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Sedimentação de amido de tapioca usando coagulante natural a partir da semente de Morinega Oleífera Lam

Settling process of cassava starch using natural coagulant from Moringa oleifera Lam seed extract

Sedimentación de almidón de tapioca con coagulante natural de la semilla de Morinega Oleífera Lam

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Resumo

O amido de mandioca tem várias aplicações conhecidas nas áreas alimentícia, de papel, colas e indústria têxtil, incluindo embalagens biodegradáveis. Em pequenas e médias indústrias, o amido é obtido pela lavagem da casca e moagem da raiz da mandioca, seguida pela separação da água e do amido por sedimentação, sendo a polpa concentrada submetida posteriormente à secagem. Uma vez que as partículas de amido são muito finas e de baixa densidade, o tempo de sedimentação são altos, resultando em bateladas com cerca de 18 a 24 h. No entanto, o uso de coagulantes favorece a formação de aglomerados de partículas com maior tamanho e mais pesados, permitindo uma sedimentação mais rápida. Neste trabalho, um coagulante natural obtido do extrato de semente de M. oleifera LAM foi utilizado na redução do tempo de sedimentação do amido de mandioca. Foi avaliado o efeito da concentração inicial das partículas e do teor de coagulante sobre o tempo final de sedimentação por meio de testes de sedimentação batelada. Os resultados mostraram que o uso do coagulante reduziu o tempo de sedimentação em proveta de 14h para 2h, permitindo uma rápida operação em batelada.
Palavras-chave: Amido de tapioca; Sedimentação batelada; Sustentabilidade.

Abstract

Cassava starch has many known applications in food, paper, glue and textile industries, including even biodegradable packaging. In small and medium industries, the starch is obtained by washing the peeled and grinded manioc roots, followed by leaving the washwater for settling before drying the concentrated slurry. Since starch particles are very fine and have a low density, their settling time is high, resulting in a batch operation that lasts for 18 to 24 h. Therefore, it becomes suitable to form larger and heavier starch sets by means of applying coagulants, allowing the particles to settle faster. In the work reported here, the natural coagulant *M. oleifera* seed extract was used to decrease the settling time of the cassava starch. The effect of the initial concentration of the particles and the coagulant content on the final sedimentation time was evaluated on the batch settling tests. The results showed that the use of the coagulant reduced the sedimentation time in a beaker from 14h to 2h, allowing a quick batch operation.

Keywords: Tapioca starch; Batch settling test; Sustainability.

Resumen

El almidón de yuca tiene varias aplicaciones conocidas en las industrias de alimentos, papel, pegamento y textiles, incluidos los envases biodegradables. En las pequeñas y medianas industrias, el almidón se obtiene lavando la cáscara y moliendo la raíz de yuca, seguido de la separación del agua y el almidón por sedimentación, la pulpa concentrada se seca posteriormente. Dado que las partículas de almidón son muy finas y de baja densidad, los tiempos de sedimentación son altos, lo que resulta en lotes de aproximadamente 18 a 24 h. Sin embargo, el uso de coagulantes favorece la formación de grupos de partículas más grandes y pesados, lo que permite una sedimentación más rápida. En este trabajo, se usó un coagulante natural obtenido del extracto de semilla de *Moringa oleifera LAM* para reducir el tiempo de sedimentación del almidón de yuca. Se evaluó el efecto de la concentración inicial de las partículas y el contenido de coagulante en el tiempo de sedimentación final, a través de pruebas de sedimentación por lotes. Los resultados mostraron que el uso del coagulante redujo el tiempo de sedimentación en un vaso de precipitados de 14h a 2h, permitiendo una rápida operación por lotes.

Palabras clave: Almidón de tapioca; Sedimentación por lotes; Sostenibilidad.

1. Introduction

Cassava or tapioca starch is the most valued and versatile maniot derived product. In culinary, it stands out due to its binder properties, as well as neutral flavor, aroma and color, being a healthy gluten-free carbohydrate source. Paper industries also apply the cassava starch in paper pulp to act as a binder, offering more resistance to their final product (Cereda, 2005).

Thailand is the major world starch exporter, able to produce from 3 to 3.5 million tons per year. Although Brazil is the second largest cassava starch producer of the world, only 0.8% of its total production was exported, since the industry generates low surplus to serve that purpose (Felipe et al., 2010). According to the Brazilian Association of Cassava Starch Producers, there were about 72 starch manufacturers in Brazil, which produced about 410.9 thousand tons in 2017. Brazil produced 536.6 thousand tons of cassava starch in 2018, about 27% more compared to the year before (Felipe, 2018).

Cassava starch processing in small and medium industries has some basic steps. After harvesting, the manioc roots are washed, peeled and finely grated, which prepares the raw material for starch extraction. The pulp is then strained over sieves or cloth while water is added, extracting the starch and leaving the fibers and bagasse. After that, the milky water is taken to settler tanks, which separates the water from the starch. The concentrated starch in the bottom may be directly taken to sun drying or left in the tank for around 15 days to ferment and produce sour cassava starch. The finished product is sieved, wrapped in paper or plastic packaging and then stored in a dry place (Matsuura et al., 2003). A first-rate quality starch can be obtained from cassava using only pure water, which makes its processing particularly fitting for developing countries and small industries.

The total time of the settling step in a cassava starch process plant varies from 18 to 24 hours per batch (Souza & Bragança, 2000). Though a complete settling is achieved, the prolonged contact of cassava starch with the water tend to cause the mass to ferment by action of enzymes and microorganisms, which provoke undesired characteristics of sour cassava starch. Another reason that encourages the reduction of the cassava starch settling time is the necessity of processing the manioc roots within 36 hours after their harvest, otherwise losses and darkening of the raw material occurs due to the action of enzymes on the tannic substances, compromising the quality of the final product (Lima, 1982). According to Sajeev & Kailappan (2008), the main process that determines the production capacity of the cassava starch industry is the settling step.

Those factors have led researchers and companies related to the cassava starch sector to seek faster and more efficient ways to process manioc roots, especially in the starch-water separation step (Saengchan et al., 2009). In the classic process used in big industries, a battery of centrifuges and a rotary vacuum drum filter are used before sending the starch to the dryer (Lima, 1982). Since those equipment are expensive, even more for small producers, improving methods of separation of water from the starch using cheaper equipment like settlers would reduce fixed and variable costs in the cassava starch industry, therefore reducing the final price of the product.

Settling of particles or flocs in suspension may suffer the influence of factors such as particle size, shape, diameter, density and concentration of suspended solids. One of the main factors that cause the high settling time of the cassava starch is the size of its particles, which vary from 8 to 22 µm (Daiuto et al., 2002). Being small and low in density, their terminal velocity is consequently low, causing an elevated residence time in the settler. Therefore, it becomes interesting to study coagulants that aggregate the small starch particles, forming heavier and larger sets which settle faster, reducing the required settling time.

Although chemical coagulants such as aluminum sulfate might improve cassava starch settling rate, studies show that its salts are related to many severe mental disabilities, such as Alzheimer’s disease (Awwa, 1990), which discourages its application in the process of a food product. Moreover, in spite of being highly effective, chemical coagulants are unfortunately pointed out as being recalcitrant and expensive.

Many studies have reported the use of natural coagulants as efficient, sustainable (Fatombi et al, 2013) and with a positive impact on settling times (Nasim et al, 2014). Among those, a highlight is given to the seeds of *Moringa oleifera* (MO), a tropical plant native to India. It has a rapid growth and development in humid and hot climates and can survive in dry and low fertility soils (Anwar et al, 2007). It produces about 2000 seeds per year, but depending on the form of cultivation, this number can be five to ten times higher. It is generally known as a medicinal plant and a source of vegetable oil. Its leaves, roots, flowers and immature pods are also a source of nutritive food (D’souza & Kulkarni, 1993; Stoppe et al., 2020). The MO pods contain seeds which mass varies between 150 and 300 mg each (Jahn, 1989).

The MO seed extract was studied by many authors, which pointed out its elevated coagulant potential, turbidity removal and antimicrobial properties (Pritchard et al., 2010; Schmitt, 2011; Anwar et al., 2007). To our knowledge, there have been no reports indicating its toxicity to humans for nutritional or water treatment purposes (Grabow et al., 1985). The active component of the MO seeds which is responsible for the coagulation property is a natural cationic soluble protein, which practically does not affect the pH and conductivity of the medium (Ndabigengesere et al., 1995).

According to data from FAO, chemical coagulants are used in cassava starch process plants to accelerate settling, but there is no study that reports the use of natural coagulants for that purpose.

Considering the factors previously mentioned, the aim of this work was to study the separation process of cassava starch from water employing MO seed extract coagulant in batch settling tests. Seeking to optimize the performance of settlers, the effect of the variables initial starch concentration in suspension and coagulant dosage were investigated using an experimental statistical design.

2. Methods

2.1. Preparation of MO seed extract

The MO seed extract preparation method was used. The pods of MO (Figure 1a) were harvested at Federal Institute of Triângulo Mineiro (IFTM), Uberaba, Brazil (Pritchard et al., 2010).

Figure 1. Photos of Moringa oleifera: (a) pods; (b) seeds; (c) peeled seeds.

(a) (b) (c)

It can be seen in Figure 1 that the MOL seeds were originally in the pods (a), surrounded by a film (b), which is then removed to obtain the seeds (c).

The seeds were manually shelled and its kernel was placed in a blender until a fine powder was obtained. An amount of 4 g of sieved powder, with mean particle diameter less than 1 mm, was moderately mixed with 200 mL of deionized water in a magnetic stirrer for 30 min, at 24 °C. After that, the suspension was left for settling for 10 min, in order to separate the remaining solids from the extract. So, the supernatant liquid was used for coagulant dosages. In order to avoid possible viscosity, pH or coagulant activity changes, a new fresh coagulant extract was prepared and immediately used on each test.

2.2. Procedure of batch settling tests

In order to evaluate the settling process of the cassava starch, an experimental design was carried out with different starch concentrations (*Co*) and coagulant dosages (*Dc*). Laboratory batch settling tests determined the cassava starch settling time. The statistical methods evaluated both factor influence on that response.

The mass corresponding to the adequate cassava starch (MatutoAmafil®) concentration determined by the experimental design was inserted on a 250 mL graduated cylinder and part of its volume filled with deionized water. The correspondent coagulant dosage was added and the graduated cylinder was completely filled with deionized water. The recipient was slowly stirred by hand inversion for 10 min. Then, it was left standing to obtain the clarified supernatant liquid interface height variation with time. The 10 min stirring time is necessary to homogenize the coagulant and to promote gradual formation of the flocs before initiating the settling test.

During preliminary batch settling tests, it was observed the formation of three settling regions, therefore two distinct interfaces. Superior phase (Region R1) had a low turbidity clarified liquid. Right below it, region R2 had a higher concentration of small particles of cassava starch. The interface between R1 and R2 was named IC for it represented full particle settling. At the bottom, region R3 had a higher concentration of large particles and delimited interface IV with R2, marking the compression zone.

2.3. Experimental Design

An experimental design was executed to study the coagulant effect on starch settling. Two variables were evaluated: cassava starch concentration (*Co*) and coagulant dosage (*Dc*). Since MO cause a little effect on the pH of the medium (Ndabigengesere & Narasiah, 1998) and its coagulant effect is not affected by pH in the studied zone (neutral to slightly acid), the factorial design did not include this variable.

Moreover, two responses were analyzed in the experimental design. One of them was the time that interfaces IV and IC took to reach 95% of its total displacement (tIV and tIC), which percentage was chosen by reason of the slow interface movement at the end of each batch settling test. Final settling bed height (H) was also analyzed, for it reveals maximum sediment compression and porosity.

Coded form of factors *Co* (x_1) and *Dc* (x_2) is given by Equations 1 and 2, respectively. The batch settling tests were carried out in tube with starch suspensions of 100, 150 and 200 g.L^{-1}. That range was chosen in order to allow visualization of the clarified interface and provide a reasonable sedimentation time, since higher concentrations favor that interface observation although the particles take more time to settle in that condition. Five different coagulant dosages were used: 0, 2, 4, 6 and 8% (v/v), leading to the performance of 15 experiments.

The experimental data was subjected to the techniques of experimental design and response surface (Alam et al., 2016; Zhao & Zhang, 2013). A multiple regression analysis was used at 10% significance level, to quantify the main effects of the variables, the interactions between them and the quadratic contributions to the responses.

$$x_1 = \frac{Co\text{-}150}{50} \qquad (1)$$

$$x_2 = \frac{Dc\text{-}4}{2} \qquad (2)$$

3. Results and Discussion

3.1 Batch settling tests

Table 1 gives the final starch sediment bed height and the time that interfaces IV and IC took to reach 95% of its total displacement. It was observed that the particles in tests in which coagulant was added took less time to settle, mostly for interface IC. Comparing tests with the same solids concentration, it was observed that the final sediment bed was higher when the coagulant was used, indicating that particles were less compacted.

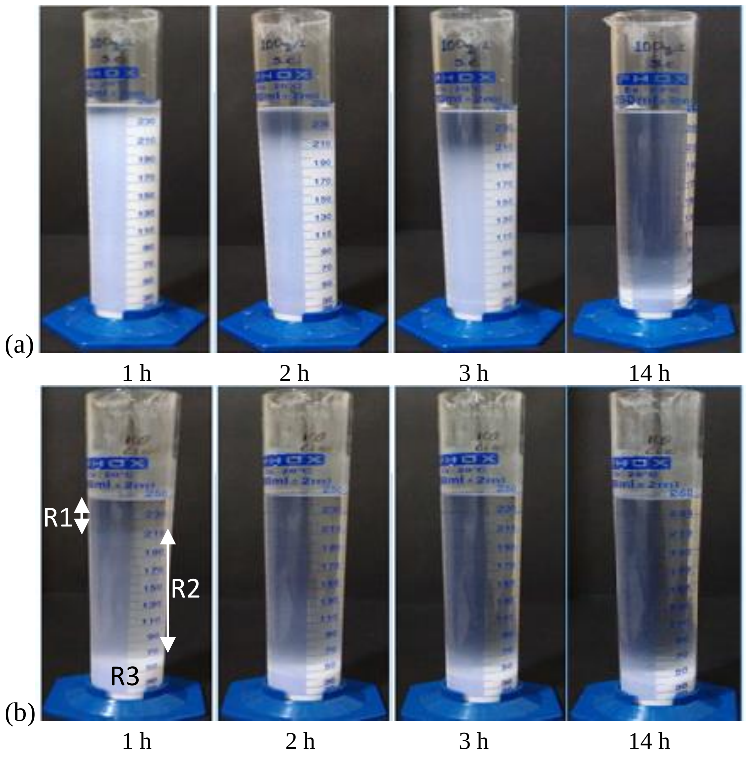
Comparing the settling times of the interfaces, it was observed that the height of Region 3 quickly became constant, that is, most of the particles settled rapidly with only the small and light particles remaining in Region 2. Therefore, statistical tests were applied to the settling time of interface IC, since it represents total sedimentation of the suspension.

Table 1. Experimental design: batch settling test results.

Test	x_1 **(Co [g.L^{-1}])**	x_2 **(Dc [%(v/v)])**	**Final settling bed height H (cm)**	**Time 95% IC** t_{IC} **(min)**	**Time 95% IV** t_{IV} **(min)**
1	-1 (100)	-2 (0)	3.15	795	120
2	0 (150)	-2 (0)	4.68	834	120
3	1 (200)	-2 (0)	6.39	870	225
4	-1 (100)	-1 (2)	3.60	88	80
5	0 (150)	-1 (2)	5.04	217	170
6	1 (200)	-1 (2)	6.66	240	260
7	-1 (100)	0 (4)	3.60	90	90
8	0 (150)	0 (4)	5.04	167	167
9	1 (200)	0 (4)	6.66	260	260
10	-1 (100)	1 (6)	3.42	87	80
11	0 (150)	1 (6)	5.22	160	160
12	1 (200)	1 (6)	6.75	260	260
13	-1 (100)	2 (8)	3.42	88	80
14	0 (150)	2 (8)	5.13	180	180
15	1 (200)	2 (8)	6.84	270	270

Figure 2 shows the batch settling tests at different times, comparing tests with and without coagulant.

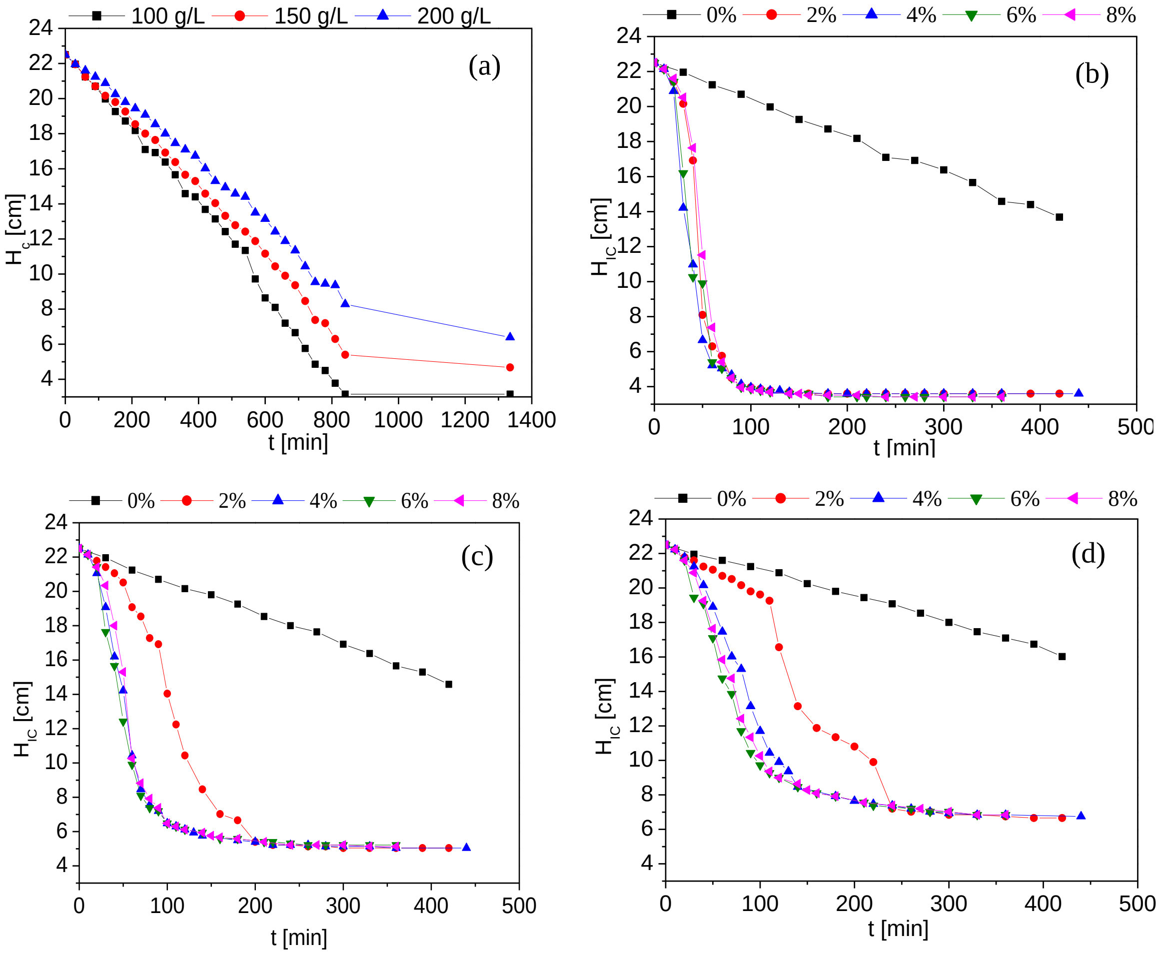
Figure 2. Batch settling tests at times 1, 2, 3 and 14 h for an initial cassava starch concentration of 100 g/L (a) No coagulant (b) 4% (v/v) of coagulant.



It can be seen in Figure 2 the three mentioned phases are observed, as well as the two interfaces (IC and IV) that were moving until both of them encountered, which caused the intermediate region R2 to disappear. The test with 4% of coagulant presented a much higher settling rate, since the clarified liquid interface went down in much less time than in the test without coagulant.

Figure 3-(a) shows interface IC height variation with time for initial starch concentrations of 100, 150 and 200 g.L^{-1} without coagulant addition. It can be seen that the particle settling velocity (the slope of the curve) was reduced with the increasing of the solid concentration. According to Sajeev et al. (2002), suspensions with higher solid concentrations show a more pronounced interaction between the particles. So, the cohesive forces slow down the particles, restricting the relative movement between particles, which reduces the settling velocity.

Figure 3. Batch settling test curves: Variation of interface IC height with time (a) Dc=0 at initial starch concentrations 100, 150 and 200 g.L^{-1}; (b) Co=100 g.L^{-1} (c) Co=150 g.L^{-1} (d) Co=200 g.L^{-1} and all coagulant dosages.



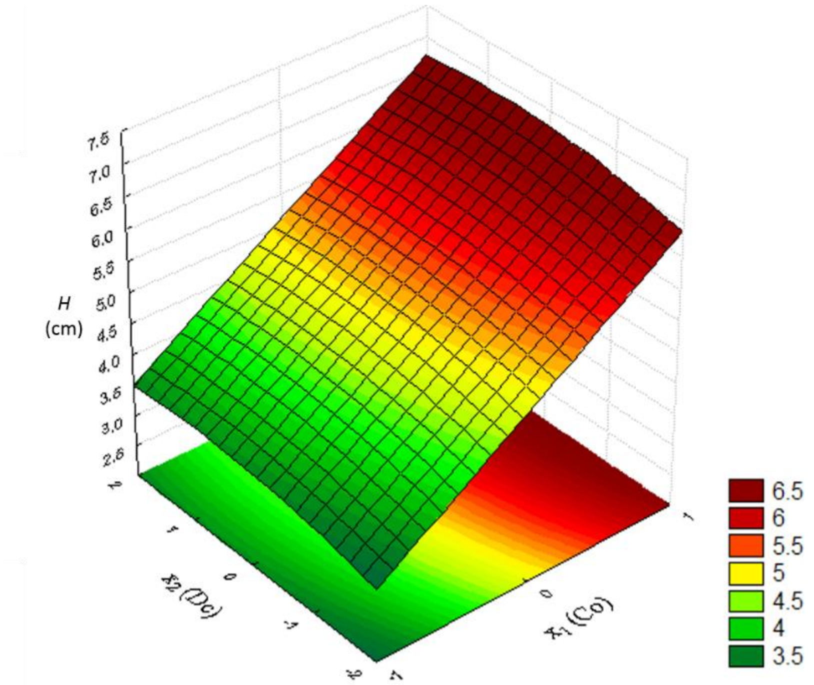
3.2 Effect of cassava starch concentration and coagulant dosage on the final settling bed height (H)

In order to evaluate the effect of the coagulant on the maximum cassava starch compaction, the final height of sediment bed was monitored. A higher thickening is of interest in the sedimentation step because a product with a smaller amount of water is obtained, favoring its subsequent drying.

Equation 3 gives the terms that significantly influenced the final height of sediment layer, in addition to the regression coefficients of each variable ($R^2 = 0.995$). The Figure 4 shows the response surface associated to Equation 3.

$$H = 5.139 + 1.611x_1 + 0.081x_2 - 0.049x_2^2 \tag{3}$$

Figure 4. Response surface showing the effect of starch concentration and coagulant dosage in final sediment bed height.



In addition to the significant effect of the isolated variables, there is also presence of the quadratic term related to the coagulant dosage. Analyzing the response surface (Figure 4), the increase in initial starch concentration in suspension leads to a thicker layer of sediment due to the greater amount of particles. As for the coagulant dosage, intermediate values (4% and 6%) led to thickened bed heights slightly higher than the other conditions, which indicates the formation of larger flocs.

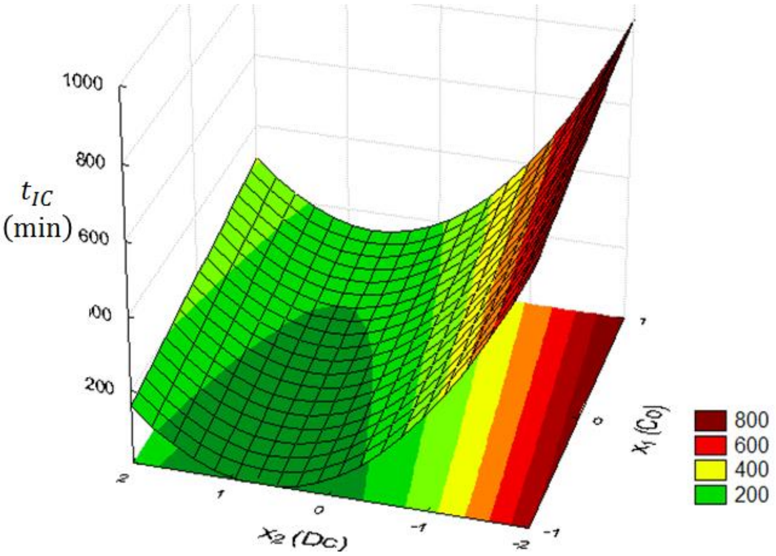
Although flocculation processes accelerate settling rate, a second effect occurs in the system. Due to the attractive forces between particles caused by the coagulant effect, the flocs that reach the bed are kept in the first contact position, not being free to pack (Landman et al. 1988). For that reason, porosity increased and the tests using coagulants had a final settling bed approximately 12% higher than the tests without coagulant addition.

3.3 Effect of cassava starch concentration and coagulant dosage on the settling time

A multiple regression analysis of interface IC settling time was applied to obtain Equation 4**,** which shows the terms that significantly influenced that response ($R^2 = 0.871$). The Figure 4 shows the response surface associated to Equation 4.

$$t_{IC} = 117.160 + 75.20x_1 - 132.00x_2 + 94.95x_2^2 \tag{4}$$

Figure 5. Response surface showing the effect of starch concentration and coagulant dosage in the settling time of interface IC



Both initial starch concentration and coagulant dosage influenced interface IC settling time, but it is noteworthy that the latter influenced more significantly. The longest settling times occurred for conditions without coagulant addition. In the other hand, an increase in coagulant dosage caused an increase in sedimentation rate, reducing settling time. However, from the 4% dosage there is an increase in settling time due to the quadratic term, as shown in Figure 5.

It can be seen that there is an optimum coagulant dosage in Figure 5, from which higher dosages disfavor settling time. This phenomenon is justified because coagulant overdosing may lead to the formation of flocs with irregular shapes, therefore with lower sphericity and terminal velocity. That behavior was observed in studies with other suspensions and coagulants, performed by Arouca et al. (2006) and Fernandes et al. (2010). On account of that, it is necessary to set and adequate dosage to avoid coagulant waste and results of lower efficiency. In this work, we suggest dosages of 4% (v/v), which corresponds to 40 (mL of coagulant)/(L of suspension), since it reduced by 80% the total settling time.

4. Conclusion

The sedimentation time of cassava starch reduced about 80% with the application of MO extract as flocculants. To promote greater efficiency, it was established to use coagulant dosages about 40 mL of extract/L of powder suspension, with initial concentration ranges between 100 to 200 g solids.L^{-1}.

The statistical analysis revealed that coagulant overdoses caused an increase in the sedimentation time, which occurs due to the formation of irregular particles with less sphericity. The use of MO coagulant caused an increase up to 12% in the final consolidated bed height, due to the presence of larger particles, which led to a more porous packed bed.

Thus, this work can contribute to increase the productivity of cassava starch factories, which use the batch sedimentation process, reducing the significantly the sedimentation time.

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