Modelling of linseed oil expression curves
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Abstract

For oilseed selection, the impact of variety on the process is not taking into account, only agricultural factors are used (biomass yield and plant resistance to diseases). The objective of this work was to find a model that allowed the determination of the quantity of oil extractible from seeds. Linseeds (*Linum usitatissimum* L.) were used in this study. The expression was conducted on small quantities of seeds using an expression cell of 20.6 cm^3 that permits to process up to 10 g of seeds. This cell was attached to a food texture analyzer and the expression was performed under an uniaxial stress. The expression was achieved at constant speed (0.1 mm/s) until reaching of the constant pressure of 120 MPa (uniaxial compression creep test). Then the pressure was maintained at 120 MPa for one hour. The expression temperature was set to 50°C via a heating ring fixed on the outer surface of the cell. The constant speed phase of expression was analyzed to determine the occurrence of the oil point (the point were oil appears at the surface of the cake) using the calculation of the specific mechanical energy (SME). The oil point occurs for volumetric bed strain between 0.45 and 0.61 for the variety 6 harvested at seven different dates before maturity. The constant pressure time versus displacement curves were modeled with a four exponential viscoelastic model analog to a generalized Kelvin Voigt model with four elements associated in serial. The model gives an access to the oilseed mechanical intrinsic characteristics relative to different deformations of microscopic and macroscopic cake volumes (called intracellular, extracellular and extraparticular volumes). The four compressibility modulii obtained permit to calculate a global compressibility modulus which represents the required stress for oil expression. The parameters obtained from the model are analyzed by multiple regression analysis to establish a correlation between them and the mass of oil extracted during expression. The obtained correlation explained 93.6% of the variability in the extracted mass of oil.

Keywords: Linseed, viscoelastic Kelvin Voigt model, oil point, oil expression

1. Introduction

Flax (Linum usitatissimum L.) is a plant cultivated since antiquity. Two different types of flax are used, one for oil production from seeds and the other for fiber production from the vegetative part of the plant. The linseed oil has mainly industrial applications in coating industry due to its drying properties. In a wide range of country linseed is also an edible with interesting nutritional properties (Rapport and Lockwood, 2001). The seed contains mainly oil (between 35 and 45%) and proteins (10.5 to 31%) (Oomah and Mazza, 1993). Linseed oil contains in the majority three types of triglycerides: more than 50% of linolenic acid and around 20% of linoleic acid and 20% of oleic acid (Bockisch, 1998).

The oil process for linseed is composed of a facultative pre-treatment by flaking and or cooking followed by expression. The residual oil contained in meal is sometimes extracted by solvent. The expression process has been widely studied. The impact of pre-treatment on the expression efficiency has been investigated by numbers of authors (Smith and Kraybill, 1933; Hickox, 1953; Dedio and Dorrell, 1977; Khan and Hanna, 1984; Tchiegang et al., 2003) as well as the impact of oilseed composition, mainly water content (Sivala et al., 1991; Hammonds et al., 1991, Dedio and Dorrell, 1977). In all these studies, expression was evaluated in term of process optimization. Koo, 1942 was the first to propose an empirical equation that describes the oil yield as a function of the seed oil content, the expression pressure, the expression time and the oil viscosity during hydraulic pressing. In the following studies, the cellular material expression was modeled using soil mechanic principles (Terzaghi, 1948, Schwartzberg, 1997). The curves are usually modeled with a two or more polyexponential equation (Schwartzberg, 1997). For this modelling, only the constant pressure is modeled, the constant speed period is often neglected. Nevertheless, some authors have developed equations that allowed the characterization of the point where the oil begins to appear at the surface of the oilseed cake (Sukuraman and Singh, 1989). They called this point the "oil point". Our objective here is to integer the oil expression characteristics in the selection process of new optimal varieties of linseeds. The different parameters obtained by modelling of the expression curves give supplementary information on the ability to expression of the seeds.

2. Materials and methods

2.1. Seeds

Seven varieties of linseed harvested in 2006 were used. The varieties were arbitrary numbered from 1 to 7. These varieties were chosen according to their different mucilage content, the mucilage content was in 2005 less than 2.5 % for varieties 4, 5 and 6, between 2.9 and 4 % for varieties 1, 2 and 3 and for variety 7 it was in order of 5.9 % (the percentage was calculated on the basis of the fresh weight of seeds). Seven harvesting dates were selected between 35 days after linseed flowering and the complete maturity stage (between 83 and 87 days after flowering) in order to study the impact of the maturity stage on the expression ability. The different dates were compared in terms of growing degree days (GDD) instead of days after flowering in order to take into account the climatic conditions. GDD was calculated using Equation 1 with a base temperature of 5°C (McMaster *et al.*, 1997).

$$GDD = \sum_{\text{Flowering date}}^{\text{Harvesting date}} \left[\frac{T_{\max} - T_{\min}}{2}\right] - T_{base} \text{ for } \left[\frac{T_{\max} - T_{\min}}{2}\right] - T_{base} > 0 \qquad [1]$$

Seed composition was determined according to AFNOR standard methods for water and oil content (AFNOR 1973 and 1966 respectively).Water content is a major factor affecting oil expression (Lanoisellé and Bouvier, 1994) then all the sample were conditioned at 4% (d.b.) water content by oven drying at 43°C before processing.

2.2. Micropress

A micropress specially designed for oil extraction was used to express linseed (Gros, 2005). This press is formed of a 20.6 cm^3 pressing cell fixed on a food texture analyzer TA.HDi (Stable Microsystems, U.K.). The oil expression was realized at constant pressure (10 MPa) for 1 hour. The applied force and piston displacement were recorded in function of expression time with accuracies of 10 N and 0.001 mm.

The experimental setup is represented in Figure 1. The seed were crushed using a Comitrol 3600SL cutting mill (Urschel, USA). A mass of 3.17 g of seeds were put between two metallic filters (to ensure draining) and three filter papers (to absorb expressed oil). A heating ring was fixed on the lateral surface of the cell to allowed expression at 50°C. Half an hour before expression, the piston descended to its initial position and the system was heated at 50°C. At the beginning of the experiment, the piston descended at constant speed (0.1 mm/s) until fixed force was reached. After one hour expression at this force, piston automatically lifted to its original position and cell was dismantled. Expressed oil was weighted and oil and water content of the meal were determined according to AFNOR standard methods (AFNOR, 1967 and 1976 respectively).

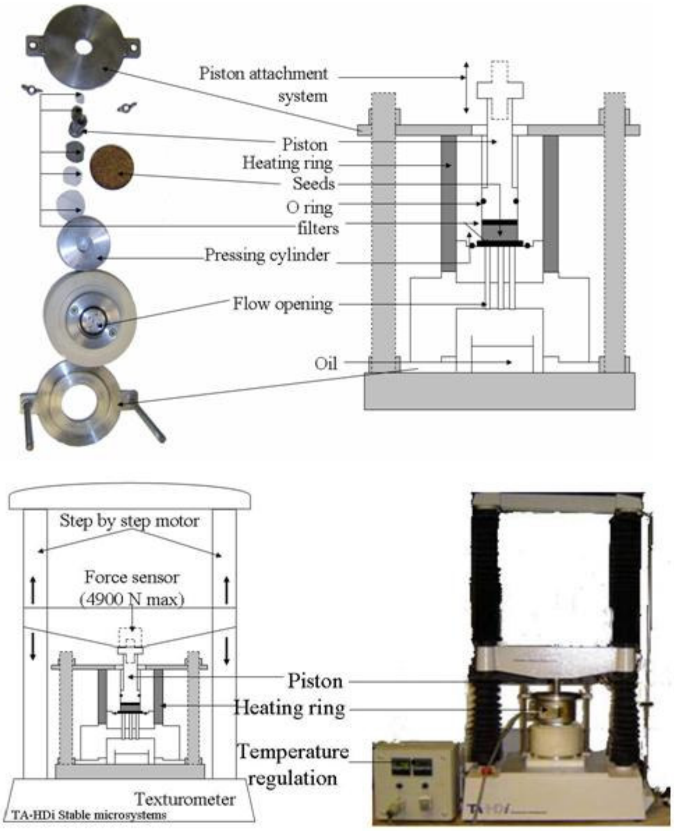


Figure 1: Experimental setup

3. Oil point determination

The oil point is defined in literature as the moment when the oilseed has been sufficiently squeezed to force oil out onto the surface of the seeds (Sukuraman and Singh, 1989; Faborode and Favier, 1996). The oil point has been determined visually by Sukuraman and Singh in 1989 as the time when oil appears at the surface of the rapeseed cake. Faborode and Favier in 1996 have determined the oil point by measure of the oil (pore) pressure. The oil point corresponds to an increase of the pore pressure and a decrease of the friction ratio. The authors have demonstrated that the oil point occurs when the bed density approach the kernel density. The calculation of the specific mechanical energy (SME) at each point of the compression leads to an exponential increase after the oil point when representing the SME in function of the volumetric bed strain.

In our experiments, the oil point was determined using SME.

The SME was calculated as the ratio of the power to the mass flow (Bimbenet *et al.*, 2002). In our case SME was calculated according to equation [2]:

$$SME = \frac{Force \text{ x } displacement}{initial\ seed\ mass} \quad [2]$$

The volumetric bed strain (ε) was calculated with equation [3] where h_0 is the initial height of the seed bed and h the actual height of the seed bed.

$$\varepsilon = \frac{h_0 - h}{h_0} \quad [3]$$

As the SME increases exponentially after the oil point, the representation of the logarithm of SME versus the volumetric bed strain allowed accessing to the equation of the exponential curve. From this exponential curve a theoretical value of SME for each volumetric bed strain was obtained. The oil point was specified at the volumetric bed strain for which the theoretical SME (calculated by exponential equation) and the experimental SME differed. The uncertainty on the SME and the volumetric bed strain permits to define precisely the oil point.

The Figure 2 represents the curves used for oil point determination. The Fig. 2a represents the logarithm of the SME in function of the volumetric bed strain. The theoretical curve corresponding to an exponential evolution of the SME after the oil point differs widely from the experimental curve for low volumetric bed strain. The second part of the figure shows the evolution of the theoretical and experimental SME in function of the volumetric bed deformation. A zoom allowed to identify the point where the theoretical and experimental curves differed and then to determine the oil point (Fig. 2c).

While the oil point occurs just before the cake density reaches the kernel density (seed cake devoid of air), the determination seems to be efficient.

degree day	P (MPa)	Volumetric bed strain	SME (J/kg)
420	3.39	0.49	3921
522	4.33	0.54	5688
653	4.38	0.61	6356
704	4.35	0.46	4804
755	2.96	0.46	3216
794	1.11	0.45	1192
1120	4.38	0.60	6271

Table 1: oil point pressure, volumetric bed strain and SME for variety 6 at different degree day.

The oil point can be determined quicker by simply determining the intercept of the two curve tangents at the beginning and at the end of the constant speed phase (Fig. 2b).

The oil point was determined for seven different harvesting dates for variety 6. Table 1 gives the oil point conditions using SME exponential increase method.

The two methods give similar results, for variety 6 at 522 degree day, the oil point takes place for a volumetric bed strain of 0.54 with the tangents method and 0.56 for exponential method. Then the two values are separate only by 3 seconds.

The oil point occurs for volumetric bed strain between 0.45 and 0.61. These values are higher than those find by Sukuraman and Singh (1989). These authors gave equations that permit to recalculate the values of bed strain and pressure using humidity and compression rate. By application of this equation to our experimental conditions, a volumetric bed deformation at the oil point of 0.388 and a pressure at the oil point of 5.96 MPa were found for a seed humidity of 5% (w.b.) and a compression rate of 6 mm/min. The oil point pressure is then lower in the present study than in Sukuraman and Singh (1989) work. Faborode and Favier (1996) have found similar values of oil point volumetric bed strain (0.497 and 0.467) and pressure (3.75 MPa and 4.69 MPa) for expression of cashew and groundnut at 12 mm/min compression rate and humidity of 4.3% and 6.0% (w.b. respectively). According to these results, they classified these seeds as soft seed with high oil content.

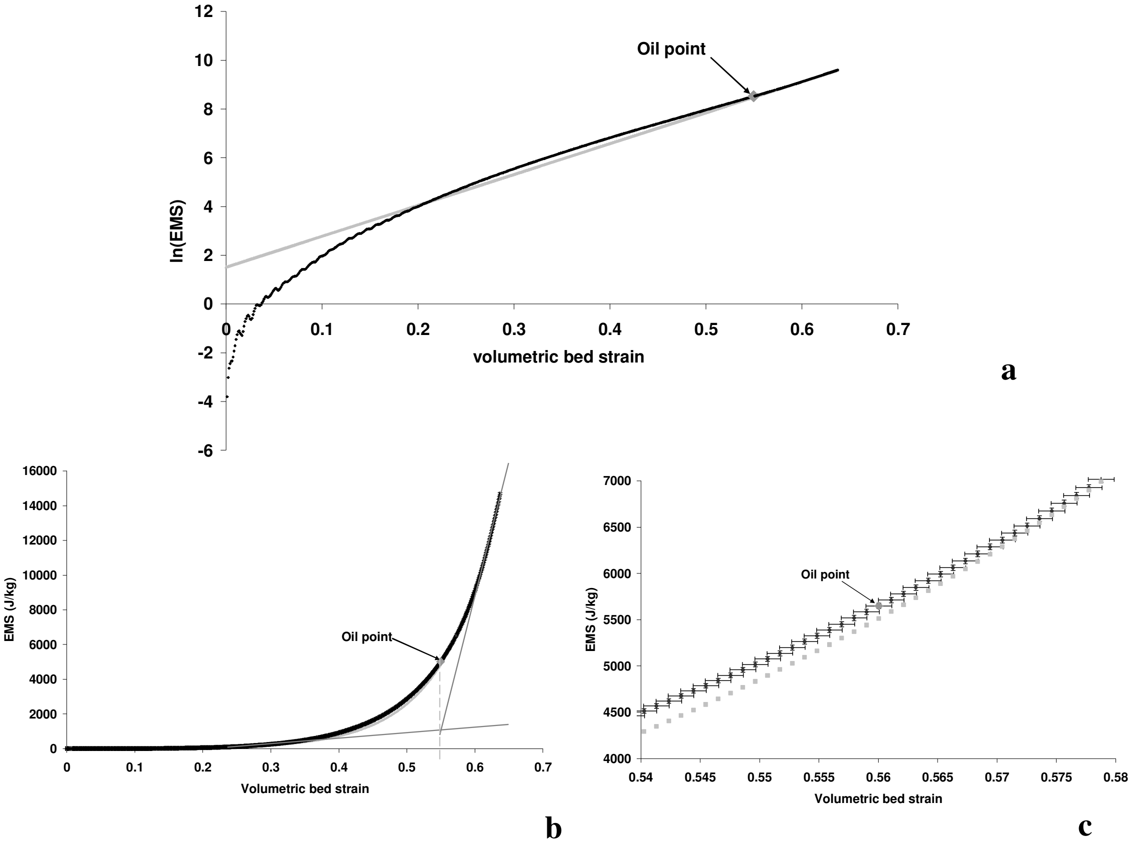


Figure 2: Determination of the exponential function (a); Determination of the oil point with tangent method (b) and with the SME method [zoom of graph b] (c)
(● experimental curve; ● theoretical curve).

4. Modelling

4.1. Model description

The expression curves obtained from one hour constant pressure expression operation were modeled with a four Kelvin-Voigt elements viscoeleastic model previously developed for oilseed extraction (Lanoisellé et al., 1996).

This model assimilates the oilseed cake to three imbricate volumes: intracellular, extra-cellular and extra-particular volume. In natural state, oil is mainly present in the intracellular volume.

The constant pressure hydraulic expression can be divided into three periods. During the initial period, the air present in extra-particular space is expulsed (Vorobiev et al., 1997). This period finished at the oil point. A mix of oil and air is expulsed from the cake during the second period, when oil replaces the intra-particular air. This period finished at the maximum instantaneous oil flow, the cake is then considered as saturated by oil. Then the third expression period commonly called consolidation begins. The modelling includes only the consolidation phase.

A two exponential model is usually used to represent the expression of fluid from biological materials (Schwartzberg, 1997). Here, the experimental data were modeled with the four exponential model present in Equation [4]. This model was divided by Lanoisellé et al. (1996) into four parts corresponding to four consolidation steps occurring simultaneously with different kinetics. These steps are: primary and creep consolidations of the extra-particular volume, extra and intra-cellular volume consolidation. Another view of this model is that the consolidation is the sum of four deformation kinetics. Each kinetic could be considerate as the result of the combination of plastic deformations of different size particles, frictions between particles, fluid expulsion from particles, fluid transfer in the particles (i.e. between adjacent cells).

The models for expression are often compared with rheological behaviors. So the four exponential model is similar to the generalized Kelvin model with four serial associated elements.

To characterize this model, three types of parameters should be determine: G_i the compressibility modulii characterizing the compression of each volume; υ_i the inverse of time delay ($1/tr_i$) characterizing the duration of each consolidation step and h_∞ the maximum oil theoretically extractible from cake for an infinite expression time.

$$h = \frac{h_\infty}{\sum\limits_{i=1_0,1,..,n} \frac{1}{G_i}} \left[\sum_{i=1_0,1,..,n} \frac{1}{G_i} (1 - e^{-v_i t}) \right] \qquad [4]$$

4.2. Model parameters identification

In order to apply the model, the data were modified, t and h were considered equal to zero at the beginning of the constant pressure period. h_∞ was determine according to Bouzrara and Vorobiev (2003), using an empirical equation derivate from equation [4]. The adapted formula where $q_0 = \lambda m_\infty$ presented in Equation [5] was used.

$$h = \frac{t}{1/q_0 + t/h_\infty} \qquad [5]$$

h_∞ is the slope inverse of the 1/h versus t curve. For low deformation rates at constant pressure, the curve is not quite linear (Figure 3). This could be due to expulsion of entrapped air (Schwartzberg, 1997).

Figure 3 represents the t/h versus t curve used to determine and the linear regression associate. The value of h_∞ is lower than the cake thickness that would appear if all the oil was expressed, so the method is adapted to the determination of h_∞.

Other model parameters (i.e. G_i and υ_i) were determined by numerical methods. For the parameters identification, Equation [4] was re-written in the following form (Equation [6]):

$$U = \frac{h}{h_\infty} = \frac{1}{1 + \sum\limits_{i=1,2,3} \sigma_i} \left[1 - e^{-v_{1_0} t} \right] + \sum_{i=1,2,3} \frac{\sigma_i}{1 + \sum\limits_{i=1,2,3} \sigma_i} \left[1 - e^{-v_i t} \right] \qquad [6]$$

Where σ_i is the ratio G_i/G_{10}.

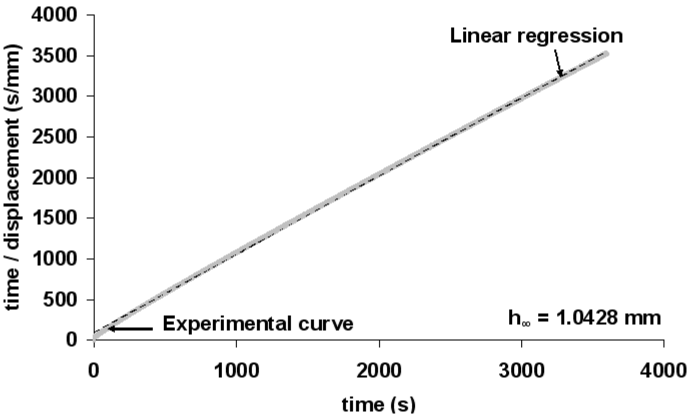
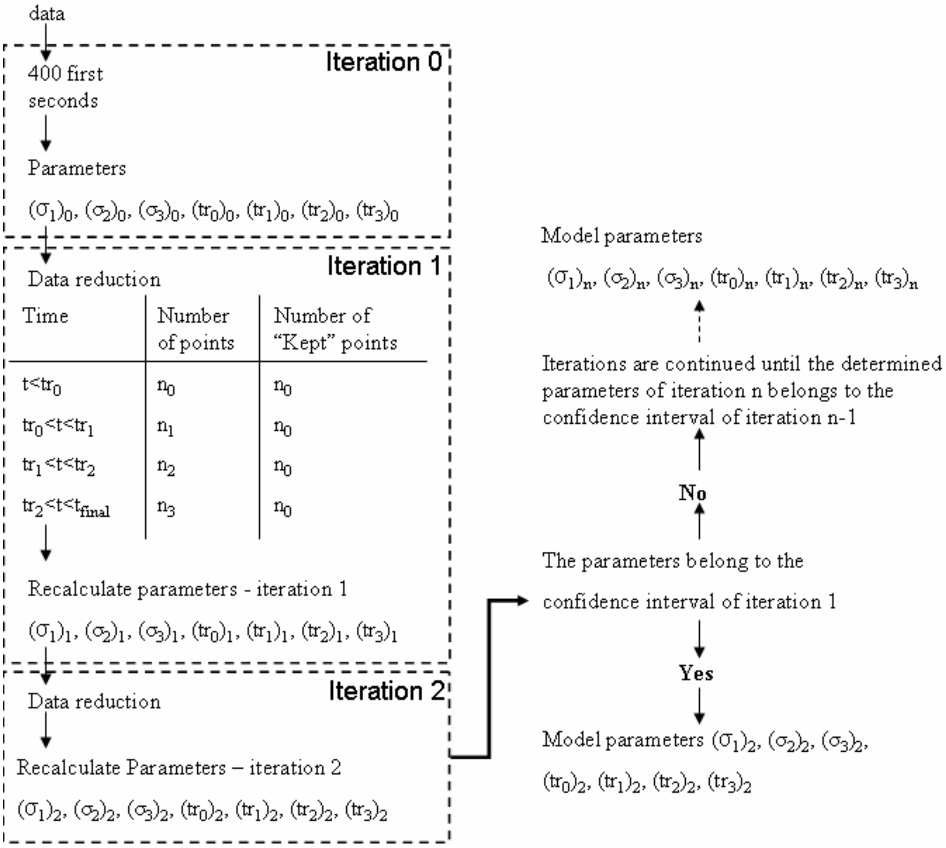


Figure 3: Linear regression for the determination of h_∞ (variety 6 at 704 degree day)



Time	Number of points	Number of "Kept" points
$t<tr_0$	n_0	n_0
$tr_0<t<tr_1$	n_1	n_0
$tr_1<t<tr_2$	n_2	n_0
$tr_2<t<t_{final}$	n_3	n_0

Iterations are continued until the determined parameters of iteration n belongs to the confidence interval of iteration n-1

The parameters belong to the confidence interval of iteration 1

Figure 4: Algorithm for the parameter determination.

σ_i were determined with TableCurve 2D software (AISN Software, Jandel Scientific, USA) according to the algorithm presented on Figure 4. To reduce the data, the ratio n_i/n_0 (the number of point of the considered phase and the number of points of the first phase) was calculated and one point over this ratio was preserved. The determination was conducted via iterations and was stopped when the determined values (σi and υ_i) belong to the 95% confidence interval of the previous iteration. Generally, 2 or 3 iterations are sufficient to complete this condition.

The compressibility modulii were calculated according to Equation [7]. Firstly, G_{10} was calculated with the use of σ_i, h_∞, and the height of cake at the beginning of constant force expression h_i. Secondly, the other compressibility modulii G_i were calculated as the ratio of G_{10} and σ_i.

$$G_{1_0} = (1+\sigma_1+\sigma_2+\sigma_3)\frac{h_c}{2h_\infty} \qquad G_1=\frac{G_{1_0}}{\sigma_1} \quad G_2=\frac{G_{1_0}}{\sigma_2} \quad G_3=\frac{G_{1_0}}{\sigma_3} \qquad [7]$$

According to the viscoelastic rheological model, the compressibility modulii were calculated for a unitary stress of 1 Pa in order to compare materials independently of the stress. A global compressibility modulus was also determined by Equation [8].

$$G=\frac{1}{{}^{1}\!/_{G_{1_0}}+{}^{1}\!/_{G_1}+{}^{1}\!/_{G_2}+{}^{1}\!/_{G_3}} \qquad [8]$$

4.3. Modelling results

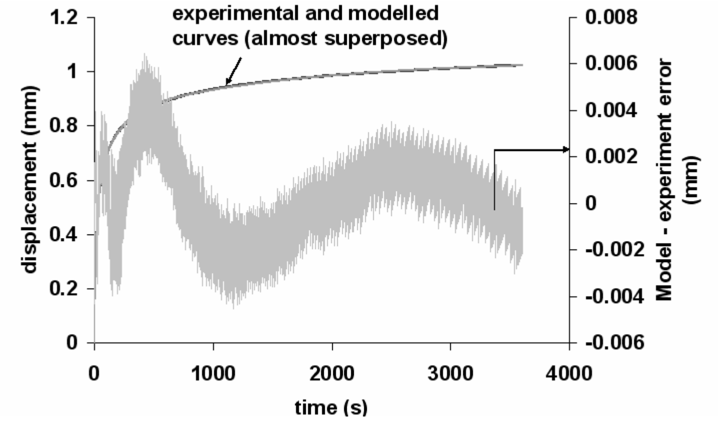


Figure 5: Experimental, modeled curve and difference between the both curves for variety 6 harvested at 704 degree day.

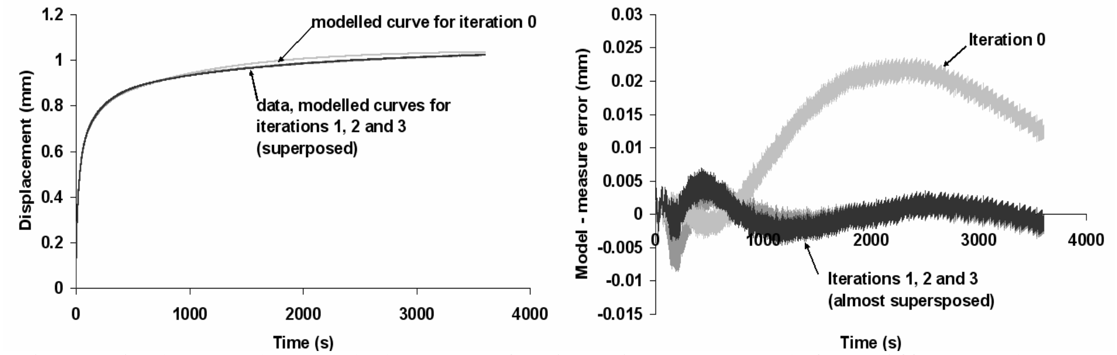


Figure 6: data and modeled curves for iteration 0 to 3 (left). Difference between experimental data and modeled curve for iteration 1 to 3 (right). (data from variety 6 at 704 degree day).

The model parameters were determined for all linseed samples. The figure 5 shows the good agreement between the experimental data and the modeled curve. The maximum difference between the two curves is inferior to 1% of the maximum displacement. The interest of the iteration system is illustrated by figure 6. The first iteration leads to a difference between experimental and modeled curve more important, the next iterations are almost superposed. The interest of the second and next iteration is to have a fixed criterion to decide when the iterations should be stopped.

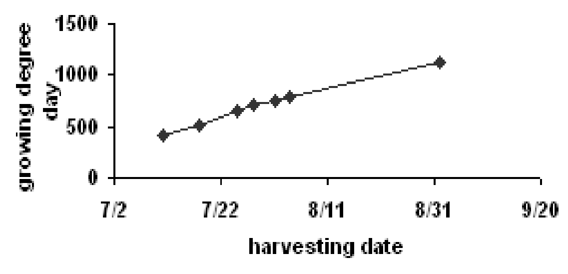
variety	degree day	harvesting humidity[a]	oil content[a]	oil mass expressed (g)	variety	degree day	harvesting humidity[a]	oil content[a]	oil mass expressed (g)
1	406	4.8	43.8	0.87	5	430	10.1	41	0.65
1	508	6.1	46.6	0.99	5	532	11	45.7	0.82
1	639	10.1	50.4	0.80	5	663	10	44.6	0.81
1	690	6	46.5	0.80	5	714	8.2	44.5	0.73
1	741	7.3	50.8	0.83	5	765	8	45.4	0.76
1	780	7.8	47.3	0.75	5	804	7.6	47.2	0.78
1	1106	8.8	46.8	0.84	5	1130	11.4	46.2	0.72
2	406	10.8	40.6	0.75	6	420	7.5	39.9	0.78
2	508	8.9	46.6	0.83	6	522	5.9	46.3	0.93
2	639	9.2	47.9	0.73	6	653	9.3	46.4	0.81
2	690	6.1	44.4	0.67	6	704	5.8	46.7	0.92
2	741	6.5	43.5	0.72	6	755	6.9	44.8	0.74
2	780	7.5	44.4	0.73	6	794	6.8	44.5	0.78
2	1106	11.6	46.6	0.67	6	1120	9.4	45.7	0.80
3	406	8.3	42.9	0.75	7	420	8.3	46	0.69
3	508	8.7	45.2	0.86	7	522	6.4	44.7	0.83
3	639	12.2	47.8	0.73	7	653	9.2	45.6	0.69
3	690	6.1	44.8	0.67	7	704	5.6	46.6	0.75
3	741	9.1	41.5	0.74	7	755	6.8	44.5	0.77
3	780	7.8	48.7	0.73	7	794	7	43.7	0.73
3	1106	9.9	42.6	0.75	7	1120	8.8	45.6	0.76
4	445	8	39.9	0.82					
4	547	5.6	46.3	0.90					
4	678	9.3	46.4	0.81					
4	729	6	45.5	0.81					
4	780	7	44.2	0.82					
4	819	7.7	46.4	0.72					
4	1145	9.5	47.3	0.73					

Table 2: evolution of the seed composition and of the extracted oil mass according to the harvesting date and the variety. [a] expressed on dry basis. Inserted in Table 2 shows the evolution of degree day in function of harvesting date for variety 6.

A correlation between the model parameters and the mass of oil extracted during expression was established by multiple regression on standardized values. The table 2 presents the composition of the seeds for each harvesting date with the corresponding mass of oil extracted. A maximum in oil mass can be observed for the second date (between 508 and 547 degree day). The correlation obtained explained 93.6% of the variability in oil mass. The expression of the correlation is:

$$\text{oil mass} = -5.38.10^{-11} - 2.456\text{x}G + 0.056\text{xG}_{1_0} - 0.040\text{xG}_1 + 0.323\text{xG}_2 + 0.246\text{xG}_3$$
$$-1.481\text{xh}_\infty - 0.233\text{xt}_{r_0} + 0.346\text{xt}_{r_1} - 0.259\text{xt}_{r_2} - 0.019\text{xt}_{r_3}$$

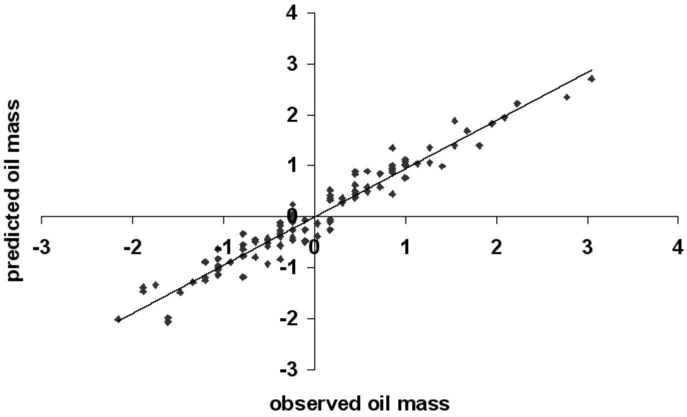


Figure 7: Observed oil mass versus predicted oil mass obtained with the multiple regression correlation.

The Figure 7 presents the relation between the observed and the predicted values, as the obtained curve is a straight line the correlation show a good agreement with experimental data.

The global compressibility modulus obtained are between 1.94 Pa for variety 1 at 690 degree day and 2.76 Pa for variety 2 at 1106 degree day. These values are compared with those obtained previously by the application of the same model on data purchased by hydraulic expression of 200 g of mature linseed at 7.5 MPa and 55°C (Lanoisellé, 1996). Our values for mature seeds are between 2.39 for varieties 1 and 3 and 2.76 for variety 2 at 1106 degree day, the global compressibility modulus for the 200 g test was 2.44 Pa. Then this model is adequate for the determination of the compressibility modulii at different scales.

5. Conclusion

The present study has permitted to characterize the oil point during the constant speed period preceding the constant pressure expression. The modelling of the constant pressure phase by a four exponential model analog to a Kelvin Voigt model leads to a good agreement between experimental data and model. The obtained model parameters permit to explain 93.6% of the variability in the mass of oil expressed. The global compressibility modulii determined for mature seeds is comparable with those found with the same model applied on larger amount of seeds (200 g).

6. Nomenclature

G_i	Compressibility modulus for an unitary stress	Pa
G	Global compressibility modulus for an unitary stress	Pa
GDD	Growing degree day	°C
h	Thickness of the press cake	m
q	Mass velocity of liquid expression in eq.3 and eq.4.	$kg.s^{-1}$
SME	Specific mechanical energy	$J.kg^{-1}$
t	Time	s
$T_{base} = 5$ °C	Reference temperature	°C
T_{max}	Daily maximal temperature	°C
T_{min}	Daily minimal temperature	°C

tr_i	Characteristic time	s
U	Consolidation rate	–
Greek letters		
λ	Coefficient	–
ε	Volumetric bed strain	–
σ_i	Compressibility modulus ratio in eq.5 and eq.6	–
υ_i	Time factor	s^{-1}
Subscripts		
0	Initial values	
1_0	Values of parameters for primary extra-particular consolidation	
1	Values of parameters for creep extra-particular consolidation	
2	Values of parameters for extra-cellular consolidation	
3	Values of parameters for intra-cellular consolidation	
c	Value at the beginning of the constant pressure phase	
∞	Theoretical values of parameters for infinite time expression	

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