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Effect of Some Operating Parameters on the Reversing Continuous Countercurrent Extraction Process

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ABSTRACT

The effect of some operating parameters on the reversing continuous countercurrent extraction process including the degree of inclination of the extractor and the percent forward progression by a screw conveyor was reported. Garcinia fruit was selected as model solid while sucrose was used as soluble solid for the diffusion system. The results showed that the degree of inclination provided certain effect on concentration profile, however was still ambiguous. For experimental purpose, 5, 7 or 10° can be used. Nevertheless, the solid and liquid phase were not in contact at the end point of the extraction unit in the case of 7° and 10° where the specified draft was 1.6. Based on the highest yield, a slope of 10° should be chosen. For a percent forward progression range of 10 - 20 %, the concentration profiles for both liquid and solid phase were almost identical. However, the lowest 5 % percent forward progression led to highest concentration profile for the liquid phase.

Keywords: garcinia fruit, *Garcinia atroviridis*, degree of inclination, yield, slope of extractor, percent forward progression, evaporation, concentration profile

INTRODUCTION

The continuous extraction process has been used in process industries for more than 50 years. Among a number of invented extractors for the process, the extraction unit that can be regarded as a prototype model of later versions of extraction machine is DDS extractor. The unit was developed by De Danske Sukkerfabriker from the Danish Sugar Corporation and introduced in the 1950s. It is one of the most widely used and versatile instruments. Its first application was for the extraction of sugar beet [1]; a modified DDS was then later used for sugar cane [2].

In 1983, the Commonwealth Scientific and Industrial Research Organization (CSIRO) of Australia in cooperation with Bioquip Australia Pty Limited developed another countercurrent extractor. Unlike the DDS diffuser, which is a twin screw extractor, the CSIRO unit is a single screw one. The unit has been called the reversing continuous countercurrent extractor (RCCE) and demonstrated to have potential for high yields of solubles, flavours and colours from wide ranging materials. According to the tests run for apples, pears, and oranges in comparison to a mechanical press, the results showed that CSIRO unit gave a higher yield of total soluble solids, a better juice quality and less suspended solid after purification [3].

Although continuous countercurrent screw extractors are still in use [4,5]. Surprisingly, the effect of some operating parameters like the degree of inclination (slope) of the extraction unit and the percent forward progression by the screw conveyer have never been clearly reported. In Europe, the degree of inclination of the DDS diffuser was only claimed to be 7°, while in the United States for finer cossette materials of sugar beet, it was 5° [1]. For the RCCE, the percent forward progression which influences the movement of solid and liquid materials is normally set to 10 % [3].

Our project aims to fill up some engineering gaps. The slope of the extraction unit and the percent forward progression of the RCCE will be studied in the range of 5 - 10° and 5 - 20 %, respectively. If the investigation is successful, the 2 operating parameters may be further included into the mathematical model for simulation purposes of the concentration profile for the RCCE process.

A garcinia fruit (*Garcinia atroviridis*) is an excellent model solid for diffusion systems due to its rigid and firm texture [6,7]. Nevertheless, the inherent nature of agricultural materials creates a problem in controlling the initial concentration uniformity. To remove this instability and obtain a unique initial concentration before running each system, instead of extracting the inherent soluble compounds like the hydroxycitric acids (HCA) from the solid phase, the diffusion of sucrose in a solid model was considered.

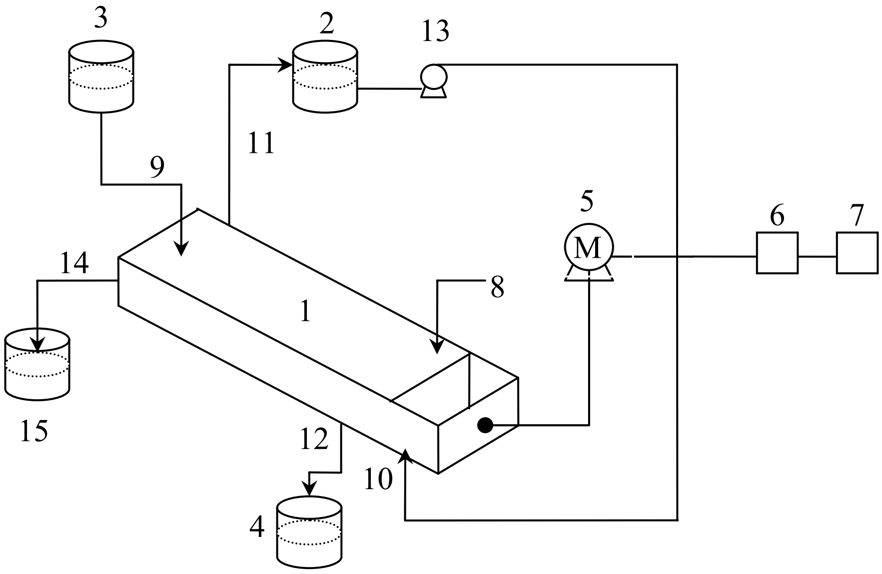
MATERIALS AND METHODS

1. Material Preparation

Fresh unripe garcinia fruits, 3 months old since fruit setting, green in color, were purchased locally from Nakhon Si Thammarat, Thailand. The unpeeled fruit were cut with a meat slicer into infinite slabs to the required thickness of ~ 2 mm. The diameter of the slab was in the range of ~ 7 - 10 cm. All materials were immersed in 0.06 % (w/v) calcium chloride for 6 h to maintain their firmness, drained for 30 min on a screen and then immersed in 60 % (w/v) sucrose solution (white, food grade) for a certain period (approximately 6 h) until an initial sugar concentration of 30 ± 3 °Brix was reached. The materials were kept at 4 °C with a relative humidity of 75 % RH until used for the reversing continuous countercurrent extraction experiments.

2. Instruments

The reversing continuous countercurrent extractor (RCCE) with a 2 m long extraction chamber was used for the experiments. The solid phase was fed at the lower end of the trough where the relative length (z) was 0 (feeding position). The entering liquid phase was introduced at the other higher end where the relative length (z) was 1. The schematic view is illustrated in **Figure 1**.



1 is the Extraction barrel or trough
2 is the Vessel of hot water circulated in the jacket around the trough
3 is the Vessel of water, the entering liquid phase, fed into the extractor
4 is the Vessel for juice collection, the extract phase accumulation
5 is the Motor for rotating the screw shaft
6 is the Inverter
7 is the Microcomputer
8 is the Area for feeding new solid raw materials at the lower end of the trough where relative length (z) = 0
9 is the Area for feeding water, the entering liquid phase, at the higher end of the trough where relative length (z) = 1
10 is the Hot water inlet to the jacket
11 is the Hot water outlet from the jacket
12 is the Juice outlet, the accumulated extract phase
13 is the Pump for hot water circulation in the jacket around the trough
14 is the Extraction point of solid raw materials from the trough
15 is the Vessel for collection of extracted solid raw materials

Figure 1 Schematic view of RCCE.

3. The Investigation on the Degree of Inclination of Extraction Unit on the Diffusion Process

The inclination (slope) to be studied was 5, 7 and 10°, respectively. Each experiment was carried out in triplicate.

Conditions and Studied Factors The appropriate conditions for RCCE experiments were fixed according to our previous work [8] as follows:- the operating temperature was 55 °C, the draft of extraction was 1.6, the percent forward progression of the screw conveyer was 10 %, the solid retention time was 55 min, and the operating time was 6 h.

Carrying out the Experiments and Sampling the Materials For each treatment, 6 kg of garcinia materials with an initial sugar concentration of 30 ± 3 °Brix were initially fed and fully distributed along the length of extraction chamber. The experiment was then further run by feeding the materials at a rate of 0.5 kg every 5 min (6 kg/h) until the total operation time of 6 h was reached. The feeding position was at the lower end of the extraction trough ($z = 0$) and at the end of the reversing cycle of screw conveyer. The water (9.6 kg/h), the entering liquid phase, was continuously fed countercurrent to the solid phase from the higher end of the trough ($z = 1$). The temperature of entering liquid phase was maintained to 55 °C by the hot water circulated in the jacket around the trough. Samplings were taken for every 20 min for the first 3 h of operation and then every 30 min for the remaining 3 h. Samples of both the liquid and solid phase were taken at each point along the trough. The relative length (z) of sampling position to total length of the through was 0.1, 0.3, 0.5, 0.7, 0.9 and 1.0, respectively. Nevertheless, only concentrations at times between 4 and 6 h (for each sampling point) are shown in this paper. The reason is that the steady state condition was reached at this interval.

Concentrations of soluble solids (mainly sucrose) for each phase of the liquid or solid samples were determined by refractometry using an Abbe Refractometer. The degree of soluble solid concentrations (°Brix) was reported and the determination was also converted to kilogram per cubic meter of occupied volume, when the density of the solid and liquid phase was known, by the Picnometry method.

4. The Investigation on the Percent Forward Progression of Screw Conveyer on the Diffusion Process

The percent forward progression (% FP) was 5, 10, 15 and 20 %, respectively. Each experiment was repeated in triplicate.

Conditions and Studied Factors The selected conditions for RCCE experiments were fixed according to the best results from previous work [8] as follows:- The slope of the extraction unit was 5°, the operating temperature was 55 °C, the draft of extraction was 1.6, the solid retention time was 55 min, and the operating time was 6 h.

Carrying out the Experiments and Sampling the Materials The experiments, sample collections and analyses were done in the same way as that described in previous experiments. Concentrations were also converted to kilogram per cubic meter of occupied volume.

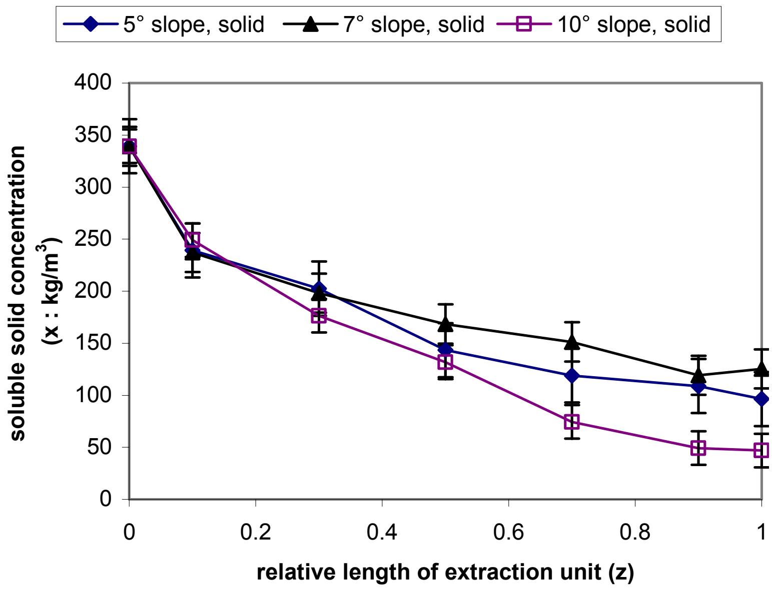
5. Evaporation and Yield Calculation for an Open System of Reverse Continuous Countercurrent Extraction Process

Material balances were made and compared by using the data collected from both experiments. Detailed calculation of the evaporation and yield are discussed later in the text.

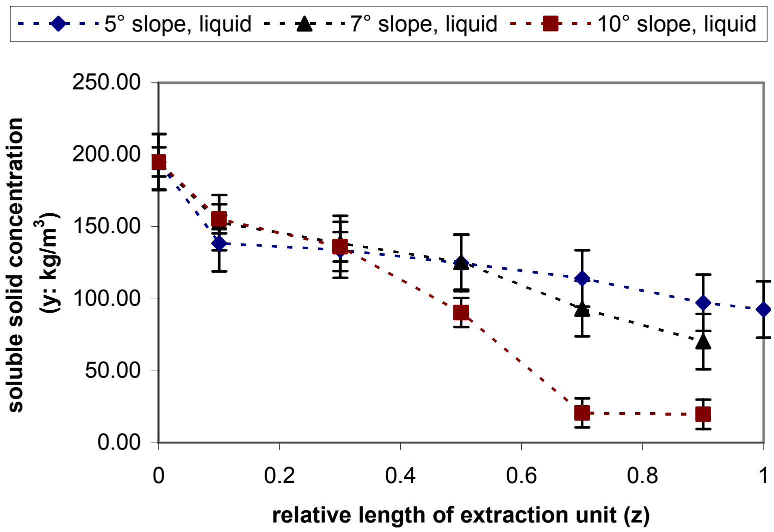
RESULTS AND DISCUSSION

1. Effect of Slope (Degree of Inclination) of Extraction Unit on Concentration Profile

The effect of the slope (degree of inclination) of the extraction unit on the concentration profile is shown in **Figures 2a** and **2b** for the solid and liquid phase, respectively. Only data collected during 4 - 6 h of operation when steady state conditions were reached and where each system was independent of time were used for the consideration. The effect of slope was still found to be ambiguous. It cannot be concluded how the degree of inclination influenced the concentration profile for both liquid and solid phase in this study. Focusing on **Figure 2b**, at a relative length less than or equal to 0.3, the order of liquid profile from top to bottom was 10, 7 and 5°; while at other points greater than 0.3, the order of the profile was 5, 7 and 10°. It is interesting to note that Lee and Schwartzberg [9] obtained a sigmoidal curve for the liquid phase similar to that obtained in our experiment at a slope of 10°. They thus simulated their data by separating the extraction unit into 2 parts, each of which had different values of overall mass transfer coefficient. The simulation was successful, however, diffusivity was not mentioned in their work [8,9].



(a)



(b)

Figure 2 Effect of degree of inclination (slope) of the extraction unit on (a) solid phase and (b) liquid phase, on the concentration profile at different points along the extractor length under steady-state conditions (4 - 6 h operating time).

According to our experiments, a degree of inclination of 5 and 7° gave a similar exponential curve for both solid and liquid phase, while at 10° the sigmoidal curve for the liquid phase was observed. Thus for further work concerning prediction purposes, simulations at 10° may be different from others. The reason may be due to different backing extents along the length of the reactor [9,10] which is caused by too high inclination beyond a certain limit of each reactor size [8]. It is interesting to note that, for slopes of 7 and 10° at the specified operating condition (draft of 1.6), the liquid and solid phase were not in contact at the end point ($z = 1$). In fact, the selected draft was on the basis of previous work [8]. The practical draft was suggested to be no larger than 2.0 due to over dilution [11].

The highest yield of soluble solids (mainly sucrose) was observed at a slope of 10° (86.18 %) while a slope of 7° gave the lowest yield (63.07 %) (**Table 1**). The slope may influence the hold-up volume and hence backmixing to a certain degree. Thus, this parameter should be further investigated. In conclusion at this moment, any value 5, 7 or 10° can be applied for experimental purposes to satisfy a required yield of greater than 60 %, in order to be comparable to the mechanical press method of juice extraction where the value of which was normally 60 % yield [12]. In addition, to clearly answer how the slope of the extraction unit influences the concentration profile, detailed experiments will need to be carried out. We suggest that this operating parameter may be varied precisely based on this study as follows: 0, 2, 4, 6, 8, 10, 12° or 1, 3, 5, 7, 9, 11, 13°. Nevertheless, the draft condition must be higher than 1.6 in order to solve the non-contact problem between the liquid and solid phases at the higher side of the inclined trough.

Table 1 Percentage of evaporation (% *evap*) and extraction yield (% yield*) from the diffusion experiments in an RCCE open system.

Operating parameter	**Parameter value/ characteristics**	**% *evap***	**% yield**
Slope	5°	4.76	71.59
	7°	6.19	63.07
	10°	3.04	86.18
% forward progression	5	8.69	69.17
	10	5.43	80.93
	15	4.02	70.50
	20	0.10	75.01

*Yield calculation was done based on no evaporation.

2. Effect of the Percent Forward Progression by Screw Conveyer of Extraction Unit on Concentration Profile

The effect of the percent forward progression (% FP) for the solid and liquid phase is shown in **Figures 3a** and **3b**, respectively. The results for the solid phase revealed no difference among all experiments (**Figure 3a**). The liquid phase, however, gave some interesting results. It was observed that the liquid profile among 10, 15 and 20 % FP were almost identical especially at $z \geq 0.5$; while the concentration curve for 5 % FP was separated from the others for all z (**Figure 3b**). In addition, the lowest yield of soluble solids (mainly sucrose) was observed for the 5 % FP treatment (69.17 %, **Table 1**). At a constant slope (5°), higher % FP decreased the evaporating rate, thus increasing the extract flow rate and concentration driving force. According to our experimental set-up, higher % FP was associated with lower turbulence due to a slower screw speed. A higher rate of evaporation caused the average concentration of 5 % FP to lie above that of the higher ones (10, 15 and 20 % FP treatment) (**Figure 3b**), causing a drop in driving force. The coupling effect of a lower concentration driving force and decreased flow rate of the liquid phase explained the lower yield for 5 % FP (**Table 1**).

In summary, the percent forward progression which influences backmixing of the solid and liquid phases in the reversing continuous countercurrent extractor to a certain degree [8] yielded no difference in the concentration profile among 10, 15 and 20 % forward progression in this work. If the energy needed for each run is not a concern, the operator can thus select any of this operating values.

3. Evaporation and Yield from Diffusion System

The percentage of evaporation (% *evap*) in the reversing continuous countercurrent extractor was calculated based on an overall operating system by Eq. (1) while extraction yield was based on Eq. (2). The results are shown in **Table 1**. The percentage of evaporation was in the range of 0 - 8.69 %. The high evaporation value might be due to the condition of the surrounding air for the open system and the screw movement. For each operating parameter, the highest yield (after correction by evaporation of the liquid phase) obtained was 86.18 % for the 10° slope while it was 80.93 % for the 10 % forward progression. It is noted that the yield can also be calculated directly from the solid phase where the evaporation in this phase is considered negligible.

$$\% evap = \frac{(S_0 + L_0) - (S_f + L_f)}{(S_0 + L_0)} \times 100 \tag{1}$$

where S and L represent the solid and liquid flow rate, respectively, while the subscripts 0 and f correspond to the inlet and outlet of the extractor, respectively.

$$\% \ yield = \frac{(x_0 - x_f)}{x_0} \times 100 = \frac{\alpha \ y_f}{m \ x_0} \times 100 \qquad (2)$$

where α is the draft of extraction, m is the equilibrium distribution, x and y represent the solute concentration in the solid and liquid phase, respectively, while the subscripts 0 and f are for the inlet and outlet of the extractor, respectively.

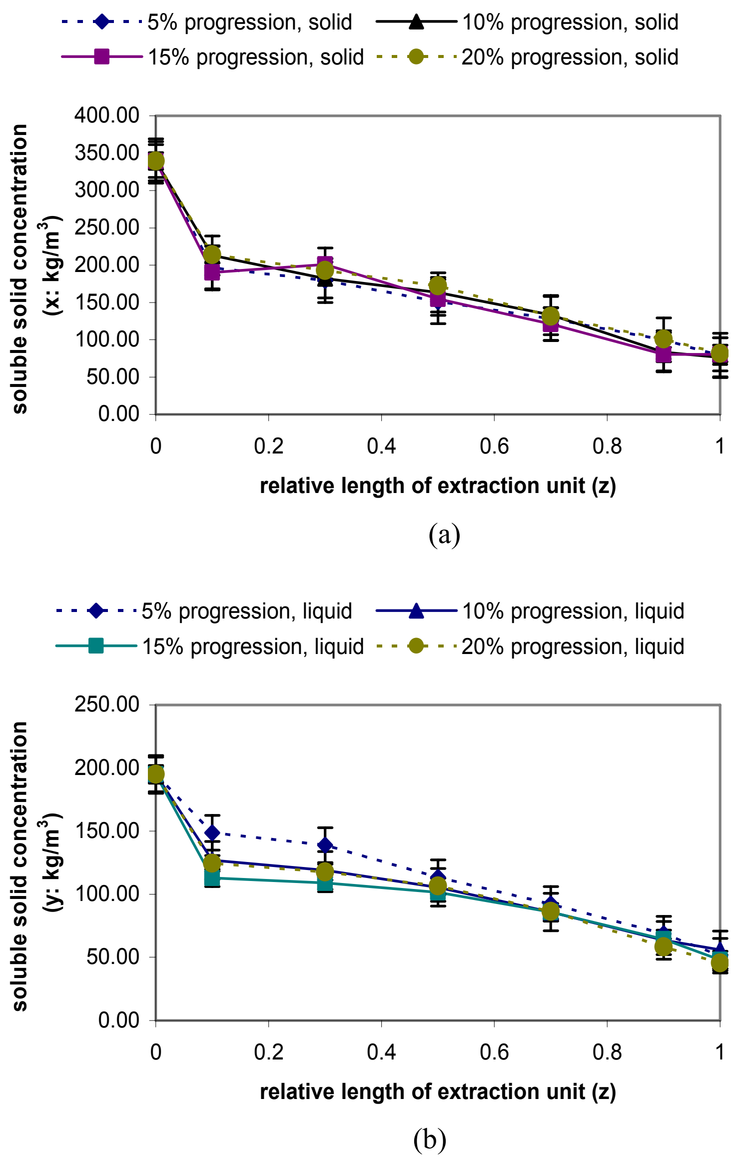


Figure 3 Effect of percent forward progression of the extraction unit on the (a) solid phase and (b) liquid phase, and on the concentration profile at different points along the extractor length at steady-state conditions (4 - 6 h operating time).

It was interesting to note that, unlike the effect of slope and percent forward progression, the effect of shape of solid raw materials gave clearly results on the concentration profiles [8]. According to previous work, the slab shape (2 mm-thick) yielded better results on concentration profiles for both liquid and solid phase in comparison to that of the block shape (7.5 × 7.5 × 7.5 cubic millimeters) [8]. The reason was due to the better surface area of mass transfer to the unit volume [10,13]. A better yield, which was corresponding to the concentration profile, was also reported in this case [8].

The appropriate mathematical model for simulation the extraction phenomenon in the reversing continuous countercurrent extraction process was backmixing-diffusion [8,10,12], especially in the case of steady-state condition [8]. However, the correction of model application may also concern with the degree of inclination at the specified percent forward progression. The simulation will thus be described for the next task.

CONCLUSIONS

The influence of the degree of inclination (slope) on the concentration profile gave rise to a certain effect, however this is still obscure. For experimental purposes, any value (5, 7, 10°) can be used. The solid and liquid phases were not in contact at the end point of extraction unit in the case of 7 and 10° under the specified conditions where the draft was 1.6. Based on the highest yield of solute extraction, a slope of 10° should be chosen. Nevertheless, a slope of 5 and 7° was also practical if the operator is satisfied with a yield higher than 60 %.

The effect of percent forward progression like 10, 15 and 20 % yielded no difference on the concentration profile for both liquid and solid phases. The lowest 5 % forward progression led to the situation that the liquid concentration profile separated from those of other experiments and the highest profile line was established due to evaporation.

For an open system, evaporation was shown to be in the range of 0 - 8.69 %, depending on operating conditions. A satisfactory yield of 80.93 % was obtained under the following conditions: a slope of 5°, 10 % forward progression, slab material with ~ 2 mm-thickness, temperature of 55 °C and draft of 1.6. It was further recommended that the slope of the extraction unit for this specific size of reactor (2 m in length) should be more varied for between 0 and 13°. However, a higher draft must be applied to solve the non-contact problem between the solid and liquid phases.

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บทคัดย่อ

ไวกูณฐ์ ฤทธิรุฒม์[1] และ ชัยรัตน์ ศิริพัธนะ[2]

อิทธิพลของพารามิเตอร์ทำงานบางประการที่มีต่อกระบวนการสกัดแบบไหลสวนทางต่อเนื่องที่มีการผสมย้อนกลับ

งานวิจัยนี้รายงานผลของพารามิเตอร์ทำงานที่มีต่อกระบวนการสกัดแบบไหลสวนทางต่อเนื่องที่มีการผสมย้อนกลับในระบบเปิดอันได้แก่ ระดับความลาดชันของเครื่องสกัดและเปอร์เซ็นต์การเคลื่อนที่ไปข้างหน้าของสกรูว์ลำเลียง โดยใช้ผลส้มแขกเป็นของแข็งจำลองและน้ำตาลซูโครสเป็นของแข็งละลายสำหรับระบบการแพร่ ผลการทดลองพบว่า ระดับความลาดชันของเครื่องสกัดมีอิทธิพลต่อรูปแบบของความเข้มข้นแต่ยังไม่สามารถสรุปแน่ชัดเกี่ยวกับผลกระทบที่เกิดขึ้น ดังนั้นในเชิงปฏิบัติจึงอาจเลือกใช้ระดับความลาดชันที่ 5 หรือ 7 หรือ 10 องศา อย่างไรก็ตาม กรณีที่ความลาดชันเท่ากับ 7 หรือ 10 องศา และเมื่อกำหนดให้ค่าดราฟท์ของการสกัดเท่ากับ 1.6 พบว่า เฟสของแข็งจะไม่สัมผัสกับเฟสของเหลว ณ จุดสุดท้ายของรางสกัด ซึ่งถ้าหากพิจารณาผลได้เป็นเกณฑ์ควรเลือกระดับความลาดชันที่ 10 องศา ส่วนอิทธิพลของเปอร์เซ็นต์การเคลื่อนที่ไปข้างหน้าของสกรูว์ลำเลียงในช่วง 10 - 20 เปอร์เซ็นต์นั้น ไม่พบความแตกต่างอย่างมีนัยสำคัญของรูปแบบความเข้มข้นทั้งในเฟสของแข็งและเฟสของเหลว อย่างไรก็ตาม การเคลื่อนที่ไปข้างหน้าของสกรูว์ลำเลียงที่มีค่าเท่ากับ 5 เปอร์เซ็นต์ จะได้รูปแบบความเข้มข้นของเฟสของเหลวจากการสกัดที่มีค่าสูงสุดเมื่อเทียบกับระดับอื่นๆ

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