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Application of a thermally assisted mechanical dewatering process to biomass

A. Mahmoud ^{a,b}, P. Arlabosse ^{a,b,*}, A. Fernandez ^{c,d}

^a Université de Toulouse, Mines Albi, CNRS, Campus Jarlard, F 81013 Albi cedex 09, France

^b Ecole des Mines Albi, Centre RAPSODEE, Campus Jarlard, F 81013 Albi, France

^c Université de Toulouse, INSA, UPS, INP, LISBP, 135 Avenue de Rangueil, F 31400 Toulouse, France

^d INRA, UMR792 Ingénierie des Systèmes Biologiques et des Procédés, CNRS, UMR5504, F 31400 Toulouse, France

A B S T R A C T

Thermally assisted mechanical dewatering (TAMD) is a new process for energy efficient liquid/solids separation which enhances conventional device efficiency. The main idea of this process is to supply a flow of heat in mechanical dewatering processes to favour the reduction of the liquid content. This is not a new idea but the proposed combination, especially the chosen operating conditions ($T < 100$ °C and $P < 3000$ kPa) constitutes an original approach and a significant energy saving since the liquid is kept in liquid state. Response surface methodology was used to evaluate the effects of the processing parameters of TAMD on the final dry solids content, which is a fundamental dewatering parameter and an excellent indicator of the extent of TAMD. In this study, a two factor central composite design was used to establish the optimum conditions for the TAMD of alfalfa biomass. Experiments were carried out on a laboratory compression cell. Experiments showed that the dewatering enhancement results only from thermal effects. With a moderate heat supply ($T_{\text{piston}} = 80$ °C), the dry solid content of the press cake can reach 66%, compared to 36% at ambient temperature. A significant regression model, describing changes on final dry solids content with respect to independent variables, was established with determination coefficient, R^2 , greater than 88%. With an energy consumption of less than 150 kWh/m³, the use of the TAMD process before a thermal drying process leads to an energy saving of at least 30% on the overall separation chain.

Keywords:

Alfalfa

Compression

Fractionation

Heat Transfer

Intensification

Response surface method

1. Introduction

The prospects of a reduction in the oil supply and also an increase in the energy demand from developing countries encourage the development of renewable energy sources such as biomass and combustible waste materials. The total amount of waste materials produced in Europe was estimated at 1900 Mt/an [1]. An important part of this flow is composed of

biomass: waste and by products of the exploitation and transformation of wood, waste of agriculture, waste and by products of the food industry, urban and industrial sewage sludge. With an annual flow estimated of 1386 Mt/an [1], waste of agriculture (straw, bagasse...) and food industry represent most of these resources. These organic waste materials, which have a high heating value on a dry basis, could be valuable energy sources. But the water content of these

* Corresponding author. Université de Toulouse, Mines Albi, CNRS, Campus Jarlard, F 81013 Albi cedex 09, France. Tel.: +33 563 49 32 37; fax: +33 563 49 32 43.

E mail address: Patricia.Arlabosse@mines-albi.fr (P. Arlabosse).

materials reduces their energy value. For example, bagasse has a gross specific energy of 19.3 MJ/kg dry, but only 7.6 MJ/kg when its moisture content is 54 wt% wb [2]. Moreover, the solid fuels resulting from these waste have the advantage of being storable and transportable on long distances and find a natural outlet in strongly consuming energy industries, like cement factories or paper mills. A first step towards a purely energy valorization of these waste requires to develop low energy consuming dewatering technologies.

Different forms of pressure assisted dewatering (filter presses, belt presses, centrifuges...) can be used to reduce the liquid content of pasty or solid waste. The separation performances depend on both the product and the technology. At best, the liquid content can be reduced to 80% and more rarely 60%, with an energy consumption of 1–10 kWh m⁻³. Anyway, for many applications, mechanical dewatering cannot ensure sufficiently low liquid content and thermal drying should be used. Though successful, the typical energy consumption of efficient dryers is of the order of 800–1000 kWh m⁻³ of water removed [3]. Thus, the energy efficiency of the overall solid/liquid separation process is related to both the liquid content at the dryer inlet and the dryer efficiency. Consequently, there is a lot of research at present into methods that enhance the dewatering ability of conventional mechanical processes. Intensification of mechanical dewatering processes can take several forms: simultaneous application of an electric field [4,5], possibly pulsed [6,7], superimposition of ultrasounds [8,9] or with heat supply [10–13]. For a few years now, we specifically investigate a thermally assisted mechanical dewatering (TAMD) process, which couples mechanical dewatering at low pressure (usually $P_{\text{applied}} < 1000$ kPa) with a moderate heating ($T < 80$ °C) of the walls of the apparatus in contact with the product. The proposed combination of pressure and temperature leads to the reduction of the water in liquid phase and, for some products, results in a significant energy saving on the overall separation process. For instance, after processing bentonite sludge clay suspension used as a sealing agent in the construction industry under moderate operating conditions ($T_{\text{wall}} = 90$ °C and $P_{\text{applied}} = 340$ kPa, followed by a final pressure step to 1650 kPa), the dry solid content reaches 81.43% [14]. Under the same operating pressures but at ambient temperature, the dry solid content of the bentonite sludge would reach only 30%, insufficient for land filling according to the regulation. For this specific application, the energy consumption of the TAMD process was estimated at 69.8 kWh m⁻³ [15]. The TAMD technology is slightly different from the Mechanical Thermal Expression (MTE) process, first investigated in the mid 1990s by Strauss and coworkers [16] and recently extended by Hoadley and coworkers in Monash University [10,17]. In its current design, the MTE comprises a preheating step prior to filtration and consolidation. The processing temperature is set above the normal boiling point of the water. To prevent evaporation, the process is held under a sufficient back pressure. Consequently, upon exposure to atmospheric pressure, a flash evaporation occurs and contributes to further moisture reduction. The MTE process has been successfully applied to low rank coals and brown coals as well as paper waste [18]. For a MTE plant with heat recovery operating at 200 °C, the moisture in lignite could be reduced from 60 to 28 wt% with an

estimated energy requirement of approximately 20% of that required in an evaporative process without heat recovery, i.e. 160 kWh m⁻³. The effectiveness of MTE dewatering of sewage biosolids and bagasse were also investigated successfully [10]. For bagasse, MTE can remove approximately 55% of the inherent water at moderate processing conditions of 150 °C and 12 MPa. The main part of the moisture reduction was attributed to the collapse of the internal material porosity, resulting from the temperature increase. Other waste or by product from agriculture or the food industry does not have this porous structure. The effectiveness of the thermally assisted mechanical dewatering process still has to be proved and the optimum processing conditions has to be determined. In the present paper, we propose an application of the TAMD process to a specific biomass, the alfalfa. Alfalfa is the most cultivated leguminous plant in the world. Characterized by a cultivation with weak environmental impact, the possible uses of this plant are the subject of research, for example, in phytoremediation of soils [19] or as a raw material [20–25]. Used as forage for animal feedings, alfalfa is usually industrially dried to guarantee a good conservation. The dry solid content, between 20 and 25% during the harvest, reached 87% after drying. Most of the time, alfalfa is dried directly but the increase in the cost of energy endangers the profitability of these processing plants. In the so called ProXan Process [26], alfalfa is pressed to extract a juice fraction, rich in proteins and pigments, prior to drying. After coagulation, centrifugation and drying, a leaf protein concentrate (LPC), called PX[®], is obtained and marketed in France. Nevertheless, the economic viability of the process depends to a large extent on the utilization of the press cake as a high quality ruminant feed [27]. With the emergence of biorefineries, production of platform chemicals [28], such as lactic acid and lysine, from both press fractions or integrated production of solid fuel from the press cake and biogas from the juice fraction [29] are under development. The mechanical fractionation of the primary raw material into a liquid fraction the green juice and a solid fraction the press cake is thus an essential process unit of a green biorefinery plant. The objectives of the study are to quantify the efficiency of the TAMD process, to determine if the separation enhancement, if any, results only from thermal effects or from coupled thermo mechanical effects and then to optimize the processing conditions from the point of view of the press cake valorization.

2. Materials and methods

2.1. Experimental set-up

At the laboratory scale, the experimental set up consists of a compression cell inserted in a CARVER[®] hydraulic press (Carver Inc., Wabash, United State), which has a maximum pressing capacity of 14800 kPa and provides the pressure required to progress a downward expression.

The hydraulic press was calibrated beforehand using a load cell (Omegadyne Inc., Sunbury, USA). The applied pressure ranges from 300 to 1125 kPa. As displayed on Fig. 1, the regulation system of the standard involves large fluctuations of the applied pressure around the set value. For a reference

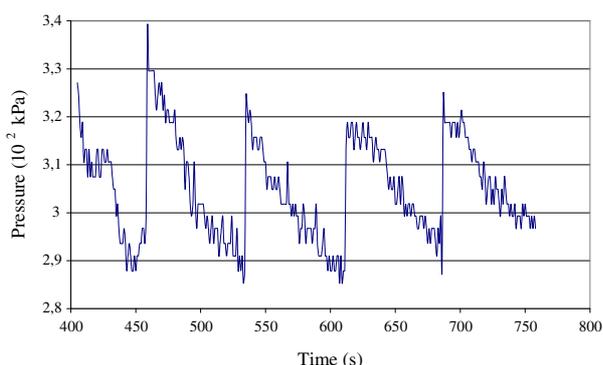


Fig. 1 Measure uncertainty introduced by the regulation system of the hydraulic press.

pressure set to 318.9 kPa, the standard deviation on the measurements is ± 10.3 kPa. For higher applied pressures, the standard uncertainty reaches a constant value of ± 8.8 kPa.

The cell (Fig. 2) consists of a compressive piston, a hollowed cylindrical vessel and a filter medium. The filtration chamber has a diameter of 148 mm and a maximum height of 60 mm. In spite of its low mechanical resistance, Teflon™ was selected as constitutive material of the vessel walls to minimize the frictions with the piston. Consequently, a stainless steel external jacket was added to ensure the mechanical resistance of the unit. The cell is fitted with a planar medium of 165.2 cm² area, which is composed of a microfibers glass filter (Whatman International Ltd, Maidstone, UK) deposited on a Teflon™ grid. To investigate the temperature impact, three electric resistances are inserted in the upper part of the compressive piston, made of copper to reduce thermal inertia. Each resistor can supply up to 350 W. Three thermocouples, including the one for the temperature regulation, are introduced into the piston. Knowing the temperatures at various

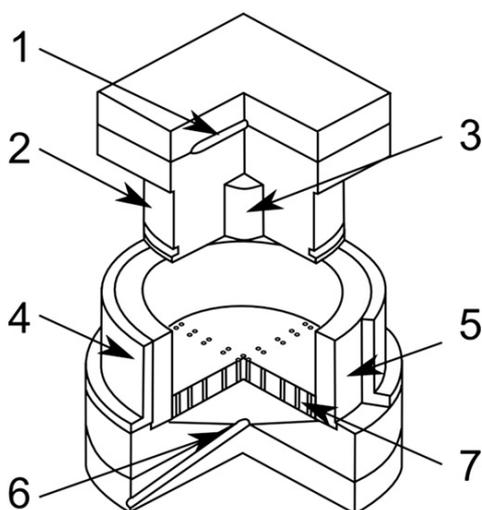


Fig. 2 Detail of the experimental cell. (1) Electric resistance; (2) Copper piston; (3) Fluxmeter; (4) Steel jacket; (5) Teflon vessel; (6) Filtrate collector; (7) Filter medium.

positions from the front side of the piston, the inverse conduction problem in the copper wall can be solved to estimate the heat flux density exchanged between the compressive piston and the product [30] and thus the thermal energy consumption of the TAMD process. The accuracy of these sensors with their acquisition lines was estimated at ± 0.1 °C.

The filtrate recovered in the collector flows out into a container laid on the computer interfaced balance. The mass is automatically recorded at set time intervals of 1 s. Finally, a movement sensor gives the thickness of the sample according to time.

2.2. Experimental procedures

About 250 g of alfalfa stems are chopped into pieces of 5 cm length before being introduced at room temperature into the TAMD process. The dewatering procedure consists of three successive stages:

- (1) a first compression at room temperature with a pressure applied of 300 kPa. If a flow is observed, these conditions are maintained during 45 min;
- (2) a second stage during which the applied pressure is kept to 300 kPa and the piston is heated to the selected temperature. These operating conditions are kept during 3 h, duration which is necessary to reach the thermal equilibrium in the press cake;
- and (3), once the thermal equilibrium in the cake has been achieved, a second expression stage at the selected operating temperature with a higher applied pressure. This increment of pressure is applied during 2 h.

At the end of the experiment, the press cake is weighed. Its dry solid content, defined as the weight of the dry sample divided by the weight of the wet sample times 100, is measured according to the AFNOR standard procedure N° X31 505. This procedure recommends a drying at 105 °C for 24 h. To check that the mass balance is preserved during the experiment, the dry solid content of the fresh alfalfa is determined using the same protocol and the moisture content of the press cake at the end of the experiment is computed knowing the mass of the juice fraction.

2.3. Feed material and storage conditions

Alfalfa (*Medicago sativa* L.) was field chopped at the pre blooming stage in a farm in the south of France. Approximately 300 mm long fresh stems with leaves were sampled at weekly intervals in the fall of 2007. The fresh alfalfa was transported to the laboratory and stored at 4 °C within 2 h after the harvest.

The impact of storage conditions on the dry solid content of the fresh alfalfa was investigated. In the first fresh sample, $3 \times 100 + 50$ g of alfalfa were selected randomly immediately after harvest. The initial dry solid content of this harvested alfalfa, measured on the sample of 50 g, was 25.19 wt%. The three samples of 100 g each were divided into three batches. In the first batch, the plants were stored in the open air. In the second batch, the plants were stored in an airtight container. In the third one, the stems were chopped into pieces of 5 cm

Table 1 Influence of storage conditions on the dry solid content of harvest alfalfa.

	Fresh alfalfa	Storage in the open air	Storage in a airtight container	Storage in a airtight container of the chopped stems
Dry solid content of sample 1 (wt%)	25.19	51.23	24.17	28.90
Dry solid content of sample 2 (wt%)	25.19	52.16	27.09	29.16
Dry solid content of sample 3 (wt%)	25.19	55.27	24.82	24.74
Mean dry solid content (wt%)	25.19	52.89	25.36	27.6

length and stored in an airtight container. Seven days later, the dry solid content of the three batches was measured (see Table 1). The storage of the whole stems in a tight container at 4 °C avoids the desiccation of the plant. After 7 days, the dry solid content remains unchanged (+0.17%) and, visually, neither yellowing nor fading is observed. This mode of conservation was retained for the following study.

3. Statistical design of experiments

A physical phenomenon can always be represented in the following mathematical form:

$$Y = f(X_1, X_2, \dots, X_n) \quad (1)$$

where Y is the output variable of interest, X_i are the input variables on which the experimenter can act and f is a mathematical function which, as well as possible, links the variations of Y with X_i .

The usual way to investigate this phenomenon consists in fixing the level of all the variables, except one, and measuring the response according to several values of the not fixed variable. At the end of this experimentation on the first variable, the curve $Y = f(X_1)$ can be plotted. This methodology must be repeated for all the other variables, which results to a high number of experiments to perform.

The main purpose of statistically designing an experiment is to collect a maximum amount of relevant information with a minimum expenditure of time and resources: results are achieved with three to four times less tests than with usual approaches. In the language of the experimental designs, X_i are called the factors and Y the response. The main difference with the conventional method is due to the fact that, to each experiment, the levels of all the factors are changed at the same time, but in a programmed and reasoned way.

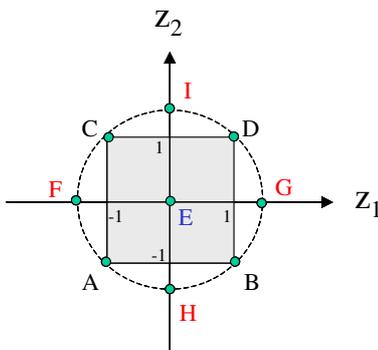


Fig. 3 Geometrical representation of circumscribed central composite design for two independent variables.

Statistically designing an experiment can meet different purposes and the type of design must be chosen consequently. The objective of the study [31] could be:

- to discriminate one a priori important factor, i.e. a factor which induces a significant change in the response for different levels of that factor. For this comparative design, randomized block designs are advised;
- to screen out the few important effects of the individual variables, called the main effects, from the many less important ones. In this case, full or fractional factorial designs should be selected;
- to estimate interaction and even quadratic effects, i.e. the effects of a variable on the response, conditional on the level of another variable, and to find improved or optimal process settings. Response surface method (RSM) is used to establish an empirical model that map the response surface using data from a design experiment and to identify the direction and parameter ranges for the response optimization.

In addition to the definition of the aim of the study, the following stages are crucial in the implementation of an experimental design [32]: the choice of the answer, the research of the factors which could be significant, the choice of their levels and finally the existence of interactions between these factors.

3.1. Design of experiments for alfalfa dewatering in the TAMD process

According to the literature, the separation enhancement in the thermally assisted dewatering process is product

Table 2 Experimental design matrix.

Run	Independent Factors			
	Actual level		Coded level	
	Temperature (°C)	Pressure (kPa)	Temperature	Pressure
1	21	1650	α	0
2	31	700	1	1
3	31	2600	1	+1
4	55.5	300	0	α
5	55.5	1650	0	0
6	55.5	1650	0	0
7	55.5	3000	0	+ α
8	80	700	+1	1
9	80	2600	+1	+1
10	90	1650	+ α	0

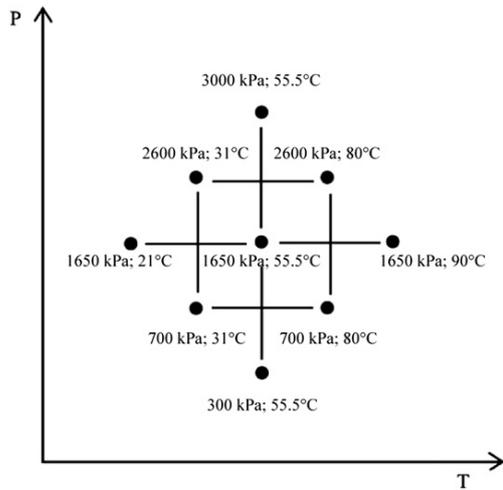


Fig. 4 Circumscribed central composite design implemented.

dependent. Prior experimental results [14] on saturated suspensions, like bentonite sludge, emphasize that significant separation enhancements, when they occur, result only from thermal effects. The objectives of the present study were to evaluate the effects of the processing parameters in a large parameter space, to determine if the separation enhancement, if any, results only from thermal effects or from coupled thermo mechanical effects and to maximize the dry solid content of the proceed alfalfa. As a consequence, RSM seems to be the most adapted design.

According to the number of factors, i.e. the two process variables, a central composite design (CCD) was selected [33,34]. The CCD consists of a 2^2 factorial design, which is the simplest and most useful design, plus two replicates of the central run and a group of 'star points' (four axial points) that permits estimation of finer features of the response surface such as curvature. More precisely, a circumscribed central composite design (CCCD) in 10 runs was retained. Indeed, CCC designs explore the largest process space and gives good accuracy of the estimates over the entire design space. The star points are at some distance α from the center based on the properties desired for the design and the number of factors in the design. The 'star points' establish new extremes for the

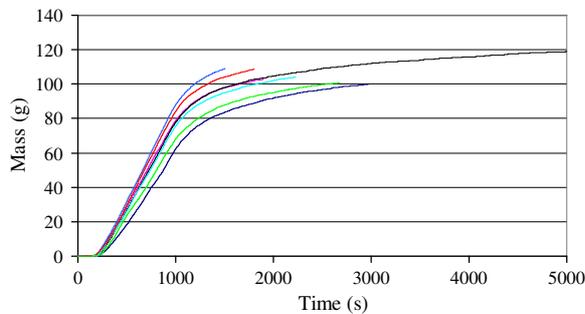


Fig. 5 Reproducibility of the experiments with plants coming from the same sampling ($T_{\text{piston}} = 80^\circ\text{C}$, $S_{\text{initial}} = 19\%$).

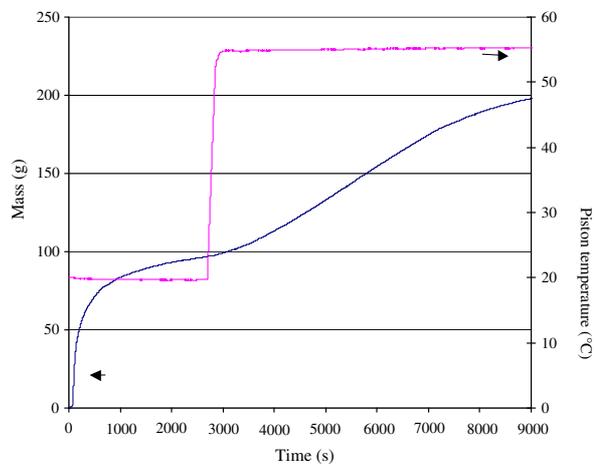
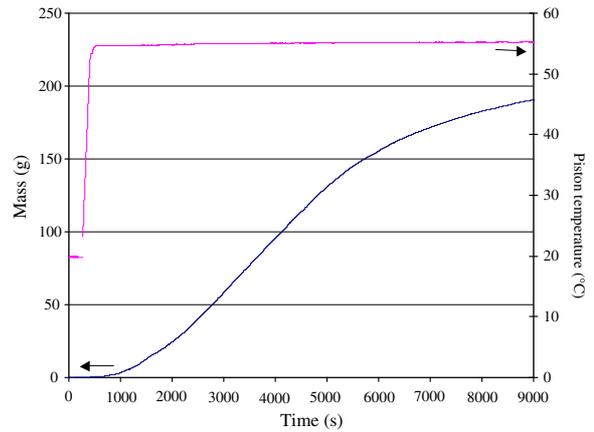


Fig. 6 Dewatering kinetics for the initially driest sample (above) and the initially moistest sample (below).

low and high settings for all factors. Fig. 3 geometrically represents the CCCD. The number of levels of a CCCD is five per factor: the central point (E), the two levels associated to the 2^2 factorial design (corresponding to the X coordinates of points A and B for the first factor for instance) and the two levels of the star points (corresponding to the X coordinates of the points F and G for the first factor for instance).

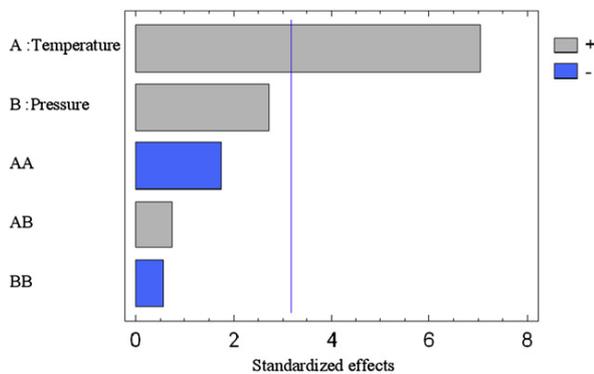


Fig. 7 Standardized Pareto chart for the final dry solid content.

Table 3 ANOVA table.

Source of variance	Sum of squares	Degree of freedom	Mean square	F ratio	p value
A: Temperature	661.275	1	661.275	49.86	0.0058
B: Pressure	99.0996	1	99.0996	7.47	0.0717
AA	40.2436	1	40.2436	3.03	0.1799
AB	4.1206	1	4.1206	0.31	0.6161
BB	7.1536	1	7.1536	0.54	0.5159
Total error	39.7851	3	13.2617		
Total (corr.)	920.225	8			

The selection of the response(s) is one of the most important stages in statistically designing an experiment. For a parameter to be a response, it has to fulfill certain conditions. A response should be: quantitative, singular, statistically effective, universal, physically realistic, simple and easily measurable. With regards to our research subject, the final dry solid content of the press cake is an excellent indicator of the extent of the TAMD process.

3.2. Implemented circumscribed central composite design

The choice of the TAMD variables and process conditions was based on former results [14,35]. The independent variables are the piston temperature, T, and the applied pressure, P. For the temperature, the lowest level associated to the 2^2 factorial design, T_{min} , was slightly above the room temperature (31 °C) while T_{max} was fixed at 80 °C to limit the temperature increase of the alfalfa. For the applied pressure, since the absolute precision on the pressure regulation is ± 10.3 kPa around the set point at low pressure range, the lowest level associated to the 2^2 factorial design, P_{min} , was fixed at 700 kPa, which leads to a relative precision of 1.47%. A maximum value of 2600 kPa was selected for P_{max} . The location of the star points on the axes of the factors depends on the selected criterion of optimality. In general, the points are set so that the errors on the regression coefficients of the empirical model be the smallest possible or the best distributed possible. Rotatability is one of these optimality criterions: the answers calculated with the

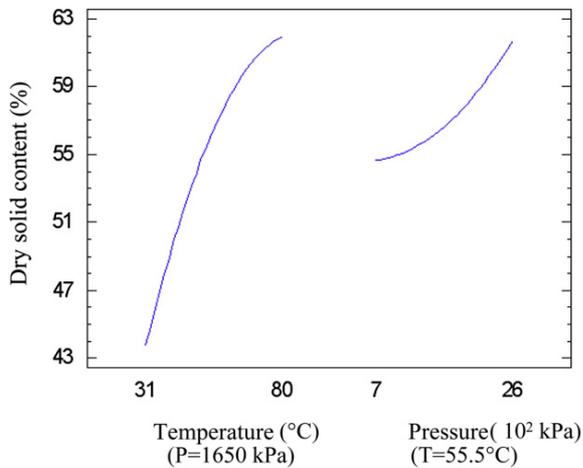


Fig. 8 Main effects of the TAMD process variables on the final dry solid content of the press cake.

model resulting from the experimental design have an identical error for all the points located at the same distance from the center of the experimental field. To maintain rotatability, the typical value of α for a 2^2 factorial design is $[2^2]^{1/4} = 1.414$. This value, which allows simultaneous rotatability and orthogonality, serves as a guide for choosing the α value, which, in any case, should also be analyzed for its convenience and feasibility. If this criterion is too restricting, near orthogonality or rotatability seems to be a reasonable criterion [32]. In our case, due to experimental constraints connected firstly to the process operation no vaporization of the liquid phase expected and secondly to the safeguarding of the proteins quality in the extracted green juice, the distance of the star points was slightly reduced from the rotatable value of 1.414 to a value of 1.15.

The experimental conditions selected by this design are listed in Table 2.

The corresponding geometrical representation is proposed on Fig. 4. With this design, the relations between the process variables and the standardized variables appearing in Fig. 3 are given by:

$$Z_1 = \frac{T - T_0}{\Delta T} \quad (2)$$

$$Z_2 = \frac{P - P_0}{\Delta P} \quad (3)$$

where T_0 and P_0 are the process variables in the center of the experimental domain, namely $T_0 = 55.5^\circ\text{C}$ and $P_0 = 1650$ kPa. ΔT and ΔP are the increments of T and P corresponding to one unit of Z_1 and Z_2 , respectively. This gives $\Delta T = 24.5^\circ\text{C}$ and $\Delta P = 950$ kPa.

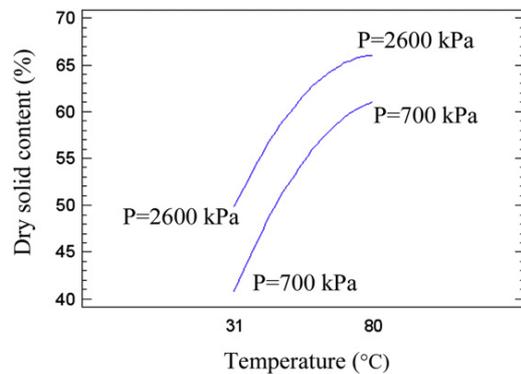


Fig. 9 Interaction effects of the TAMD process variables on the final dry solid content of the press cake.

Table 4 Observed and predicted responses.

N° Experiment	Temperature (°C)	Pressure (kPa)	Observed dry solids content (%)	Predicted dry solids content (%)
1	21	1650	32.48	36.30
2	31	700	43.59	40.82
3	31	2600	53.2	49.89
4	55.5	300	54	54.76
5	55.5	1650	56.6	56.60
6	55.5	1650	56.7	56.60
7	55.5	3000	63.19	64.71
8	80	700	60.01	61.04
9	80	2600	65.56	66.05
10	90	1650	63.56	62.02

3.3. Choice of the response function and data analysis software

The output variable (Y) can be expressed as a function of the independent variables, called the response function. A quadratic model, which is almost always sufficient in industrial application to completely describe the process behavior, was selected [36]. The choice of a quadratic model involves that, for each factor assessed, the variance is partitioned into linear, quadratic and interactive components. This model can either be calculated on standardized values or on the experimental ones. Both calculations yield useful information: the reduced centered values allow a mutual comparison of the importance of the different factors with respect to a certain response whereas the dimensional values deliver a predictive model. The full second order polynomial function, which predicts the response (Y) in all experimental regions with all possible terms, is written [33] as follows:

$$Y = a_0 + a_1T + a_2P + a_{12}TP + a_{11}T^2 + a_{22}P^2 \quad (4)$$

where Y is the predicted response, a_0 is the interception coefficient, a_1 and a_2 are the linear terms, a_{12} is the interaction term, a_{11} and a_{22} are the quadratic terms.

Knowing this approximation of the real dependence between Y and the X's, we can find directions on the $Y=f(T,P)$ surface that lead to the general vicinity of the response surface optima.

The parameters of the model were estimated from experimental data by the least squares regression using Statgraphics Centurion XV and Matlab software. The significance

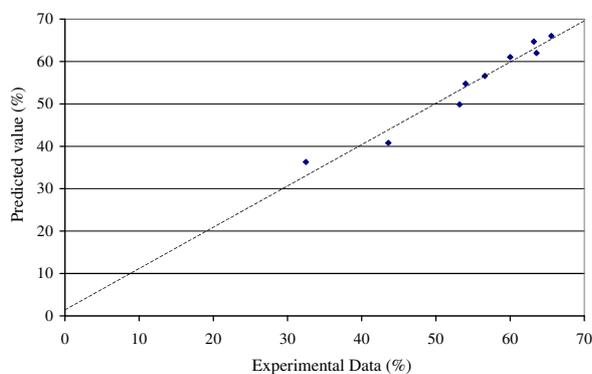


Fig. 10 Final dry solid content from the empirical model and the experimental data.

of all terms in the polynomial function was assessed statistically using the F ratio at probability (p) of 0.05, indicating a less than 5% probability of making a mistake by rejecting a true null hypothesis of no effect.

4. Results and discussion

4.1. Evaluation of process repeatability and estimate of the pure error

Usually, center points are used to evaluate the process repeatability and estimate of the pure error. But, taking into account the strong heterogeneity between two alfalfa samplings or in the proportion of stems and leaves in the various plants of one sampling, the repeatability of the process was evaluated prior to the RSM experiments. For these preliminary tests, a simplified protocol has been implemented: the third stage of the experimental procedure defined in paragraph 2.2 was not implemented. The dewatering experiments were reproduced at least 6 times with the same process operating conditions to evaluate the reproducibility of the results.

The measurement dispersion resulting from possible different proportions of stems and leaves in one sampling can be estimated from the dewatering kinetics obtained for a wall temperature of 80 °C plotted in Fig. 5. In spite of the heterogeneity of the raw material, the kinetics are appreciably the same, the standard deviation on the filtrate mass being particularly weak for $t < 1000$ s. The standard deviation on the final dry solid contents of the dewatered alfalfa is $\pm 1.17\%$.

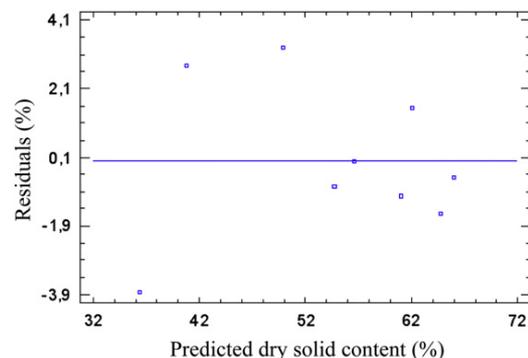


Fig. 11 Residual plot for the empirical model.

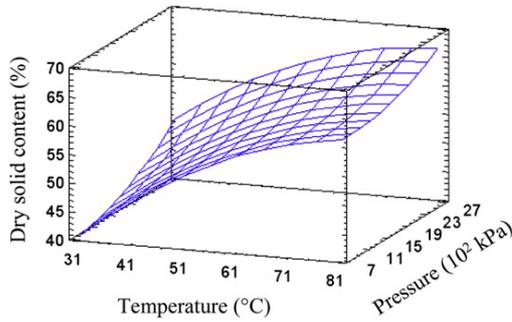


Fig. 12 Estimated response surface of the dry solids content of the alfalfa as a simultaneous function of the processing pressure and temperature.

The measurement dispersion resulting from different alfalfa samplings is of the same order of magnitude. Two samplings were performed at weekly intervals. The dry solid contents of the fresh alfalfa were 21% and 16%, respectively. The main difference in the dewatering kinetics appear during the first dewatering stage at ambient temperature, as can be seen on Fig. 6. For the initially wettest sample, 97.2 g of filtrate is recovered at ambient temperature, almost immediately after the pressurization, as can be seen on the bottom plot. For the driest sample (see the upper plot), heat supply is needed to dewater the alfalfa. Indeed, no flow is recovered during compression at room temperature (for $t < 400$ s). At the end of the compression stage at room temperature, the dry solid content of the initially driest sample is unchanged whereas that of the initially wettest sample has increased from 16 to 29.3%. For the second processing stage, the piston temperature

is heated to 55.5 °C. An additional flow of filtrate (100.6 g) is observed for the initially wettest sample, increasing the dry solid content of the cake to 47.86% at the end of the process. For the initially driest alfalfa sample, 190.6 g of green jus is extracted during this thermally assisted compression stage, increasing the dry solid content of the cake to 45.79%. Thus, after two and a half hours of processing, the total mass of recovered filtrate is lower for the initially driest alfalfa, inducing a measurement dispersion on the final dry solid of the cake. The deviation in the dry solid content of the fresh alfalfa induces a standard deviation of $\pm 1.02\%$ on the final dry solid content of the dewatered sample.

4.2. Analysis of the CCC design

The standardized Pareto chart, plotted in Fig. 7, displays the effects in decreasing order of significance. The length of the horizontal bar is proportional to the value of the t statistic, t_i , of the corresponding effect. The t statistic, t_i , is equal to the absolute value of the estimated effect, $|a_i|$, divided by the standard error on the effect, σ_i . The vertical line is used to identify which effects are statistically significant. Its position is fixed using Student table [33], knowing n the degrees of freedom, i.e. the number of experiments carried out minus the number of effects (i.e. the number of coefficients in the response function), and p the value of the risk. With $n = 3$ and $p = 0,05$, the Student table gives $t_{crit} = 3.182$. Any bars beyond this vertical line are statistically significant at the selected significance level, set in the present analysis at 5%. Thus, the temperature is the only significant main effect and there is no significant interaction effect.

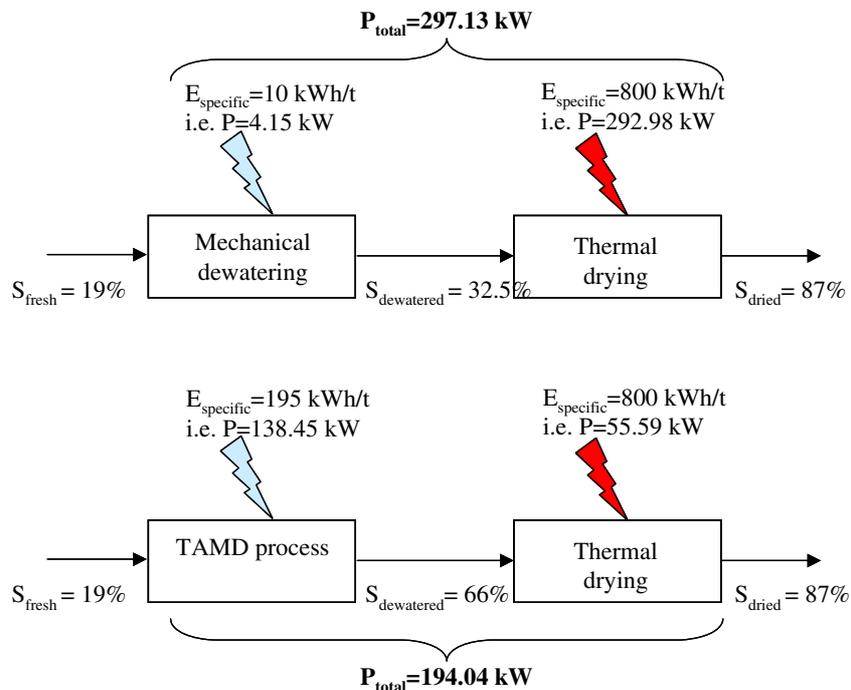


Fig. 13 Reference and alternative scenarii for the energy balance on the global separation chain (S = solid content). Calculations carried out on the basis of 1 ton/h of fresh alfalfa.

This was confirmed by the ANOVA table (see Table 3), which tests the statistical significance of each effect by comparing the mean square with an estimate of the experimental error. This ratio, called the F ratio, is used to compute the p value. Small p value (here less than 0.05 since we operate at a 5% significance level) indicates that the hypothesis of equal means between two distributions is false and thus that the effect is significant. The temperature is the only effect which has a probability lower than 0.05 and, thus, is significant at the 95% confidence level. At this stage of the analysis, the first two objectives of the present study are achieved. Significant separation enhancement is noted and it results only from thermal effects.

The main effect plot (see Fig. 8) shows how each factor affects the final dry solid content of the sample. It gives the overall effect of a variable on the response by setting all other factors at midpoint levels. Clearly, the temperature has a bigger impact on the response than the pressure: the main effect of temperature is 18.3 while the main effect of pressure is only 7.3.

The interaction plot between temperature and pressure is given on Fig. 9. Parallel lines mean that the effect of the first variable, T, is independent of the second variable, P, implying little or no interaction.

4.3. Empirical model

Using the data in Table 4, we can empirically model the final dry solid content (S) as a function of the two variables T and P. The equation of the fitted model is:

$$S = 11.4126 + 1.1345 T + 0.0418(P/100) - 0.0044 T(P/100) + 0.0062 T^2 + 0.0173(P/100)^2 \quad (5)$$

with T in °C and P in kPa.

The model R^2 , namely the amount of S variation explained by the model, is 95.68%. After adjusting for the number of model parameters, the adjusted R^2 is 88.47%.

Fig. 10 shows the predicted dry solid contents plotted versus the observed values: all data points lie close to the 45° dash line, implying that the model provides an adequate approximation to the experimental data.

The normal distribution of the dry solid content distribution can be verified using the residual plot in Fig. 11. Fig. 11 shows the residual, i.e. the difference between the value predicted by equation (5) and the observed value, versus the predicted dry solid content. Normality is satisfied and the empirical model is adequate. Moreover, the standard deviation of estimate is 3.64%.

4.4. Optimal process settings

The parametric directions to obtain the highest dry solid content can be found using this empirical model. The response surface of the final dry solids content is displayed on Fig. 12. The height of the surface represents the predicted value of the final dry solid content over the space of temperature and pressure. Highest dry solid contents are obtained at high temperature and high pressure. To maximize Y with the

minimum temperature and the minimum pressure, the optimum point is 80.71 °C and $P = 2993.5$ kPa.

Considering a thermal energy consumption of the TAMD process of 195 kWh/t of filtrate for a processing temperature of 80 °C [15], a final dry solid content of the press cake of 66% after mechanical dewatering and of 87% after thermal drying, the use of the TAMD process before a thermal drying leads to an energy saving of at least 34.7% on the overall separation chain (see Fig. 13). These calculations were performed on the basis of 1 ton/h of moist alfalfa, using the dryer energy consumption (800 kWh/t) estimated by the French Environment and Energy Management Agency on industrial rotary dryer used to proceed alfalfa [37].

If the green juice recovered during the mechanical dewatering has to be beneficially used, less severe temperature conditions will be necessary to preserve its quality, especially its content of proteins. At 55.5 °C, a 33.76% increase of the dry solids content originally present is expected for a 300 kPa applied pressure. If a second expression stage is performed at higher pressure, for instance 3000 kPa, an additional profit of almost 10% will be registered. On the same design assumptions, an energy savings of 47% is achieved on the overall separation chain, considering a thermal energy consumption of the TAMD process of 135 kWh/t of filtrate for this processing temperature.

5. Conclusion

The TAMD was used to effectively dewater alfalfa biomass under a variety of processing conditions ranging from 21 to 90 °C and from 300 to 3000 kPa. It has been illustrated that the TAMD can be used to remove up to 83% of the inherent water from alfalfa under moderate processing conditions, the enhancement being less than 43% in a conventional mechanical dewatering process. Response surface methodology has been used to estimate the interaction and even the quadratic effects of the processing temperature and the processing pressure and to find optimal process settings to increase the final dry solids content of the press cake. A circumscribed central composite design in 9 runs (and one replicate) was implemented. Its analysis emphasizes that temperature is the only significant main effect at the 95% confidence level and that there are no significant interaction effects. A quadratic equation was used to fit the experimental data and the adequacy of the model was checked considering the multiple determination coefficient, R^2 , and F ratio. After adjusting for the number of model parameters, the adjusted R^2 is 88.47%. The adequacy established, the second order equation can be used as a predictive model. The parametric directions to obtain the highest final dry solid content were found using this empirical model. The highest dry solid contents are obtained at high temperature. Finally, an energy balance was performed on a separation process including a thermal drying of the dewatered press cake up to 87% dry solid content. The reduction of the liquid content in the alfalfa press cake, with a low energy consumption, leads to an energy saving of at least 35% on the overall separation chain.

Recent preliminary experiments emphasize that the potential exists to adapt this technology to a large range of

biomass materials. Current research concerns the process integration using Pinch analysis as well as understanding of the mechanisms involved in the thermally assisted mechanical dewatering, which is essential for the filter design and for the identification of the potential market of the technology.

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