

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

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² This paper is dedicated in memoriam to our friend Dr. Claude Marouzé.

22 Abstract

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

37

38 Keywords :

39 Modeling, Starch, Extraction, Water-consumption, Simulation, Recycling-process.

40 Introduction

41 Cassava (*Manihot esculenta* Crantz) is a major source of starch used in most of the tropical regions from
42 Latin America, Africa and Asia [1, 2]. Thus, there is global diversity in cassava starch manufacturing
43 technologies, which result in different performances for extracting starch from the roots. The processing
44 yield range from 17% in Ivory Coast, 21% in Brazil and up to 25% in Thailand [3-5]. In South-East Asia, the
45 manufacturing process in large-scale industries is characterized by streams that vary in complexity and
46 require extensive processing to achieve high end-product quality with an optimal consumption of water [6].
47 In the case of cassava starch manufacturing at small-scale, the process is usually conducted non-continuously
48 either through manual or mechanized unit operations [7], in which a volume of up to 22 m³ of water is
49 required for extracting one ton of starch [8]. Additionally, none of the small-scale agro industries in Vietnam
50 use recycling and waste water treatment devices; and, large quantities of liquid wastes are usually discharged
51 into the environment [9]. Several authors proposed some optimizations of the manufacturing process at a
52 small-scale, in which improvements concern either the extraction yield [10] or more rapid separation of
53 starch from fruit water by adding hydrocyclones [11]. Nevertheless, starch agro industries rarely adopt these
54 technologies, which require large space and great investments. Therefore, the optimization of the extraction
55 unit operation (EUO) for preventing the management of large liquid waste quantities remains a challenge for
56 small agro industries [12, 13].

57 Moreover, building up an experiment is usually more accessible at lab-scale than at pilot or industrial-scale,
58 like in agro industries, in which the production rate can reach a few hundred kilograms per hour. In such
59 case, the amount of raw material required per extractor may be too large to build up a realistic optimization
60 trial of the manufacturing process. This article aims at extending the residence time distribution, a classical
61 concept in food engineering [14, 15], to cassava starch extraction at household scale. The novelty of our
62 work is not only to diagnose the functioning of the reactors, but also to simulate the reduction of water
63 consumption through a recycling process. Moreover, the recycling option designed in this study is assessed
64 in a starch production unit from Vietnam, and by considering realistic equipments, capital availabilities, and
65 starch quality.

66

67 Materials and methods

68

69 Manufacturing process and equipment

70 We used the basic equipment for cassava wet starch production at a small-scale. The equipment consisted of
71 a rasping machine to crush the roots, which were previously washed and slightly peeled as described
72 elsewhere [8]; a stirring-filtering tank for screening the cassava pulp; and a settling tank where the extracted
73 starch was separated from its aqueous suspension under gravity. Two different types of reactors were tested
74 in order to study the EUO (figure 1). The TB reactor (0.72 m^3) consisted of a vertical stirring-filtering
75 extractor. The TC reactor, which consisted of a vertical rasping-stirring-filtering reactor, was composed of
76 two superposed cylindrical compartments : a rasping chamber (0.04 m^3) and a stirring-filtering chamber
77 (0.14 m^3). The TB and TC extraction stages (TB_{EUO} , TC_{EUO}) were evaluated at household-scale in two
78 different processing units from a root processing cluster located in the red-river Delta in north Vietnam [8].
79

80 Modeling starch extraction without recycling

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

98 dynamic model shown on figure 2, in which the differential equations (1) and (2) describe a cascade of $n+1$ -
99 reactors.

100 The RTD_{exp} and the RTD_{th} were compared using unconstrained nonlinear minimization [17] with Matlab 7.4
101 [18]. The calibration and validation of the model consisted of adjusting the theoretical and experimental
102 space times; thus, the algebraic equations in the complex domain using the Laplace transformation (i.e.
103 transfer-functions) were obtained for each reactor.

104

105 Simulation of the recycling process

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

114

115 Starch quality analysis

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

122 *Ash Content*: Starch ash content was calculated following heating to 550 °C for 3 h as per AOAC official
123 method [19].

124 *Crude fiber content*: The fiber content was determined for the loss on ignition of dried residue remaining
125 after digestion of cassava flour (2 g) with 1.25% H₂SO₄ and 1.25% NaOH as per AOAC official method
126 [20].

127 *pH and titratable acidity*: 10 g of dried starch was blended with about 100 ml deionized water (10% w/V) for
128 30 minutes using a magnetic stirrer. The pH of the blended solution was determined on the supernatant at
129 ambient temperature after centrifugation. The total acidity was then measured on the supernatant by titration
130 with 0.001 N NaOH to equivalent point (pH 7). The result was calculated in milliequivalent hydrogen ions
131 per 100 g dry matter (mEq H⁺/100 g DM).

132

133 Results and Discussion

134

135 Modeling starch extraction without recycling

136 Figure 3 shows the starch concentration dynamics (RTD_{exp}) obtained with TB extractor. The experimental
137 results suggest a simplification of the equations used for modeling the EUO. In the case of TB extractor, the
138 cassava pulp, which contained the starch to extract, was loaded only once before starting stirring; so, starch,
139 which was used as the tracer, was supplied through impulse signal inputs. Therefore, the solution (3) was
140 used on Matlab[®] for modeling the EUO with TB extractor (TB_{EUO}). Figure 3 also shows that, the model
141 describing TB_{EUO} as two-CSTRs in series provides a good agreement with experimental data. Thus, the
142 results point out a difference between the space time of the reactor (V_R/Q_w) and the mean residence time,
143 which indicates the presence of dead zones according to the theories predicting the non ideals RTDs [21].
144 This difference might be due to the variations in the water-flow rate, which was subjected to change (within
145 the range 41-50 l) during the 50 min trial in order to maintain V_R at constant level.

146 Figure 4 shows the starch concentration dynamics (RTD_{exp}) obtained with TC extractor. The experimental
147 results suggest a simplification of the equations used for modeling the EUO. In fact, the starch concentration
148 profiles indicate that the extractors correspond to a cascade of two CSTRs rather than to a series of CSTRs
149 and tubular reactors. In the case of TC extractor, the roots were loaded continuously during the first 30 s after
150 starting stirring. So, starch, which was used as the tracer, was supplied through a square pulse input. In that
151 case, the differential equations (4) and (5), which depend on two parameters τ_1 and τ_2 , are solved with
152 Matlab. Therefore, the program determines the optimal values of τ_1 and τ_2 by minimizing the error between
153 the experimental and simulated starch concentrations (with Matlab's `fminsearch` function).

154 Figure 4 also shows that the model, describing TC_{EUO} as two-CSTRs in series, provides a good agreement
155 with the experimental data. In addition, the results are replicable for three batches indicating that the model

156 can be used for a greater number of batches. In this case, the transfer-functions which define the dynamic
157 behavior of the TC_{EUO} are valid with any type of input. So, we used Simulink to valid this model and to
158 simulate different recycling scenarios.

159

160 Simulation of the recycling process

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

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

176 Figure 6 presents the results of the simulation performed with TC on Simulink. For non-recycling process,
177 the water-use values (V_w) between the model and the real extractor in the processing unit were comparable.
178 For the four recycling simulations, in which low-cost apparatus was included (a three-way ball valve, an
179 intermediate tank, a pump, and pipes) and different weights were assigned to parameters S_{recy} and V_{recy} ; the
180 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
181 43%. Thus, S_{recy} and V_{recy} were maintained constant for each simulation in order to limit extra-labor work.
182 However, high values assigned to S_{recy} and V_{recy} were not considered (figure 6); indeed, for values higher
183 than 20 g/l and 130 l respectively, the processors may face overwhelming difficulties in investing in new
184 equipments (for transporting high-concentrated slurry) and in storing and recycling larger volume of liquid

185 (which requires an expansion of the processing unit). Some studies showed that the consumption and the
186 volume of effluent from a factory could be reduced by 50% when a single hydrocyclone was used.
187 Nevertheless, the hydrocyclone technology requires space and investments, and was tested only at pilot scale
188 [22, 23]. In addition, high level of impurities in final starch may occur when concentrating the starch slurry
189 [4]. Consequently, new trials might be conducted in our study in order to adapt and to complete the current
190 optimization, which thankfully requires limited space and low-cost investments.

191

192 Starch quality

193 The starch composition did not show significant differences between the process without recycling and the
194 process with recycling, neither in crude fibers ($0.88\% \pm 0.1$), nor in ash content ($0.29\% \pm 0.0$). The great
195 variation coefficients within the starch component indicated that the determination of the starch composition
196 was not sufficient to characterize the differences between starches collected from non-recycled and recycled
197 processes, like previously noted in Thailand or in Colombia [4, 24]. However, the pH value of the two types
198 of starches collected from non-recycled and recycled processes were indicatively different with 5.1 ± 0.00 and
199 4.2 ± 0.06 respectively. Significant differences were also observed between these two types, in titratable
200 acidity with 1.18 ± 0.04 and 6.48 ± 0.11 mEq H⁺/100 g respectively. These results are in accordance with a
201 previous study conducted in Thailand; in which no great significant change was observed in composition of
202 lactic acid fermented starch; however, the authors noted that the acidification caused a modification in
203 pasting and thermal properties of starch within a very short period of time [25]. Further investigations on
204 granule shape [26] and cyanogenic compounds [6] would help to better understand the acid factor parameter
205 when water is used in low quantity.

206

207 Conclusion

208 The residence time distribution (RTD) methodology was extended to cassava starch extraction at household
209 scale. The RTDs in two different cassava starch extractors from Vietnam (TB and TC) were measured by
210 tracing the liquid outflow with starch. The dynamic model obtained was used to simulate a recycling process
211 performed with TC extractor and allowed a saving of 43% water without having to majorly modify the
212 equipment. The model proposed in this study can be used in other locations where information is needed for

213 facing water use reduction challenge. However, with recycling technologies, small scale processors may face
214 new challenges in order to reach starch quality standards required by end-users.

215

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221

222 Appendix A. Abbreviations and units

223 $C(t)$ starch concentration as a function of time (g/l)
224 $C_e(t)$ concentration of starch contained in feed material (g/l)
225 C_{Sre} concentration of starch contained in recycled-slurry (g/l)
226 CSTR continuous stirred-tank reactor
227 EUO extraction unit operation
228 $F_{s-r}(t)$ inflow of starch released from washed and slightly peeled roots (g/s)
229 $F_{recy}(t)$ inflow of starch contained in recycled-slurry (g/s)
230 p Laplace complex variable
231 Q_{recy} average inflow of recycled-slurry (l/s)
232 Q_w average inflow of filtered-water (l/s)
233 RTD_{exp} experimental residence time distribution
234 RTD_{th} theoretical residence time distribution
235 S_{eb} threshold concentration of starch indicating the end of the batch (g/l)
236 S_{recy} threshold concentration of starch indicating the beginning of the recycling process (g/l)
237 τ space time
238 τ_{tr} space time for tubular reactor
239 TB type B
240 TC type C
241 t_{eb} time indicating the end of a batch (s)

242	t_{recy}	time before switching valve for storing the slurry (s)
243	t_{roots}	time to feed the extractor with slightly peeled -roots (s)
244	TR	Tubular reactor
245	V_R	volume of the stirred-tank (l)
246	V_{recy}	volume of recycled-slurry (l)
247	V_{Stor}	volume of slurry collected in the intermediate storage tank (l)
248	V_w	volume of filtered-water consumed per quantity of fresh roots (l/kg)

249

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307 starch granule structure - function properties: influence of time and conditions at harvest on four cultivars of
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309

310

311

312 **Legends**

313 Figure 1. Cassava starch extraction flow sheet for two types of vertical stirred-tank reactors. Single vertical
314 stirred-tank reactor (a) and rasper/extractor stirred-tank reactor (b). The round symbols marked ‘S’, ‘W’ and
315 ‘C_s’ correspond to starch inflow, water inflow and to starch concentration respectively.

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

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

324 Figure 4. Modeling residence time distribution for a rasper/extractor stirred-tank reactor. The graph shows
325 the residence time distribution (RTD) as a function of time for experimental data (dots with errors bars)
326 compared to the model curve. Each plot represents an average of triplicates samples with repeated trials with
327 standard deviations. τ and p stand for space-time and Laplace complex variable respectively.

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

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

336

Figure 1
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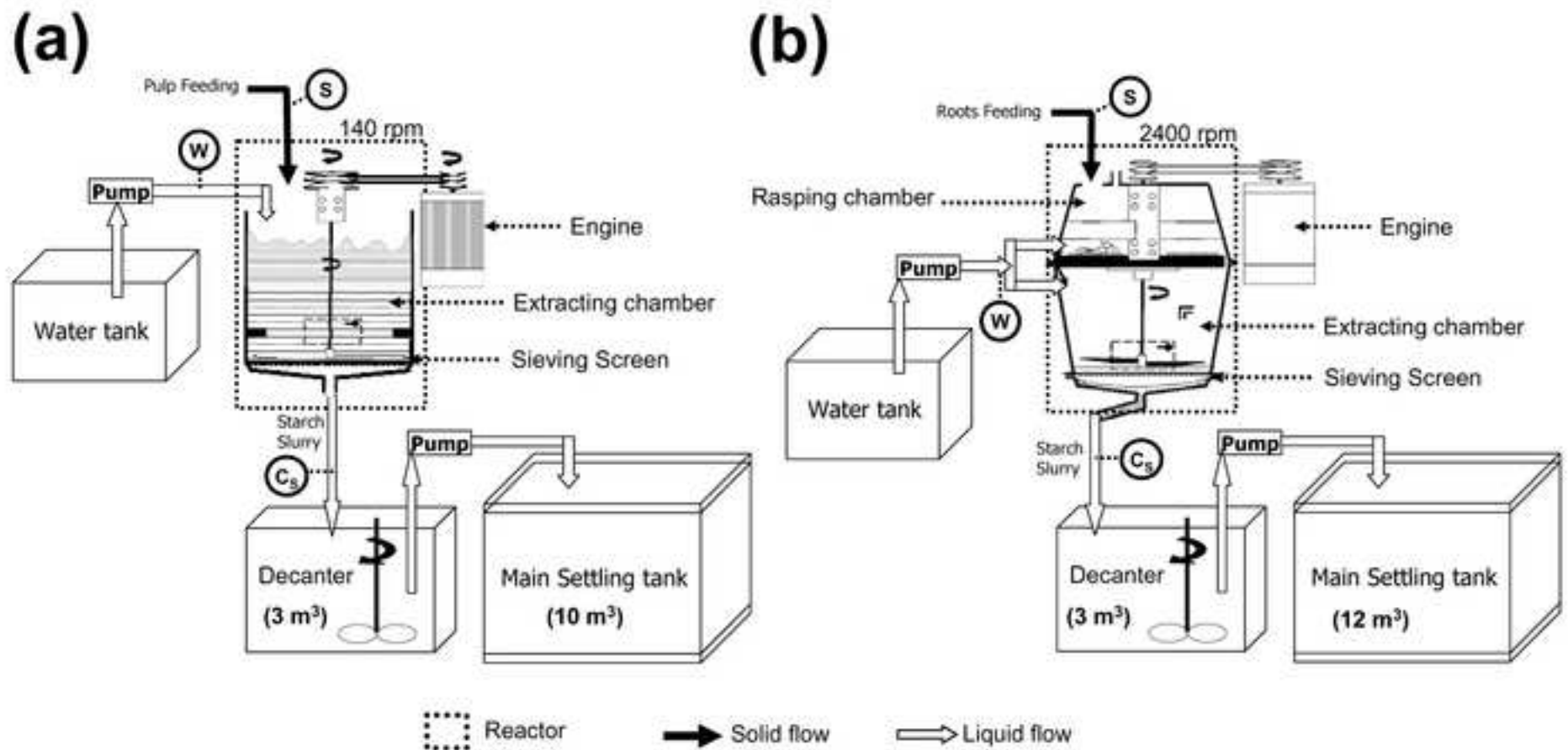
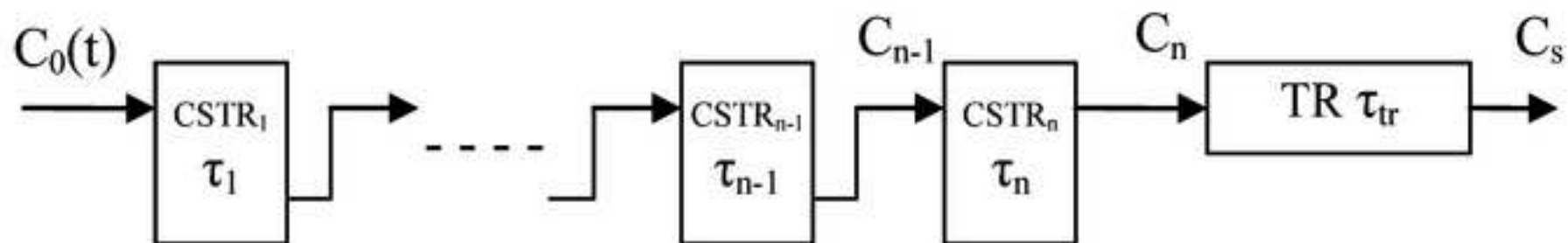


Figure 2
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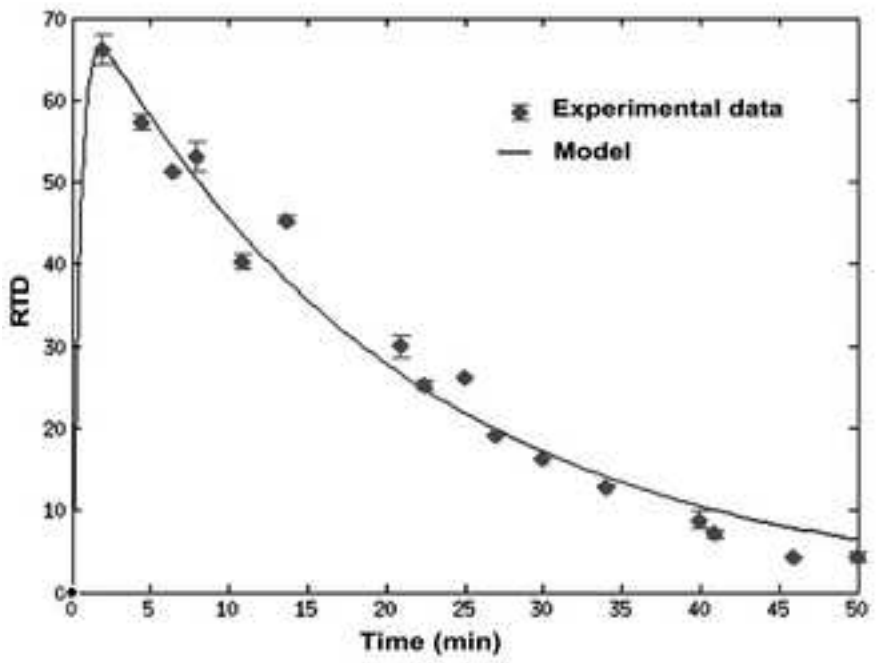
Mass balance of CSTR in stage i
$$\frac{dC_i}{dt} = \frac{1}{\tau_i} (C_{i-1}(t) - C_i) \quad (1)$$

Mass balance of TR in last stage
$$C_s(t) = C_n(t - \tau_{tr}) \quad (2)$$

Figure 3
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Solution to the differential equation (1) if $\tau_1 \neq \tau_2$

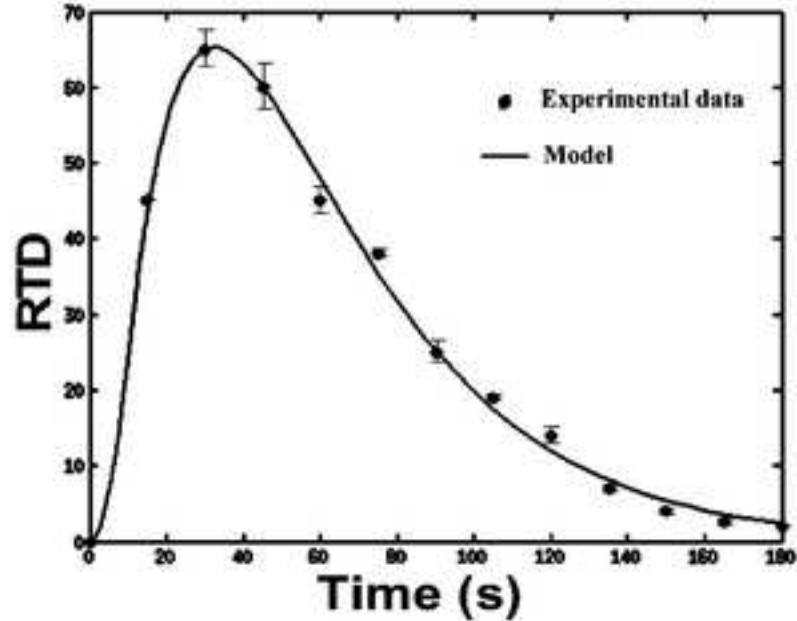
$$\frac{\tau_1}{(\tau_1 - \tau_2)} * \left(e^{\left(-\frac{t}{\tau_1}\right)} - e^{\left(-\frac{t}{\tau_2}\right)} \right) \quad (3)$$



Parameters	Two-CSTRs in series with $\tau_1 \neq \tau_2$
V_R [l]	723
Q_w [l/min]	45.6
Space time of the reactor [min]	15.8
τ_1 [min]	19.41 (± 1.70)
τ_2 [min]	0.63 (± 0.14)
$\tau_{mod} = \tau_1 + \tau_2$ [min]	20.04 (± 1.57)
Transfer functions of the mixing system	$\frac{1}{19.41p+1}$ and $\frac{1}{0.63p+1}$

Figure 4

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$$\text{Mass balance for CSTR}_1 \quad \frac{dC_1}{dt} = \frac{1}{\tau_1} (C_e(t) - C_1) \quad (4)$$

$$\text{Mass balance for CSTR}_2 \quad \frac{dC_s}{dt} = \frac{1}{\tau_2} (C_1(t) - C_s) \quad (5)$$

Parameters	Two-CSTRs in series with $\tau_1 \neq \tau_2$
V_R : volume of the tank [l]	101
Q_w [l/s]	1.45
Space time of the reactor [s]	69.6
τ_1 [s]	16.5 (\pm 4.2)
τ_2 [s]	41.3 (\pm 6.4)
$\tau_{\text{mod}} = \tau_1 + \tau_2$ [s]	57.8 (\pm 2.4)
Transfer functions of the mixing system	$\frac{1}{16.5p+1}$ and $\frac{1}{41.3p+1}$

Figure 5
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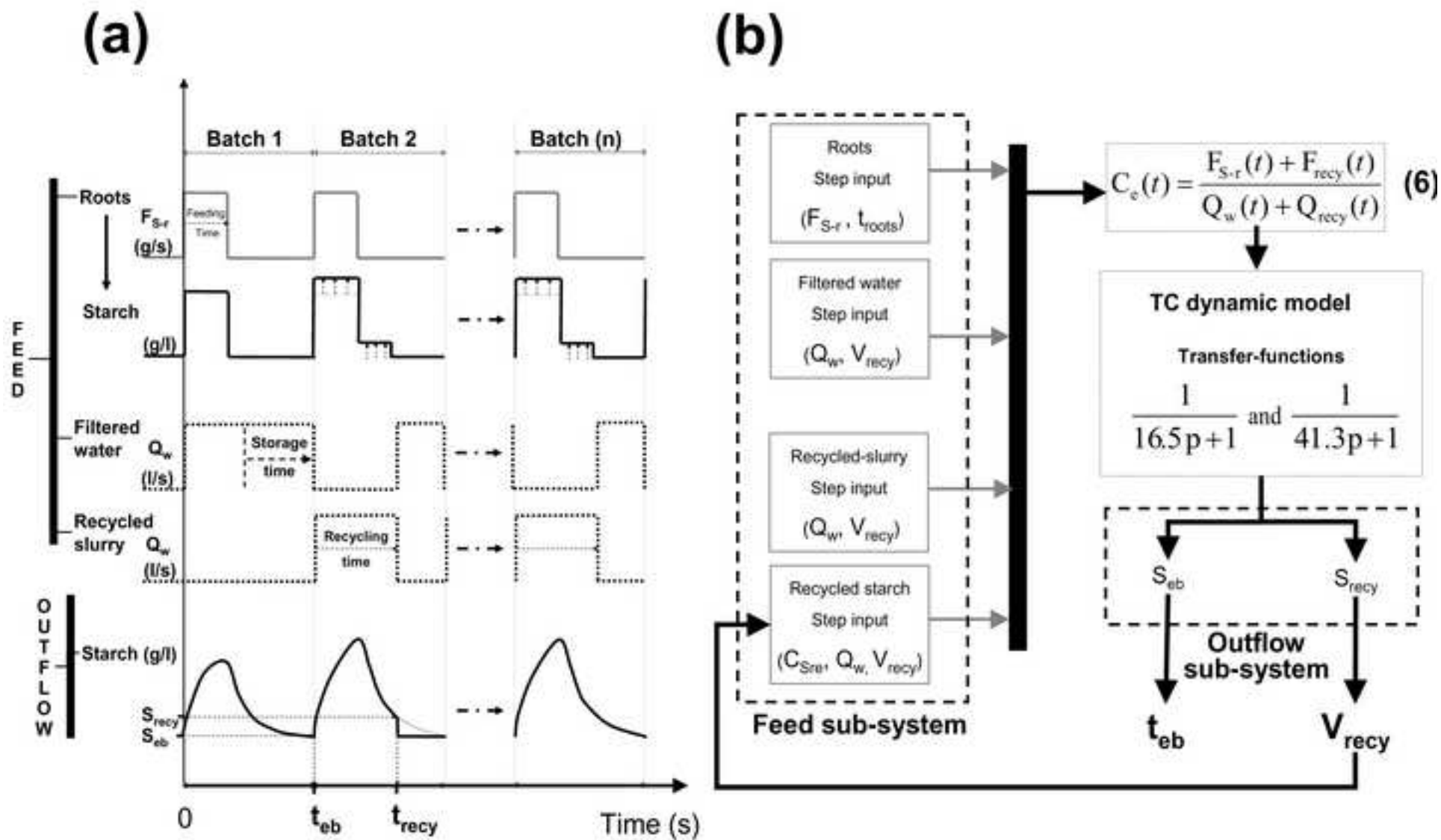
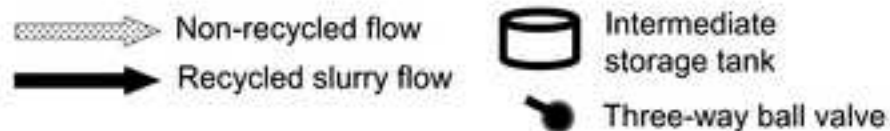
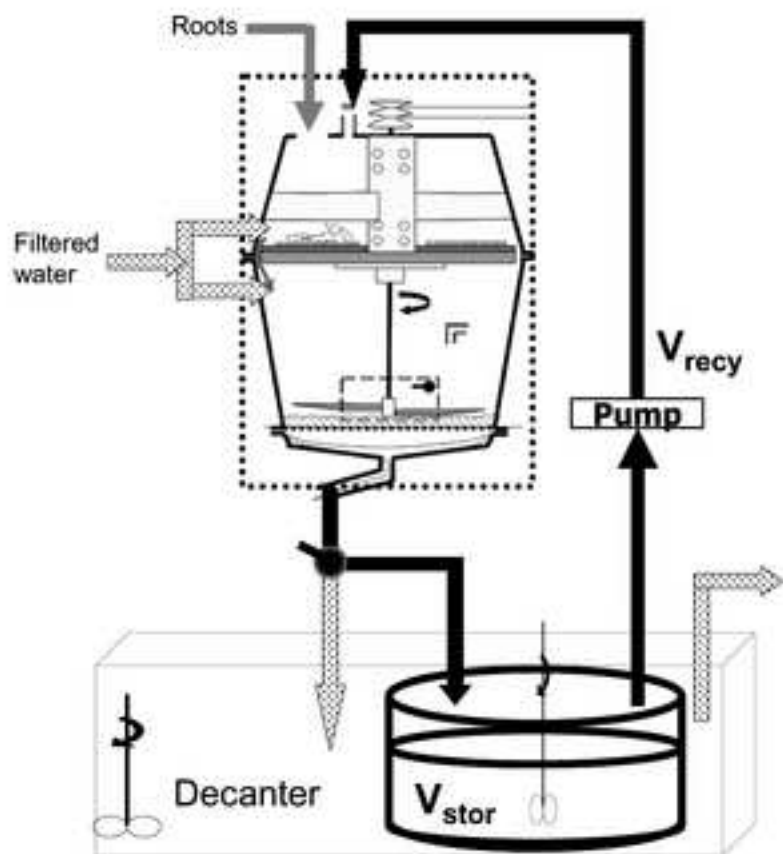


Figure 6
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(a)



(b)

Parameter	S_{recy} [g/l]	V_{recy} [l]	Simulation (48 batches)		
			V_{stor} [l]	V_w [l/kg]	Water-volume Saving [%]
Without recycling					
processing unit	0	0	0	12.8	0
model	0	0	0	13.8	0
With recycling					
option 1	3.4	40	47	11.9	14
option 2	10	100	102	9.1	35
option 3	20	100	139	9.1	35
option 4	20	130	151	8.4	43