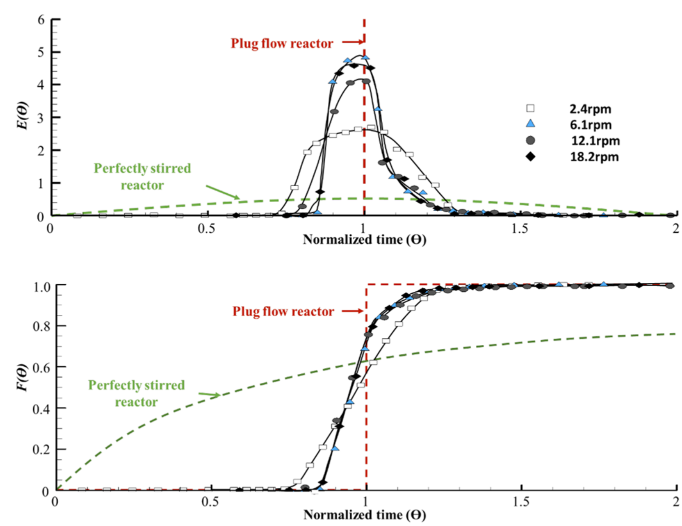

Fig. 8. Experimental and normalized distribution functions for the Olexa arrangement.

passage time, especially at low rotation speeds. For example, at the rotation speed 2.4 rpm, the experimental residence time for the Reinartz arrangement was about 20% higher than the theoretical one (33.8 min *vs.* 27.7 min). This difference may be attributed to the presence of mixing sections previously identified for the Reinartz arrangement (Bogaert *et al.*, 2018b). In fact, this screw press was instrumented by installing sixteen pressure sensors and three temperature probes throughout the screw. This instrumentation allowed the identification of different functional sections (feed, compression and mixing/relaxation) related to the screw geometry. In the compression sections, high pressure leads to oil expression and the formation of highly compressed cake. In the mixing sections, pressure falls to zero and the press-cake becomes friable. In these sections, the reversed screw paths induce perturbations on the flow pattern and part of the matter is then trapped in recirculation loops explaining thus the dispersion observed for this arrangement (Bogaert *et al.*, 2018b).

3.3 Flow modeling

Experimental normalized functions E(θ) and F(θ) were plotted and the obtained curves were compared to those of ideal reactors (plug flow reactor and perfectly stirred reactor). It is important to remind that the normalized distribution function E(θ) is a Dirac distribution (line surmounted by an arrow) for an ideal plug flow reactor and a flat parabola for a perfectly stirred reactor (dashed curves in Figs. 7 and 8). On the other hand, the schematic representation of the cumulative distribution function F(θ) is a "stepped" curve for an ideal plug flow reactor and a logarithmic shape curve for a perfectly stirred reactor. The typical curves for ideal reactors are represented in Figures 7 and 8 and compared to those experimentally obtained for the screw presses (Olexa and Reinartz) used in this study at different rotation speeds.

For the Olexa arrangement, the E(θ) experimental curves are bell shaped and well symmetric characterizing a plug flow. For the Reinartz arrangement, experimental curves are slightly flatter and asymmetric, mainly at low rotation speed (2.4 rmp). These results confirm the existence of flow defects previously detected and mentioned above for the Reinartz screw

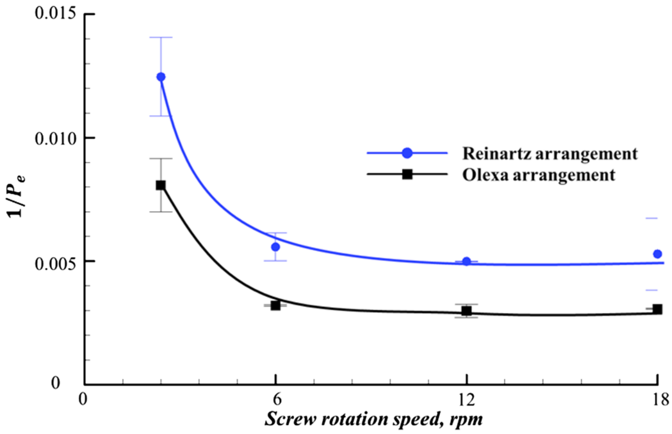

Fig. 9. 1/Pe versus the screw rotation speed for Reinartz and Olexa screw arrangements.

arrangement. On the other hand, F(θ) curves are sigmoidal and their shape looks closer to a plug flow reactor than to a perfectly stirred reactor. The profile is similar for all rotation speeds, except for 2.4 rpm which differs a little from a "stepped" curve. Experimental curves and the axial dispersion model (Eq. (16)) were used to quantify the dispersion degree in the press. This model allows quantifying the intensity of transfers occurring between two adjacent portions of a plug flow reactor and to characterize the flow behavior. For this purpose, the value of $E(\theta)_{max}$ was graphically determined from the corresponding E(θ) curve. Equation (18) was then used to determine the matter axial dispersion occurring in the press. The dispersion degree is proportional to $1/Pe = D_a/uL$. In other terms, when 1/Pe is close to 0 the dispersion is very low and the flow pattern is close to a plug flow reactor. Higher values of 1/Pe imply higher dispersion and a flow pattern becomes closer to a perfectly stirred reactor. The experimental values of 1/Pe are presented in Figure 9. The results confirm the existence of axial dispersion in the press, particularly at 2.4 rmp. In fact, it can be observed that the values of 1/Pe decrease rapidly with the increase of the rotation speed from 2.4 to 6.1 rpm before reaching a plateau for both Olexa and Reinartz arrangements. Generally, the values of 1/Pe were under 0.01 meaning that the axial dispersion was low, except for the Reinartz arrangement at 2.4 rpm, where the dispersion index was about 0.013 indicating higher matter dispersion. Moreover, results show that the values 1/Pe calculated for the Reinartz arrangement are about 1.6 times higher than those calculated for the Olexa, whatever the rotation speed. This confirms our previous observations.

4 Conclusion

RDT method was successfully applied for the oil seeds flow characterization in a continuous screw press. The experimental methodology, based on the detection of a biological tracer (erucic acid) by gaseous chromatography, was first validated with a material balance checking. The mass of erucic acid introduced in the press during the pulse injection fitted the mass recovered in oil and press cake with an error under 5% confirming the reliability and accuracy of the experimental methodology. The experimentally mean residence time calculated in this study was very close to the theoretical passage time, especially for the Olexa arrangement. For the Reinartz arrangement, a slight deviation highlighted the impact of the low pressure sections inducing mixing phenomena. The variance of the residence time increased when the screw rotation speed decreased, revealing an axial dispersion. Flow modeling revealed that the flow pattern was close to a plug flow reactor, with a slight axial dispersion, especially for the Reinartz arrangement at low rotation speeds. The data obtained in this work can be very useful for the prediction of screw presses performance and to design more efficient presses for the oil seeds expression.

Acknowledgements. Authors thank Terres Inovia and Olead (Pessac, France) for financial support and for providing canola seeds and Olexa (Arras, France) for supplying the screw arrangement.

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Cite this article as: Bogaert L, Mhemdi H, Vorobiev E. 2020. Residence time distribution and flow pattern modeling of oilseeds in a pilot screw press. *OCL* 27: 65.