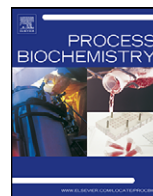




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Short communication

## Modeling small-scale cassava starch extraction. Simulation of the reduction of water consumption through a recycling process

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This paper is dedicated in memoriam to our friend Dr. Claude Marouzé.

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### ABSTRACT

The purpose of this study was to model the extraction unit operation of the cassava starch manufacturing process and to propose a realistic recycling simulation in order to reduce the volumes of effluents. The model was developed from reactors which are commonly used for cassava starch extraction at a household scale in Vietnam. The reactors were tested using inflow starch as a marker at the beginning of the batch process. The experimental residence time distribution (RTD<sub>exp</sub>) was calculated by the outflow of the starch concentration. Using Matlab®, the RTD<sub>exp</sub> was compared to the theoretical residence time distribution (RTD<sub>th</sub>). The dynamic model obtained was built up on Simulink® and tested with four different strategies of recycling methods. Sedimented starch was collected from the different types of processes; the pH value and the titratable acidity of starch were then measured. The results showed a good correlation between RTD<sub>exp</sub> and RTD<sub>th</sub>. The reactors were described by a model of two mixed tanks in series. The simulation of the recycling process revealed a reduction in quantity of water used up to 43%; however, the recycling process increased significantly the titratable acidity of starch up to 6.48 ± 0.11 mequiv. H<sup>+</sup>/100 g dry matter.

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### 1. Introduction

Cassava (*Manihot esculenta* Crantz) is a major source of starch used in most of the tropical regions from Latin America, Africa and Asia [1,2]. Thus, there is global diversity in cassava starch manufacturing technologies, which result in different performances for extracting starch from the roots. The processing yield range from 17% in Ivory Coast, 21% in Brazil and up to 25% in Thailand [3–5]. In South-East Asia, the manufacturing process in large-scale industries is characterized by streams that vary in complexity and require extensive processing to achieve high end-product quality with an optimal consumption of water [6]. In the case of cassava starch manufacturing at small-scale, the process is usually conducted non-

continuously either through manual or mechanized unit operations [7], in which a volume of up to 22 m<sup>3</sup> of water is required for extracting 1 ton of starch [8]. Additionally, none of the small-scale agro industries in Vietnam use recycling and wastewater treatment devices; and, large quantities of liquid wastes are usually discharged into the environment [9]. Several authors proposed some optimizations of the manufacturing process at a small-scale, in which improvements concern either the extraction yield [10] or more rapid separation of starch from fruit water by adding hydrocyclones [11]. Nevertheless, starch agro industries rarely adopt these technologies, which require large space and great investments. Therefore, the optimization of the extraction unit operation (EUO) for preventing the management of large liquid waste quantities remains a challenge for small agro industries [12,13].

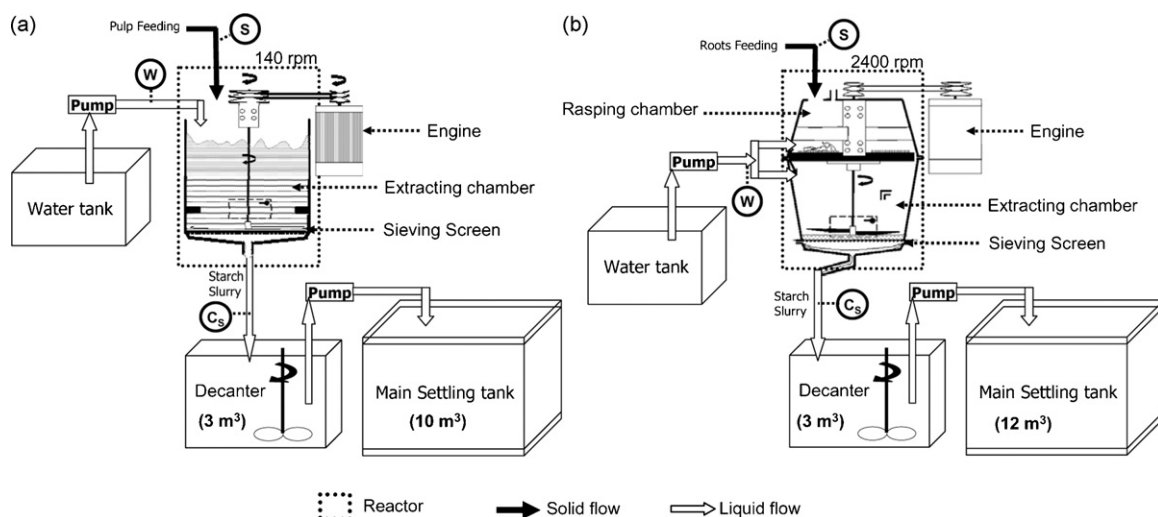
Moreover, building up an experiment is usually more accessible at lab-scale than at pilot or industrial-scale, like in agro industries, in which the production rate can reach a few hundred kilograms per hour. In such case, the amount of raw material required per extractor may be too large to build up a realistic optimization trial of the manufacturing process. This article aims at extending the residence time distribution, a classical concept in food engineering [14,15], to cassava starch extraction at household scale. The novelty of our work is not only to diagnose the functioning of the reactors,

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**Fig. 1.** Cassava starch extraction flow sheet for two types of vertical stirred-tank reactors. Single vertical stirred-tank reactor (a) and rasper/extractor stirred-tank reactor (b). The round symbols marked 'S', 'W' and 'C<sub>s</sub>' correspond to starch inflow, water inflow and to starch concentration, respectively.

but also to simulate the reduction of water consumption through a recycling process. Moreover, the recycling option designed in this study is assessed in a starch production unit from Vietnam, and by considering realistic equipments, capital availabilities, and starch quality.

## 2. Materials and methods

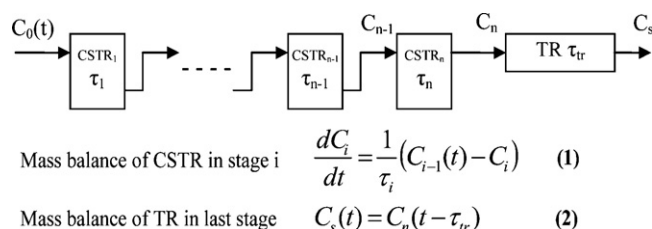
### 2.1. Manufacturing process and equipment

We used the basic equipment for cassava wet starch production at a small-scale. The equipment consisted of a rasing machine to crush the roots, which were previously washed and slightly peeled as described elsewhere [8]; a stirring–filtering tank for screening the cassava pulp; and a settling tank where the extracted starch was separated from its aqueous suspension under gravity. Two different types of reactors were tested in order to study the EUO (Fig. 1). The TB reactor (0.72 m<sup>3</sup>) consisted of a vertical stirring–filtering extractor. The TC reactor, which consisted of a vertical rasing–stirring–filtering reactor, was composed of two superposed cylindrical compartments: a rasing chamber (0.04 m<sup>3</sup>) and a stirring–filtering chamber (0.14 m<sup>3</sup>). The TB and TC extraction stages (TB<sub>EUO</sub>, TC<sub>EUO</sub>) were evaluated at household scale in two different processing units from a root processing cluster located in the red-river Delta in North Vietnam [8].

### 2.2. Modeling starch extraction without recycling

The model is established for the purpose of simulation of the reduction of water consumption, but without reducing the daily production capacities of the processing units. The starch slurry is considered as a binary mixture of water and starch, which is supposed to be chemically inert. We chose modeling the starch concentration dynamics during the EUO rather than other physical phenomena of the process behavior that are not directly related to starch extraction and water consumption. The trials were carried out for each type of extractor with a minimum of three batches conducted successively. A batch consisted of following up a set of input and output parameters during the EUO. The input parameters were the average water inflow ( $Q_w$ ) and the starch inflow ( $F_{s-r}$ ). The water inflow rate was obtained by reporting time and water consumption, measured by water-meters (Fig. 1).  $Q_w$  was considered to be constant for non-recycling process. The moisture content and starch content of raw material were measured by the enzymatic colorimetric method described previously [16].  $F_{s-r}$  was obtained by weighing washed and slightly peeled roots or pulp and reporting the feeding time with a given quantity of raw material. The output parameters were the extraction time and the  $RTD_{exp}$ , which were obtained from the starch concentration dynamics measured by specific gravity in g/dm<sup>3</sup> and expressed in g/l as functions of times. For non-recycling process, we supposed that each reactor initially contained pure water, so that the concentrations of starch in the vessels were zero. We considered the  $RTD_{th}$  related to mixed tanks using impulse and square pulse inputs as starch tracers. The mass balance of starch in each tank, accounting for the starch that enters, leaves and accumulates, led to the dynamic model shown in Fig. 2, in which the differential equations (1) and (2) describe a cascade of  $n + 1$  reactors.

The  $RTD_{exp}$  and the  $RTD_{th}$  were compared using unconstrained nonlinear minimization [17] with Matlab 7.4 [18]. The calibration and validation of the model consisted of adjusting the theoretical and experimental space times; thus, the



**Fig. 2.** Modeling the cassava starch extraction stage by a cascade of  $n + 1$  reactors. A squared box and the rectangular box correspond to continuous stirred-tank reactor (CSTR) and tubular reactor (TR), respectively. The differential equations describe the dynamic model of  $n + 1$  reactors as indicated in the text.  $C$  and  $\tau$  stand for starch concentration and space time, respectively.

algebraic equations in the complex domain using the Laplace transformation (i.e. transfer-functions) were obtained for each reactor.

### 2.3. Simulation of the recycling process

A diagram of the recycling process was built up on Simulink 6.6 R2007a (The MathWorks Inc., US Patent). The diagram included the transfer-functions of the mixing system. A simulation of recycling consisted of running a series of successive batches. Different variables were monitored after each batch, and gathered at the end of the whole simulation. These variables were the total quantity of extracted starch, the threshold of the end of the batch ( $S_{eb}$ ), the threshold for starting the recycling process ( $S_{recy}$ ) and the volume of the re-used starch milk ( $V_{recy}$ ). Based on a set of parameters, different strategies of recycling methods were applied to the reactor which presented the highest performance. A simulation of a non-recycling process was also tested and then compared to the results obtained with recycling.

### 2.4. Starch quality analysis

#### 2.4.1. Starch content in starch slurry

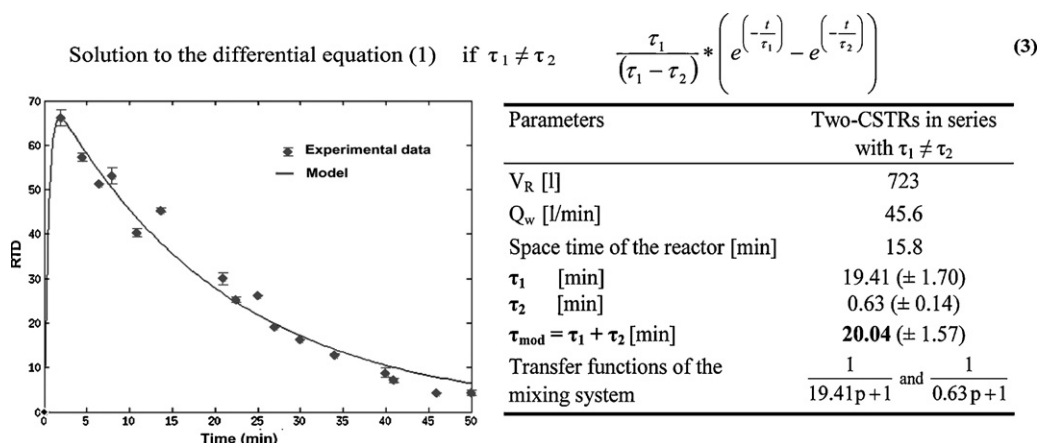
Starch content in the starch slurry was obtained by a density method [8]. Two hydrometers were used (model Dujardin-Salleron) to measure the density of aliquots (500 ml) collected at regular intervals from the outlet of TB and TC extractors during the duration of the extraction process. Wet starch samples were dried (at 50 °C during 48 h) and used to prepare a range of suspensions, in which specific gravity was measured. A standard curve between the starch milk density and starch concentration (g/l) was obtained.

#### 2.4.2. Ash content

Starch ash content was calculated following heating to 550 °C for 3 h as per AOAC official method [19].

#### 2.4.3. Crude fiber content

The fiber content was determined for the loss on ignition of dried residue remaining after digestion of cassava flour (2 g) with 1.25% H<sub>2</sub>SO<sub>4</sub> and 1.25% NaOH as per AOAC official method [20].



**Fig. 3.** Modeling the starch concentration dynamics for a simple vertical stirred-tank reactor. The graph shows the residence time distribution (RTD) as a function of time for experimental data (dots with errors bars) compared to the model curve. Each plot represents an average of triplicates samples with repeated trials with standard deviations.  $\tau$  and  $p$  stand for space time and Laplace complex variable, respectively.

2.4.4. pH and titratable acidity

10 g of dried starch was blended with about 100 ml deionized water (10% w/V) for 30 min using a magnetic stirrer. The pH of the blended solution was determined on the supernatant at ambient temperature after centrifugation. The total acidity was then measured on the supernatant by titration with 0.001 N NaOH to equivalent point (pH 7). The result was calculated in milliequivalent hydrogen ions per 100 g dry matter (mequiv.  $H^+$ /100 g DM).

3. Results and discussion

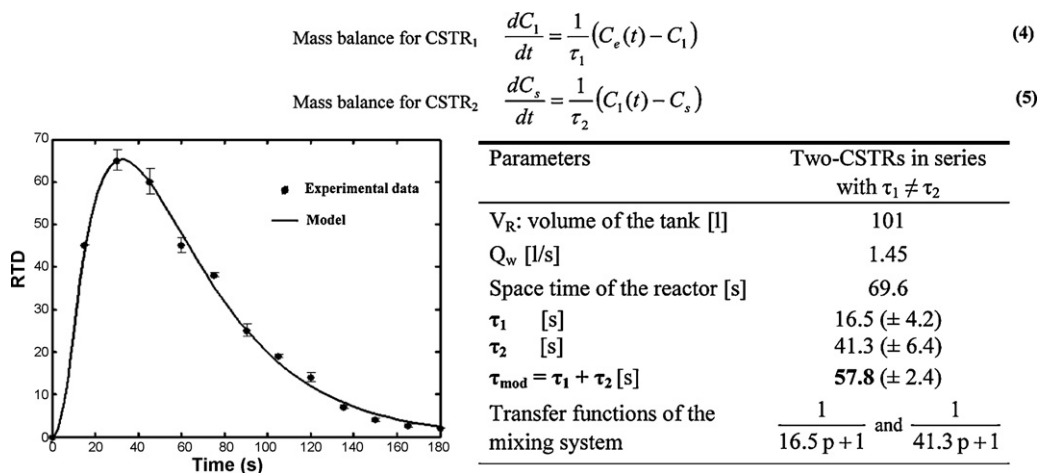
3.1. Modeling starch extraction without recycling

Fig. 3 shows the starch concentration dynamics ( $RTD_{exp}$ ) obtained with TB extractor. The experimental results suggest a simplification of the equations used for modeling the EUO. In the case of TB extractor, the cassava pulp, which contained the starch to extract, was loaded only once before starting stirring; so, starch, which was used as the tracer, was supplied through impulse signal inputs. Therefore, the solution (3) was used on Matlab® for modeling the EUO with TB extractor ( $TB_{EUO}$ ). Fig. 3 also shows that, the model describing  $TB_{EUO}$  as two CSTRs in series provides a good agreement with experimental data. Thus, the results point out a difference between the space time of the reactor ( $V_R/Q_w$ ) and the mean residence time, which indicates the presence of dead zones according to the theories predicting the non ideals

RTDs [21]. This difference might be due to the variations in the water-flow rate, which was subjected to change (within the range 41–50 l) during the 50 min trial in order to maintain  $V_R$  at constant level.

Fig. 4 shows the starch concentration dynamics ( $RTD_{exp}$ ) obtained with TC extractor. The experimental results suggest a simplification of the equations used for modeling the EUO. In fact, the starch concentration profiles indicate that the extractors correspond to a cascade of two CSTRs rather than to a series of CSTRs and tubular reactors. In the case of TC extractor, the roots were loaded continuously during the first 30 s after starting stirring. So, starch, which was used as the tracer, was supplied through a square pulse input. In that case, the differential equations (4) and (5), which depend on two parameters  $\tau_1$  and  $\tau_2$ , are solved with Matlab. Therefore, the program determines the optimal values of  $\tau_1$  and  $\tau_2$  by minimizing the error between the experimental and simulated starch concentrations (with Matlab's fminsearch function).

Fig. 4 also shows that the model, describing  $TC_{EUO}$  as two CSTRs in series, provides a good agreement with the experimental data. In addition, the results are replicable for three batches indicating that the model can be used for a greater number of batches. In this case, the transfer-functions which define the dynamic behavior of the  $TC_{EUO}$  are valid with any type of input. So, we used Simulink to valid this model and to simulate different recycling scenarios.



**Fig. 4.** Modeling residence time distribution for a raspier/extractor stirred-tank reactor. The graph shows the residence time distribution (RTD) as a function of time for experimental data (dots with errors bars) compared to the model curve. Each plot represents an average of triplicates samples with repeated trials with standard deviations.  $\tau$  and  $p$  stand for space time and Laplace complex variable, respectively.

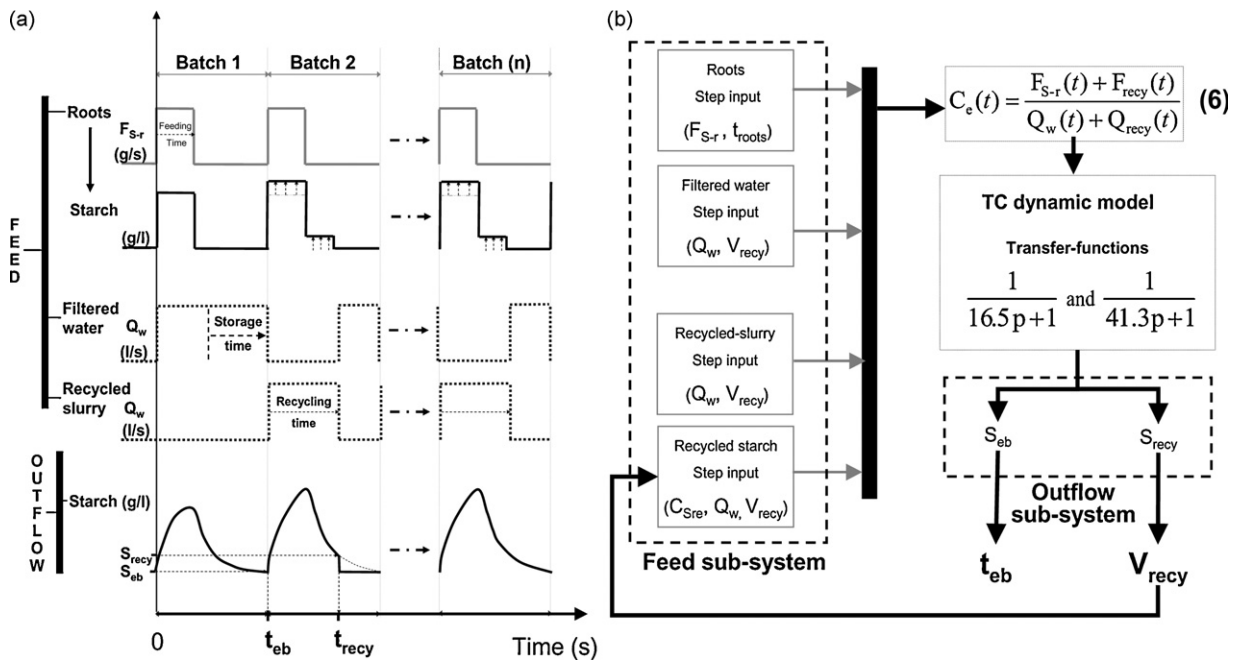


Fig. 5. Implementation of the recycling model assuming the extractor to be a series of continuous stirred-tank reactors. Diagram of the process parameters, namely feed rate and starch outflows (a). Algorithm for the recycling model parameter estimation (b). The symbols are described in the list of abbreviations.

### 3.2. Simulation of the recycling process

It was shown previously that TC reactors were largely adopted within the processing units; particularly because rasing-extraction with TC technology resulted in increasing processing efficiency with low quantities of starch in cassava bagasse compared to TB technology [8]. However, the TC technology presents the highest consumption of water per ton of starch. Therefore, the simulation of the recycling was performed only with TC reactor. The recycling consisted of storing a volume of starch slurry at the end of each extraction batch; and in re-using a volume of this slurry for the following batch. Fig. 5 shows the diagram of the recycling simulation, in which the feed and the outputs are represented as functions of time from the first batch (without recycling) to the last batch (with recycling). The diagram also shows the different thresholds  $S_{eb}$  and  $S_{recy}$  which correspond to  $t_{eb}$  and  $t_{recy}$ , respectively. The number of batches run on Simulink (48) was similar to the number of batches performed in a processing unit during 1 day of production.

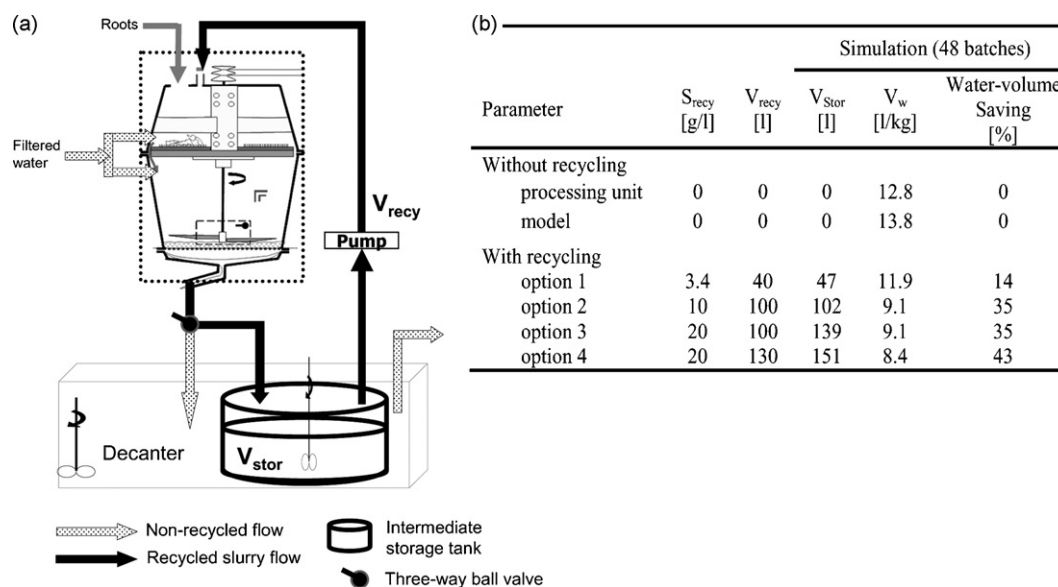
In order to determine the value of  $t_{eb}$ , a first simulation was performed without recycling; then,  $t_{eb}$  was reported in the model and used subsequently for the whole simulation with recycling.  $C_{Sre}$  corresponds to the starch concentration of the starch slurry in the storage tank, in which a given quantity was reused for the following batch. Thus, Eq. (6) was added to the design to express the relation between the inflow starch concentration  $C_e(t)$  and the different parameters in the feed during the recycling process.

Fig. 6 presents the results of the simulation performed with TC on Simulink. For non-recycling process, the water-use values ( $V_w$ ) between the model and the real extractor in the processing unit were comparable. For the four recycling simulations, in which low-cost apparatus was included (a three-way ball valve, an intermediate tank, a pump, and pipes) and different weights were assigned to parameters  $S_{recy}$  and  $V_{recy}$ ; the results show that  $V_w$  was in the range of 8.4–11.9 l/kg which corresponds to a saving of filtered water up to 43%. Thus,  $S_{recy}$  and  $V_{recy}$  were maintained constant for each simulation in order to limit extra-labor work. However, high values assigned to  $S_{recy}$  and  $V_{recy}$  were not con-

sidered (Fig. 6); indeed, for values higher than 20 g/l and 130 l, respectively, the processors may face overwhelming difficulties in investing in new equipments (for transporting high-concentrated slurry) and in storing and recycling larger volume of liquid (which requires an expansion of the processing unit). Some studies showed that the consumption and the volume of effluent from a factory could be reduced by 50% when a single hydrocyclone was used. Nevertheless, the hydrocyclone technology requires space and investments, and was tested only at pilot scale [22,23]. In addition, high level of impurities in final starch may occur when concentrating the starch slurry [4]. Consequently, new trials might be conducted in our study in order to adapt and to complete the current optimization, which thankfully requires limited space and low-cost investments.

### 3.3. Starch quality

The starch composition did not show significant differences between the process without recycling and the process with recycling, neither in crude fibers ( $0.88 \pm 0.1\%$ ), nor in ash content ( $0.29 \pm 0.0\%$ ). The great variation coefficients within the starch component indicated that the determination of the starch composition was not sufficient to characterize the differences between starches collected from non-recycled and recycled processes, like previously noted in Thailand or in Colombia [4,24]. However, the pH value of the two types of starches collected from non-recycled and recycled processes were indicatively different with  $5.1 \pm 0.00$  and  $4.2 \pm 0.06$ , respectively. Significant differences were also observed between these two types, in titratable acidity with  $1.18 \pm 0.04$  and  $6.48 \pm 0.11$  mequiv.  $H^+$ /100 g, respectively. These results are in accordance with a previous study conducted in Thailand; in which no great significant change was observed in composition of lactic acid fermented starch; however, the authors noted that the acidification caused a modification in pasting and thermal properties of starch within a very short period of time [25]. Further investigations on granule shape [26] and cyanogenic compounds [6] would help to better understand the acid factor parameter when water is used in low quantity.



**Fig. 6.** Recycling process with the rasper/extractor stirred-tank reactor. Process flow diagram with apparatus (a), and results of the simulation of the recycling performed on 48 batches (b).  $S_{recy}$ : threshold concentration of starch indicating the beginning of the recycling process;  $V_{recy}$ : volume of recycled-slurry;  $V_{stor}$ : volume of slurry collected in the intermediate storage tank;  $V_w$ : volume of filtered water consumed per quantity of fresh roots.

#### 4. Conclusion

The residence time distribution (RTD) methodology was extended to cassava starch extraction at household scale. The RTDs in two different cassava starch extractors from Vietnam (TB and TC) were measured by tracing the liquid outflow with starch. The dynamic model obtained was used to simulate a recycling process performed with TC extractor and allowed a saving of 43% water without having to majorly modify the equipment. The model proposed in this study can be used in other locations where information is needed for facing water use reduction challenge. However, with recycling technologies, small-scale processors may face new challenges in order to reach starch quality standards required by end-users.

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#### Appendix A. Nomenclature

$C(t)$	concentration of starch as a function of time (g/l)
$C_e(t)$	concentration of starch contained in feed material (g/l)
$C_{Sre}$	concentration of starch contained in recycled-slurry (g/l)
CSTR	continuous stirred-tank reactor
EUO	extraction unit operation
$F_{S-r}(t)$	inflow of starch released from washed and slightly peeled roots (g/s)
$F_{recy}(t)$	inflow of starch contained in recycled-slurry (g/s)
$p$	Laplace complex variable
$Q_{recy}$	average inflow of recycled-slurry (l/s)
$Q_w$	average inflow of filtered water (l/s)
RTD <sub>exp</sub>	experimental residence time distribution

RTD <sub>th</sub>	theoretical residence time distribution
$S_{eb}$	threshold concentration of starch indicating the end of the batch (g/l)
$S_{recy}$	threshold concentration of starch indicating the beginning of the recycling process (g/l)
$\tau$	space time
$\tau_{tr}$	space time for tubular reactor
TB	type B
TC	type C
$t_{eb}$	time indicating the end of a batch (s)
$t_{recy}$	time before switching valve for storing the slurry (s)
$t_{roots}$	time to feed the extractor with slightly peeled -roots (s)
TR	tubular reactor
$V_R$	volume of the stirred-tank (l)
$V_{recy}$	volume of recycled-slurry (l)
$V_{stor}$	volume of slurry collected in the intermediate storage tank (l)
$V_w$	volume of filtered water consumed per quantity of fresh roots (l/kg)

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