A one dimensional model for the prediction of extraction yields in a two phases modified twin-screw extruder

L. Prat^{a,*}, P. Guiraud^a, L. Rigal^b, C. Gourdon^a

^a Laboratoire de Génie Chimique-UMR 5503 CNRS, INP-ENSIACET-18, chemin de la loge-31078, 118 rte de Narbonne, 31077 Toulouse, Cedex 4, France

^b Laboratoire de Chimie Agroindustrielle UA INRA 31A1010, INP-ENSIACET-118 route de Narbonne-31077, Toulouse, Cedex, France

Abstract

Solid/liquid extraction is performed on raw plant substrate with a modified twin-screw extruder (TSE) used as a thermo-mecanochemical reactor. Visual observations and experimental residence time distributions (RTD) are used to develop a solid transport model based on classical chemical engineering method. Modeled and experimental residence times are compared. The transport model is then coupled with a reactive extraction model in order to predict extraction yields.

Keywords: Twin-screw extruder; Solid/liquid extraction; Two-phase residence time distribution; Transport phenomena; Raw plant substrate

1. Introduction

The twin-screw extruders (TSE) ability to carry out simultaneous combination of several unit operations in a single continuous reactor makes it very valuable for the valorization of agroressources wastes [1]. For instance, N'Diaye et al. [2,3], but also Bouzid et al. [4] or Isobe et al. [5] have demonstrated the feasibility of extraction processes from various raw plant matters.

Part of Fig. 1 shows the process operating principle for this type of two-phase application. This type of machine consists of a seven eight-shaped elements barrel enclosing two corotating intermeshing screws. Raw plant matter and extraction solvent are fed into the barrel by the first transport screws and are mixed together in the transport and kneading elements. As the liquid is absorbed by the matter, solubilization occurs. Further down, reversed transport screw elements (RSE) cause severe compression of the vegetable matter and allow the separation of the two phases. Liquid phase is then retrieved through a barrel filter located just upstream from the RSE.

As it is clearly demonstrated on Fig. 2 for the case under consideration in this paper (extraction of hemicelluloses from hardwood populus via alkaline solubilization), TSE allows to obtain higher extraction yields for reduced contact times in comparison with a batch reactor. This comparison points out the main interest of performing vegetable extraction processes with a TSE.

In a previous work, Prat et al. have proposed a description of the functioning of the TSE for such twophase processes, focusing on Residence Time Distributions (RTD) of the phases and on the relative contribution of the screw elements to the RTD under the influence of the operating parameters (feed rates, screw rotational speed, NaOH concentration in the alkaline solvent).

If the ability of TSE to carry out high performance solutes extraction from vegetable matters does not have to be more demonstrated, no tool allows, up to now, to predict the extraction yields of this process as a function of the operating parameters. The aim of this work is to propose a one dimensional model for this prediction, on



Fig. 1. Global process in a modified CLEXTRAL BC45 TSE and model processing.

the basis of the example of hemicelluloses extraction from hardwood populus by NaOH aqueous solution. This model couples a one dimensional approach for the solid transport along the TSE and a kinetic description of the solubilization process which has been established via batch experiments. These kind of approach has been successfully applied to a lot of other polyphasic processes like liquid–liquid extraction columns for instance.

The first paragraph proposes a system analysis, leading to the modeling methodology. In the second paragraph, the models describing transport, reaction and mass transfer are developed. The third paragraph presents the matter, the material and the experimental methods. Then, in the last paragraph, the model results are presented and compared with the experimental data.



2.1. Two-phase transport by the TSE

Prat et al. [6] have proposed a qualitative description of the functioning of the TSE and studied the two phase RTD in this particular application. The reactor has been separated into two functional parts: before the RSE, the two phases (liquid and impregnated solid) are present, meanwhile from the RSE to the die, only impregnated solid can be found. In this second part, the underpressured elements (i.e. the RSE, the half transport screw just before the RSE and the die) are totally filled with the solid matter, which is not the case in the first part of the reactor. The TSE is then not totally full of matter under classical working conditions.



Fig. 2. Hemicelluloses extraction yields in TSE and in batch reactor.



Fig. 3. TSE opening experiments results (*, solid weight percentage, *, solid mass, —calculated solid mass from experimental residence time).

Visual observation and RTD study prove that the solid accumulates in the kneading elements and in the RSE (as can be seen in Fig. 3 for the central functioning point of the experimental domain). This confirms the known restrictive effects [7] of these elements in term of transport. As far as solvent extraction is concerned, the first part is the more important because it is where the extractive reaction occurs. The second part, from the RSE to the die, is necessary for the solid/liquid final separation and, as a consequence, for the correct functioning of the reactor.

2.2. Reactive extraction from raw plant substrates

A description and a modeling for the reactive extraction of hemicelluloses from poplar wood in batch reactor have been proposed by Prat et al. [8]. Poplar cells are assumed to be cylinders hollowed by a central canal. The hemicelluloses are situated in the cells walls [9].

The kinetic and transport phenomena taking place during the solid-liquid extraction of hemicelluloses from popular wood are separated in five steps [8], as described in Fig. 4:

- solvent (NaOH solution) axial diffusion from the bulk phase to the central canal in the cells;
- solvent radial diffusion from the central canal to the reactional sites into the walls (primary and secondary walls);
- NaOH reaction, liberation and solubilization of the hemicelluloses;
- hemicelluloses radial diffusion into the walls;
- hemicelluloses diffusion from the central canal to the bulk phase.

The conducted experiments gave data for hemicelluloses concentrations in the central canal and in the bulk phase as a function of time in different operating



Fig. 4. Hemicelluloses extraction steps.

conditions in term of temperature and NaOH concentration.

From these experimental data, a parameter identification allowed to quantify the diffusion coefficients and the reaction kinetic law which is of the form:

$$-\frac{\mathrm{d}C_{\mathrm{L}}(t)}{\mathrm{d}t} = kC_{\mathrm{L}}(t) = 0.047\sigma^{3.4} \,\mathrm{e}^{-18900/RT}C_{\mathrm{L}}(t) \tag{1}$$

where $C_{\rm L}(t)$ is the concentration of the non-solubilized hemicelluloses, σ , the NaOH concentration in the solvent and T, the temperature.

2.3. Modeling principle

The present work separates the reactor into local functional elements. Two different kinds of elements will be distinguished, the elements where the reaction occurs (the transport screw elements) and the elements where compression and then solid/liquid separation is combined with the reaction (the kneading elements, the reversed screw elements and the half transport element before the RSE).

In a first step, the solid residence time is calculated for each screw element. The equations are described in paragraph Section 3.1.

Then, the resulted local residence time is used with the kinetic law (1) to calculate the liberated mass of hemicelluloses from the wood structure.

Finally, in the restrictive elements, a local mass balance is done in order to quantify the mass transfer and obtain the hemicelluloses mass in the liquid phase. Some simplifications will be done, considering the mechanical effect of the TSE.

This model processing is described in Fig. 1.

3. Global process model

3.1. Transport model

Some hypothesis are made in the transport model in order to simplify the coupled phenomena, the physicochemical properties and changes of these properties.

All the elements of the screw are considered as independent from the others (there is no retroaction of the behavior). Consequently, the transport phenomena properties linked to the element only depend on their geometry. This hypothesis has been justified by a first local approach in previous work [6] and give a good representation of the matter transport.

The transport will be described in terms of mean residence times. It will then be possible to obtain the two phase masses (the mass is the product of the mean residence time by the mass flow), and, with the values of the densities, the occupied volumes. As the model consider each screw element as an independent module, the physico-chemical properties of the phases keep a constant value in each of the screw elements.

3.1.1. The transport screw elements

Except for the half transport screw just before the RSE, it will be considered that the matter is pushed by the screws and moves forward at a maximum mean velocity equals to Np (with N, the screw rotational speed, and p the pitch). To represent the actual velocity of the matter, a slip velocity of the matter with the screw is introduced. It will permit to consider the friction of the matter with the screw and with the barrel. The residence time in the transport screw elements τ_{screw} will then be modeled by:

$$\tau_{\rm screw} = \frac{L}{Np(1-\alpha)} \tag{2}$$

where, L is the length of the element; N, the screw rotational speed; p, the pitch of the element and $Np\alpha$ the slip velocity of the matter relative to the screw.

As this expression does not take the volume into account, a test will be made in order to verify that the matter volume implied by τ_{screw} does not exceed the maximum authorized by the geometry of the system. As this never happen, no other equation will be propose in this work.

3.1.2. The restrictive elements in the first part of the reactor

The restrictive elements in the first part of the reactor (i.e. the two kind of kneading elements) are totally filled. As the radial and axial mixing in these elements are important, the general expression of the mean residence time τ can be expressed as follows:

$$\tau = \frac{V}{\dot{V}} \tag{3}$$

where, V is the volume of the element; and \dot{V} the volumic flow of matter in the element.

Since there are two phases in these elements, the volume is shared between them. The volumic flows (\dot{V}_{sol} for the solid and \dot{V}_{liq} for the liquid) are of the form:

$$\dot{V}_{\rm sol} = \frac{Q_{\rm S}}{Q_{\rm P}} \tag{4}$$

and

$$\dot{V}_{\rm liq} = \frac{Q_{\rm L} - Q_{\rm S}((1-\omega)/\omega)}{\rho_{\rm liq}}$$
(5)

where, $Q_{\rm L}$ is the mass flow of liquid in the reactor; $Q_{\rm S}$, the mass flow of solid; ω the solid weight percentage in the solid phase in the element; $\rho_{\rm P}$, the apparent density of the solid particles and ρ_{liq} , the density of the liquid phase.

Furthermore, the total volume V of the element is:

$$V = V_{\rm sol} + V_{\rm liq} \tag{6}$$

where, V_{sol} is the volume occupied by the impregnated solid; and V_{liq} , the volume occupied by the liquid phase.

As the phases hold up is unknown, β , the ratio between the solid phase and the liquid phase velocities can be expressed by:

$$\beta = \frac{\dot{V}_{\rm sol} V_{\rm L}}{\dot{V}_{\rm liq} V_{\rm S}} = \frac{\tau_{\rm liq}}{\tau_{\rm sol}} \tag{7}$$

where τ_{sol} , is the solid phase residence time in the kneading element; and τ_{liq} , the liquid phase residence time in the kneading element.

Combining and rearranging Eqs. (3)–(7), gives:

$$\begin{cases} \tau_{\rm sol} = \frac{V\rho_{\rm P}}{Q_{\rm S} + (\rho_{\rm P}/\rho_{\rm liq})(Q_{\rm L} - Q_{\rm S}((1-\omega)/\omega))\beta} \\ \tau_{\rm liq} = \beta\tau_{\rm sol} \end{cases}$$
(8)

3.1.3. The restrictive elements in the second part of the reactor

The restrictive elements in the second part of the reactor (i.e. the RSE, the half transport screw just before the RSE and the die) are supposed to be totally filled. Equation Eq. (3) is still available, even if in this part of the reactor, there is only impregnated solid.

From Eq. (2), and with the corresponding volumes and density, the residence time in the RSE τ_{rse} and in the die τ_{die} are:

$$\tau_{\rm rse} = \frac{V_{\rm rse}}{Q_{\rm S}} \rho_{\rm rse} \tag{9}$$

and

$$\tau_{\rm die} = \frac{V_{\rm die}}{Q_{\rm S}} \rho_{\rm die} \tag{10}$$

where $\rho_{\rm rse}$ is the apparent solid density in the RSE and $\rho_{\rm die}$ is the apparent solid density in the die.

3.2. Reaction and mass transfer model

In [8], Prat et al. proposed a first order kinetic law for the hemicelluloses liberation (1).

Concerning the mass transfer, as the solid matter is under pressure in the restrictive elements, the hemicelluloses diffusion is neglected compared with the convection due to the pressure. In these elements, the solid weight percentage goes from ω_1 to ω_2 ($\omega_1 < \omega_2$). Then, the liquid mass m_{liq} which transfers from a solid mass m_{sol} to the liquid phase is expressed by the following equation:

$$m_{\rm liq} = m_{\rm sol} \left(\left(\frac{1 - \omega_1}{\omega_1} \right) - \left(\frac{1 - \omega_2}{\omega_2} \right) \right) \tag{11}$$

4. Matter, material and experimental methods

The local models previously described need different kind of parameters:

- elements geometrical parameters (pitches, length, volumes);
- operating parameters (N, Q_L, Q_S) ;
- indirect responses of the system (ω , the solid weight percentages along the reactor);
- physicochemical properties (densities, kinetic laws);
- parameters describing the behavior of the matter (the β parameters, and the slip velocity).

Furthermore, experimental data for solid residence time and extraction yields are to be obtained in order to validate the model.

4.1. Model parameters acquisition

Geometrical parameters are directly measurable on the elements and operating parameters are given by the experiments conditions.

4.1.1. Solid weight percentage in the impregnated solid

Solid weight percentage ω in the impregnated solid along the reactor depends on the mechanic behavior of the screw elements. Therefore, the results obtained during the stopping and opening of the reactor allow to use mean values for all experiments. ω will only vary with z, the axis of the extruder. In all experiments, profiles of the solid weight percentage in the solid phase have quite the same values. Consequently, these values (Fig. 3) will be used in the model, considering one value for each of the screw elements.

4.1.2. Densities of the two phases

The liquid density will be considered as a constant, even if its composition changes with the progress of the extractive reaction (at the worst conditions for this hypothesis—i.e. the minimum liquid flow, the maximum solid flow and the maximum hemicelluloses extraction—, the density of the liquid will only varies of 5% from its initial value).

The physicochemical properties of the vegetable matter used in this work are ρ_W the real density (the density of the wood, without considering the empty space due to the porosity), ρ_P the apparent density of a wood particle, and ε the porosity. The considered

porosity is the useful porosity for the process, i.e. the porosity accessible to the solvent.

 $\rho_{\rm P}$, $\rho_{\rm W}$ and ε are linked by the following equation (without considering the contribution of air):

$$\rho_{\rm P} = \rho_{\rm W} (1 - \varepsilon) \tag{12}$$

 ε and ρ_W are intrinsic properties of the matter and can be obtained with a single experiment: a known mass of vegetable matter m_W is mixed with a known mass of solvent m_{liq} . After the transfer equilibrium is reached (i.e. at the maximum of the solid impregnation), the volume of the two mixed phases V and the solid weight percentage ω in the solid phase are measured. The porosity is then filled with the solvent and the following equations can be written:

$$\rho_{\rm w} = \frac{m_{\rm w}}{V - (m_{\rm liq}/\rho_{\rm liq})} \tag{13}$$

and

$$1 - \omega = \frac{\rho_{\text{liq}}}{\rho_{\text{liq}} + (1 - \varepsilon/\varepsilon)\rho_{\text{w}}}$$
(14)

where ρ_{liq} is the density of the solvent.

4.2. TSE experiments

4.2.1. Feedstock

Debarked chips of populus are ground to an average size of $15 \times 2 \times 2$ (mm) using an Electra type VS1 industrial grinder. The composition of the wood chips is determined using American Standard Test & Measurement Methods (ASTM D-1103, D-1106, D-1787). The wood contains 52% of cellulose, 21-24% of lignin, 16-19% of hemicelluloses, and some pectins, proteins, aromatic compounds and aliphatic acid. The moisture content of the initial chips is about 4%. Wood chips are fed in from a bin via a feeding screw and a chute (see Fig. 1 for reactor design).

The extractive solutions are injected using a volumetric pump at a single point (180 mm from the beginning of the screw).

4.2.2. Twin-screw extruder (TSE)

Experiments are performed using a modified CLEX-TRAL BC45 TSE (see Fig. 1). It is a fully intermeshing corotating TSE. Seven sections form the 1.4 m long barrel. Four sections are heated by induction belts and cooled by water circulation. Conical holes (1 mm entry per 2 mm exit) in a plate form the filter element added at the end of the barrel in order to extract the liquid from the slurry. The die consists of a 25 mm long cylindrical hole, 12 mm in diameter. Five different kinds of elements are used in the axis profile: T2F (trapezoidal two-flight screw), C2F (two-flight screw), MAL2 (10 mm bi-lobe kneading element) always positioned with a $\pi/2$ shift (i.e. in a neutral position for transport compared with conveying screw elements), MAL0 (10 mm right-handed mono-lobe kneading element), positioned with a $\pi/13$ shift (i.e. in a positive position for transport) and C2FC (25 mm pitch reversed two-flight screw element). 100 mm in length elements were principally used but 50 mm in length elements were also available. The pitch of the transport screw elements varies from 66 to 25 mm. All experiments are carried out with a reverse screw element with peripheral slots grooved in the screw flight for leakage flow.

4.2.3. Experimental processing

The domain of work is limited by several conditions [2]. First, clogging of the reactor has to be avoided, but the more important is the correct functioning of the RSE. One function of this element is to allow the matter to form a 'plug'. This plug is necessary to press the solid in order to separate the two phases, but unfortunately, it only appears under specific conditions. Furthermore, the RSE can totally stop the transport of solid matter and, as a consequence, the TSE. Consequently, the 'authorized' range of parameters is organized around a central functioning point in terms of screw rotational speed N, solid feed rate $Q_{\rm S}$, liquid feed rate $Q_{\rm L}$ and NaOH concentration σ . The ranges of these parameters are reported on Table 1. All the experiments are conducted at a fixed barrel temperature of 55 °C (the best temperature to obtain good extraction yields and to limit degradation of hemicelluloses). Furthermore, for the study of the profile influence, only the kneading elements and RSE are replaced in half or totality by 33 mm pitch transport elements.

For all the experiments, the following parameters are measured after the permanent operating conditions have been reached:

- liquid and solid rates at the exit of the reactor in order to perform mass balances;
- $-\omega$, the solid weight percentage in the exiting solid phase characterizing the solid/liquid separation and also needed in order to perform the mass balance;
- R, the extraction yield (in term of hemicelluloses mass recovered in the liquid phase compared with the mass contained in the initial wood) [2];

Table 1 Experimental domain

	Symbol	Central point	Minimum	Maximum
Speed screw (rpm)	Ν	175	110	250
Solid feed (kg h^{-1})	Qs	4.8	3.2	6.7
Liquid feed(kg h^{-1})	$Q_{\rm L}$	36.5	21.5	51.5
NaOH massic % in	σ	5	3	7
liquid				

- τ_{sol} , the mean residence times of the solid phase (the mean residence times determination is described in [3]).

After these measurements, the extruder is brutally stopped and opened in order to observe the solid situation.

5. Modeling results

5.1. Transport model results

5.1.1. Model analysis

The global residence times for the solid and the liquid phases are of the form:

$$\tau_{\rm S} = \sum_{\rm transport \ screws} \frac{L}{Np - v_{\rm S}} + \sum_{\rm kneading \ elements} \times \frac{V\rho_{\rm P}}{Q_{\rm S} + (\rho_{\rm P}/\rho_{\rm liq})(Q_{\rm L} - Q_{\rm S}(1 - \omega/\omega))\beta} + \frac{V_{\rm rse}\rho_{\rm rse}}{Q_{\rm S}} + \frac{V_{\rm die}\rho_{\rm die}}{Q_{\rm S}}$$
(15)

and

$$\tau_{\rm L} = \sum_{\rm transport \ screws} \frac{L}{Np - v_{\rm S}} + \sum_{\rm kneading \ elements} \times \frac{V\rho_{\rm P}\beta}{Q_{\rm S} + (\rho_{\rm P}/\rho_{\rm liq})(Q_{\rm L} - Q_{\rm S}(1 - \omega/\omega))\beta}$$
(16)

In [6], the important decrease of $\tau_{\rm sol}$ with $Q_{\rm S}$ and N, the decrease of $\tau_{\rm liq}$ with $Q_{\rm L}$ and N and the neutrality of σ towards $\tau_{\rm sol}$ and $\tau_{\rm liq}$ (as already said in paragraph Section 2) have been found. The mathematical expressions (Eqs. (15) and (16)) are in agreement with the main experimental observations upon the influences of the operating parameters on the mean residence times.

5.1.2. Adjustable parameters

The proposed model has three non-intrinsic parameters which will be considered as adjustable:

- $-\alpha$, the slip velocity between the matter and the transport elements;
- $-\beta_1$, the ratio between the solid and the liquid phase velocities in the kneading elements MAL2;
- $-\beta_2$, the ratio between the solid and the liquid phase velocities in the kneading elements MAL0.

 $Np\alpha$ the slip velocity, arises from the friction between the matter and the screw and between the matter and the barrel. It can also help to consider the transport limitation due to the phenomena in the two screw intermeshing area. It is, of course, a simplified way of representing these phenomena.

The introduction of the β parameter into the modeling of the kneading elements allows to represent the hold up of the two phases. Due to its expression, the volumes ratio can be written in a form which implies the volume flows in a proportional way:

$$\frac{V_{\rm L}}{V_{\rm S}} = \beta \frac{\dot{V}_{\rm liq}}{\dot{V}_{\rm sol}} \tag{17}$$

5.1.3. Model adjustment

Due to the great numbers of the operating parameters, in order to analyze the results obtained, the calculated times versus the experimental mean residence times will be represented for the two phases.

The difference between experimental and calculated values will be characterized by a criterion of the form:

$$\eta = \frac{1}{n} \sum_{j=1}^{n} \frac{2|\tau_{j} \exp - \tau_{j} \mod}{(\tau_{j} \exp + \tau_{j} \mod)}$$
(18)

where, *n* is the number of experiments; $\tau_{j \text{ exp}}$, the solid experimental residence time of the experiment *j*; and τ_{j} mod is the solid modeled residence time of the experiment *j*.

5.1.4. Model results

The criterion for the solid residence time α is minimized by the Gauss–Newton based method and the results are plotted in Fig. 5. The three adjustable parameters become $\alpha = 0.3$, $\beta_1 = 1.42$ and $\beta_2 = 2.05$. The criteria is $\eta = 0.17$.

The obtained value of α shows that the velocity in the transport screw element is a compromise between the flight velocity and the rotational speed due to the shearing. In the MAL elements, the matter velocity is due to the upstream pressure and the transport contribution of the flight. The values of $\beta 1$ and $\beta 2$ are higher than one, showing that the solid phase runs out

quicker than the liquid phase. This is due to the fact that the friction is more important for the solid than for the liquid phase. Furthermore, as the MAL2 is positioned with a $\pi/2$ shift, it has a low transport effect and has a value of β lower than the MAL0 element.

The results are good concerning the solid residence times, except for low values of the screws rotational speed (experiments with N = 100, the lower limit). Total liquid residence times are overvalued by the model in these conditions. Furthermore, when tracing the mass of solid phase along the reactor, (the mass is the product of the residence time in each element by the solid feed rate), the global tendencies of the experiments can be found, as shown in Fig. 3 for the central functioning point.

In the model, N only influences the global residence times by the transport screws local residence times (Eqs. (15) and (16)). But the contributions of these elements represent only 4–10% of the modeled solid residence time which can not explain the experimental variations with N. As it has be shown in [3], the screw rotational speed has a major influence upon ω , the solid weight percentage in the solid exiting phase, and then upon the behavior of the RSE which conditions the solid/liquid separation and consequently, ω . This means that N should intervene in the RSE element. But, for the purpose of solid/liquid extraction modeling in the TSE, only the first part of the reactor residence time is important as the extractive reaction occurs in this part.

The transport model presents some default for the prediction of solid residence time in the RSE at low values of the axis speed rotation (N < 100 rpm). Even if there is a lot of simplifying hypothesis, it gives good results in the main part of the experimental domain. The model needs a single adjustable parameter for each screw element type. These three parameters have the same value for all the experimental conditions. This model will then be used for the global prediction of extraction yields.



Fig. 5. Experimental vs. calculated solid residence times.



Fig. 6. Experimental vs. calculated extraction yields.

5.2. Extraction yields results

The extraction yields prediction is obtained by coupling the transport model, the reaction and the transfer model, as described in Fig. 1. The temperature used for the kinetic law is the experimental temperature measured in the TSE at the interface barrel-matter. The results are plotted in Fig. 6 for all the experiments. The global model gives a good prediction of the experimental data in the whole domain investigated.

These results lead to several observations on the adopted method and on the studied process. First of all, the good prediction justifies the utilization of coupling between a one dimensional model for transport by screw element and a first order kinetic. Besides, the tentative method in batch reactor for the acquirement of the kinetic gives results exploitable in continuous reactors, presumably because of the simplified reactional way adopted. Finally, the obtained results suggest that the contribution of TSE to solid–liquid extraction processes is firstly located in its ability to separate the two phases and then suppress the limitative diffusion step.

6. Conclusions

Problems generated by the complexity of the vegetable matter and the important coupling of phenomena in a reactor where the matter can not be directly observed are numerous. This work proposes a first approach model for the prediction of solid–liquid extraction yields in a modified TSE. The model has been constructed from visual observations and chemical engineering analysis by coupling transport, reaction and transfer descriptions.

This work proposes a one dimensional model for solid transport phenomena. The model needs a single adjustable parameter for each screw element type. These parameters characterize the mechanic phenomena between matter and TSE and depend on the couple (chosen matter-screw element). A theoretical study of these parameters could be the next step in modeling the two-phase flow transport in the TSE. As far as solid– liquid extraction is concerned, the solid residence time prediction gives usable values in all studied domain. Reaction is based on a previous work upon hemicelluloses extraction in a batch reactor from poplar wood. The hemicelluloses diffusion is neglected and mass transfer is due to the under pressure elements which cause solid–liquid separation all along the TSE.

This study allows to point out that the main interest of the TSE for solid–liquid extraction lies in the suppression of the diffusion step during the operation. Furthermore, it validates the experimental method for the obtaining of kinetic parameters in batch reactor. For other TSE applications with solid-liquid flows, a more precise study of the RSE will be needed, in order to understand the behavior of such a screw element. This understanding will be of first interest for raw material for instance. As there is only solid in the RSE, developed models for polymer industries or agro-industry [10] will certainly be usable with some modifications due to the compressibility of the wood particles.

Appendix A: Nomenclature

C(t)	Hemicelluloses mass concentration function of
	time $(g l^{-1})$
C2F	Two-flight screw element
C2FC	Reversed two-flight screw element (RSE)
$m_{\rm L}$	Liquid mass (g)
$m_{\rm S}$	solid mass (g)
MAL0	mono-lobe kneading element
MAL2	bi-lobe kneading element
N	screw rotational speed (rpm)
р	screw element pitch (mm)
$Q_{\rm S}$	solid feed rate (kg h^{-1})
$Q_{\rm L}$	liquid feed rate (kg h^{-1})
R	extraction yield (%)
RSE	reversed screw element
Т	temperature (°C)
T2F	trapezoidal two-flight screw element
V	phase volume (m ³)
\dot{V}	the volume flow $(m^3 s^{-1})$

Greek letters

- α coefficient for the determination of the slip velocity
- β kneading element adjustable parameter
- ε wood particle porosity
- η optimization criterion
- $\rho_{\rm P}$ particle apparent density (kg m⁻³)
- $\rho_{\rm Liq}$ liquid density (kg m⁻³)
- σ NaOH mass percentage in liquid phase
- $\tau_{\rm S}$ solid phase mean residence time (s)
- ω solid weight percentage in the exiting solid phase

References

- L. Rigal, Technologie d'extrusion bi-vis et fractionnement de la matière végétale, 40 ans d'extrusion bi-vis chez CLEXTRAL, Recueil de conférences, Firminy (France), 8–10 October 1996.
- [2] S. N'Diaye, Fractionnement de la matière végétale: mise au point d'un procédé thermo-mécano-chimique et modélisation du réacteur bi-vis. doctorat thesis INPT, Toulouse, France, 1996.
- [3] S. N'Diaye, L. Rigal, P. Laroque, P.F. Vidal, Extraction of hemicelluloses from poplar, populus tremuloides, using an

extruder-type twin screw reactor: influence of the main factors, Biores. Technol. 57 (1996) 61-67.

- [4] N. Bouzid, G. Vilarem, A. Gaset, Extraction des huiles essentielles par des techniques non conventionnelles, Revista Italiana E.P.P.O.S., Agosto'97, 3–11, 1997.
- [5] S. Isobe, F. Zuber, K. Uemura, A. Nogushi, A new twin-screw press design for oil extraction of dehulled sunflower seeds, J. Am. Oil Chem. Soc. 69 (9) (1992) 884–889.
- [6] L. Prat, P. Guiraud, L. Rigal, C. Gourdon, Two phase residence time distribution in a modified twin-screw extruder, Chem. Eng. Proc. 38 (1999) 73–83.
- [7] R.E. Altamore, P. Ghossi, An analysis of residence time distribution pattern in a twin-screw cooking extruder, Biotechnol. Prog. 2 (3) (1986) 157–163.
- [8] L. Prat, P. Guiraud, L. Rigal, C. Gourdon, Solid–liquid reactive extraction with raw plant substrate, ECCE2 congress, 1999.
- [9] E. Sjötröm, Wood Chemistry, Fundamentals and Applications, Academic Press, Inc, NewYork and London, 1981.
- [10] J. Tayeb, B. Vergnes, G. Della Valle, A basic model for a twinscrew extruder, J. Food Sci. 54 (1989) 207–214.