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Obtaining ethanol from cassava waste (manihot esculenta crantz)

Obtenção de etanol a partir de resíduos de mandioca (manihot esculenta crantz)

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Abstract

Aiming to add knowledge to the search for new possibilities of alternative biomass to the biomasses that are already adopted by the sugar-energy industries, this work sought to analyze the possibility of using cassava husks that are residues from the production of starch to obtain fuel ethanol. Treatments to optimize the acid hydrolysis process were carried out, having as variants the concentration of sulfuric acid with 1%, 3% and 5% (m/v), the sieve granulometry varying in 16, 24 and 32 mesh, also having different autoclaving times varying from 10, 35 and 60 minutes with cassava husks (*Manihot esculenta Crantz*), in order to obtain a broth with a high content of fermentable sugars. Therefore, it was possible to verify that the use of cassava husks (cassava plant residues) as sources for obtaining ethanol have promising properties using a concentration of 5% sulfuric acid, a 32 mesh sieve and autoclaving the samples for a period of 22.5 minutes.

Keywords: Ethanol; alcoholic fermentation; biomass; biofuel.

Resumo

Tendo como objetivo acrescer conhecimento para a busca de novas possibilidades de biomassas alternativas às biomassas que já são adotadas pelas indústrias sucroenergéticas, este trabalho buscou analisar a possibilidade de utilizar cascas de mandioca que são resíduos da produção das fecularias para obtenção de etanol combustível. Foram realizados tratamentos de otimização de processo de hidrólises ácidas, tendo como variantes a concentração de ácido sulfúrico com 1%, 3% e 5% (m/v), a granulometria da peneira variando em 16, 24 e 32 mesh, tendo também diferentes tempos de autoclavagem variando de 10, 35 e 60 minutos com as cascas de mandioca (*Manihot esculenta Crantz*), com a finalidade de obtenção de um caldo com alto teor de açúcares fermentescíveis. Portanto foi possível verificar que a utilização das cascas de mandioca (resíduos da fecularia) como fontes de obtenção do etanol possuem propriedades promissoras utilizando a concentração de 5% de ácido sulfúrico, peneira de 32 mesh e autoclavando as amostras por um período de 22,5 minutos.

Palavras-chave: Etanol; fermentação alcoólica; biomassa; biocombustível.

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Introduction

Currently, the new world order has sought self-sufficiency for energy generation, associated with a diversification of the energy matrix that can meet the internal demand of countries, if a new fossil fuel crisis happens. For this reason, interest in research and technical studies of socioeconomic and environmental impacts through the use of biomass for the production of biofuels has increased (PACHECO, 2006).

Brazil stands out internationally in the energy production scenario, as a country with a clean energy matrix, with carbon dioxide emissions below the world average, not only as a result of policy or planning for this purpose, but due to the adequacy of our potential to a situation of scarcity of hydrocarbons (NITSCH, 1991; SCHUTTE; BARROS, 2010).

For more than a century, Brazil needed to import oil to be used mainly as a raw material for fuels, as in the case of diesel oil and gasoline, and it could also be used as a production base for plastic, synthetic polymers, rubbers, oil lubricant, as inputs for the manufacture of medicines and cosmetic products, cleaning products, among other purposes, according to the ANP (National Agency of Petroleum, Natural Gas and Biofuels). In the period of greatest supply crisis in 1973, as a result, investments were made in other energy sources, such as hydroelectric plants and alcohol production with the creation of the National Alcohol Program (Proálcool) through a government decree in 1975 (BARCELOS, 2021; NITSCH, 1991; SCHUTTE; BARROS, 2010).

In 1979, with the second oil crisis, Brazil was able to present the second phase of Proálcool in analogy to the production of cassava biofuel, through the support and incentives granted by Proálcool for the purpose of producing fuel ethanol and then it was possible to manufacture sweet potato alcohol. At the time, it was considered that sweet potato could compete with cassava and sugarcane in terms of alcohol productivity, but it was not successful due to low productivity in the research (MENEZES, 1980).

The so-called modern biomass has shown itself as a renewable alternative that has grown with its participation and has presented significant projections for the future, being able to be used in several forms of advanced technologies, as in the case of the production of biofuels such as ethanol, biodiesel and biogas, and also generating heat and electricity by burning wood and agricultural waste (GOLDEMBERG, 2017).

Some plants that have large amounts of starch, as in the case of cassava, have been an alternative source for use in biomass, being a large source of carbohydrates that are very important for plants, and their physicochemical properties make it a carbohydrate of great importance to industries. Starchy plants have gained prominence for the production of ethanol because they are rustic and easy to handle crops. (CEREDA, 2008; ROCHA; DEMIATE; FRANCO, 2008).

Cassava is a plant that has been cultivated since the native peoples of antiquity on this continent, being from a tropical region and presenting good development in favorable conditions in all tropical and subtropical climates. It has South American origin, and this plant belongs to the *Euphorbiaceae* family, of the Manihot genus and *Manihot esculenta Crantz* species (AMARAL; JAIGOBIND; JAISINGH, 2007). Its bagasse or bran, as they are popularly known, is a mass acquired during processing that separates the starch, presenting approximately 75% of moisture and, after being dried, it becomes constituted by approximately 63.6% of starch, and having glucose, protein, phosphorus, calcium, potassium, ether extract and fiber as the other constituents (CARVALHO; CEREDA, 2005).

Carbohydrates are sugar molecules and are found abundantly in nature, being characterized as a main source of energy for living beings. In foods, they are characterized as agents that impart sweetness and browning in carbonyl reactions from carbohydrates and as gum-forming agents, also acting on the texture of foods. They consist mainly of carbon, oxygen and hydrogen (NASCIMENTO *et al*., 2016).

At different concentrations and with variations in temperatures and reaction times in the processes of hydrolysis of biomass, cellulose is catalyzed by the action of specific acids or enzymes, called acid or enzymatic hydrolysis (WOICIECHOWSKI *et al*., 2002).

The Ministry of Agriculture, Livestock and Supply (MAPA), recommends the use of an autoclave as a methodology to carry out the hydrolysis processes in non-reducing sugars, since the high pressure when combined with the high temperature provided by the autoclave causes the starch to be transformed in reducing sugars in the form of glucose, but the smaller sugars that were present in the samples, in the case of glucose and sucrose, end up being degraded after this process (NASCIMENTO *et al*., 2016).

The hydrolysis of starch causes the chain to become shorter, due to the breaking of chemical bonds. Even if it increases the difficulty in obtaining reducing sugars by

acid hydrolysis in relation to enzymatic hydrolysis, alternatively, acid hydrolysis can be carried out in a more practical and faster way, presenting extremely lower process costs (GRÄF *et al*., 2018; RIBEIRO; GODINHO; MARQUES, 2009).

The acid hydrolysis process is carried out through the use of different types of acids, such as sulfuric, hydrochloric, phosphoric or nitric acid, which can be concentrated or diluted. It is essential that the pH of the hydrolyzate is neutralized after the acid hydrolysis so that the medium presents favorable conditions for fermentation (SILVA, 2013).

Although the chemical processes for the production of ethanol are well known, the most used is the fermentation process because it is more economically viable, and this advantage is due to the large number of natural raw materials that are cheaper and are presented in the form sugary and starchy (SCHMIDELL *et al*., 2001).

The objective of this work was to quantify the glucose and the total sugars present in the samples, to optimize the acid hydrolysis process, having as variants the sulfuric acid concentration, the autoclave time and the sieve granulometry. Also explore the potential of the response surface methodology in the optimization of hydrolysis processes to estimate the amount of ethanol that can be produced from the fermentable sugars obtained from the acid hydrolysis performed with cassava husks (starch residues).

Experimental part

Sample preparation

The tests were carried out at the LaQuiBio Laboratory (Biomass, Biofuels and Bioenergy Chemistry Laboratory), belonging to the Chemistry Department of the State University of Londrina.

The samples of starch residues (cassava husks) were submitted to an oven for drying for a period of 48h at 50 °C. Subsequently, they were ground in an industrial blender and sieved in sieves with granulometry ranging from 16, 24 and 32 mesh.

Sixteen test tubes were separated to start the process of optimizing acid hydrolysis according to the Box-Behnken experimental design, in which different variables were tested, such as concentrations of 1%, 3% and 5% (m/v) of sulfuric acid (H_2SO_4), sieve granulometry of 16, 24 and 32 (mesh) and different autoclave times being 10, 35 and 60 minutes. To carry out the tests, 1g of sample was used for 10 mL (final volume) of sulfuric acid, with its respective concentration. After the autoclave procedure, the samples were cooled to room temperature and centrifuged. Then the pH of the samples were adjusted to 4.5 to 5 with saturated calcium carbonate.

Experimental Design

Initially, the experiment was carried out in the laboratory to obtain results regarding the concentrations of glucose and total sugars using the Box-Behnken experimental design to optimize acid hydrolysis and favorable conditions for alcoholic fermentation of cassava husks to obtain ethanol. In the next step, the independent variables X_1, X_2, and X_3 were transformed respectively into coded variables x_1, x_2, and x_3 using the Statistica software to estimate the yield of ethanol (STATISTICA SOFTWARE FOR WINDOWS, 2018).

The variables of the experiment performed are shown in Table 1.

Table 1 – Coded levels (-1, 0, 1) of the Box-Behnken factorial design for the variables sulfuric acid concentration, autoclaving time, and per mesh.

Variable	Levels of variation		
	-1	0	1
H_2SO_4 concentration (% m/v)	1	3	5
Autoclave time (min.)	10	35	60
Sieve mesh	16	24	32

Source: The authors.

The experiment design matrix for hydrolysis optimization is shown in Table 2.

Quantitative determination of glucose - reducing sugars

The determination of glucose content was performed using the DNS method (3,5-dinitrosalicylic acid) – (MALDONADE; CARVALHO; FERREIRA, 2013). The DNS method is based on the UV-Vis Spectrophotometry Technique. In this method, the reducing sugars form enediols that provide electrons to reduce the 3,5-dinitrosalicylic reagent to 3-amino-5-nitrosalicylate, producing an orange color that will be more intense the greater the concentration of reducing sugars present in the reaction medium. of oxide-reduction. The samples for the DNS calibration curve were homogenized and read in spectrophotometers at 540 nm.

Table 2 – Coded levels (-1,0,1) of the Box-Behnken factorial design for the variables sulfuric acid concentration, autoclaving time, and per mesh.

Original variables			Coded variables			
Sample	H_2SO_4 (% m/v)	Autoclave time (min.)	Granulometry (mesh)	Design -1	0	1
1	1	10	24	-1	-1	0
2	5	10	24	1	-1	0
3	1	60	24	-1	1	0
4	5	60	24	1	1	0
5	1	35	16	-1	0	-1
6	5	35	16	1	0	-1
7	1	35	32	-1	0	1
8	5	35	32	1	0	1
9	3	10	16	0	-1	-1
10	3	60	16	0	1	-1
11	3	10	32	0	-1	1
12	3	60	32	0	1	1
13	3	35	24	0	0	0
14	3	35	24	0	0	0
15	3	35	24	0	0	0

Source: The authors.

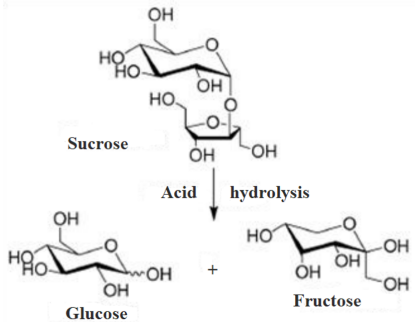
Quantitative determination of total sugars

The quantitative determination of total sugars was performed using the phenol-sulphuric acid method (DUBOIS *et al*., 1956). This method is based on the determination of simple sugars, such as polysaccharides and their derivatives, including methyl esters with free reducing groups. After dehydration by sulfuric acid and subsequent complexation of the resulting products with phenol, the color change of the solution occurs, which can be measured in the visible region and is proportional to the amount of sugars present in the sample. This reaction is sensitive and color stable. The analyzes were performed according to the methodology (DUBOIS *et al*., 1956). Total sugar contents could be determined by spectrophotometry at a wavelength of 490nm and also using a standard glucose curve (1%) with a range of 10 to 90 mg (SILVA *et al*., 2003). The samples were homogenized for the calibration curve and were later read in a spectrophotometer at 490 nm, which can be seen in Table 3.

Estimation of alcohol production

The estimation of the alcohol content was performed considering the glucose fermentation reaction based on the chemical reaction represented in Figure 1.

Figure 1 – Sucrose hydrolysis reaction.



$$C_6H_{12}O_6 \xrightarrow{Zimase} 2CH_3CH_2OH + 2CO_2$$

Source: Adapted from Oliveira, Ferreira and Souza (2009).

Results and discussion

The pre-treatment phase is considered one of the most important steps for the conversion of biomass into fermentable sugars to obtain ethanol, as it is a step that seeks to ensure efficiency and reduce production costs (MARTINEZ, 2016).

The glucose concentration, determined by the DNS method, obtained from the hydrolysis of cassava husk, as well as the estimated values of the alcohol concentration in milligrams of alcohol per gram of dry matter are presented in Table 4.

The phenol-sulphuric acid method determined the amount of total sugars in cassava husk and the results obtained are shown in the Table 3.

Table 3 – Values of concentration of total sugars in cassava husk.

Sample	ABS	Total sugars (ug/mL)	Total sugars (ug/dilution)	Total sugars (mg/g)	Concentration (%)
1	0.578	40.164656	401646.56	401.6	40.2
2	0.65	47.3468	473468	473.5	47.3
3	0.644	46.748288	467482.88	467.5	46.7
4	0.872	69.491744	694917.44	694.9	69.5
5	0.878	70.090256	700902.56	700.9	70.1
6	0.66	48.34432	483443.2	483.4	48.3
7	0.833	65.601416	656014.16	656.0	65.6
8	0.842	66.499184	664991.84	665.0	66.5
9	0.59	41.36168	413616.8	413.6	41.4
10	0.814	63.706128	637061.28	637.1	63.7
11	0.807	63.007864	630078.64	630.1	63.0
12	0.763	58.618776	586187.76	586.2	58.6
13	0.743	56.623736	566237.36	566.2	56.6
14	0.754	57.721008	577210.08	577.2	57.7
15	0.546	36.972592	369725.92	369.7	37.0

Source: The authors.

Table 4 – Values of glucose concentration in cassava husk and estimated concentration of alcohol.

Sample	Glucose (mg/g of cassava husk)	Alcohol concentration (mg/g)
1	56.8	29.03
2	166.9	85.3
3	235.6	120.42
4	195.5	99.92
5	77.8	39.76
6	138.2	70.64
7	202.5	103.5
8	259.8	132.79
9	121.1	61.9
10	145.2	74.21
11	224.8	114.9
12	169.4	86.58
13	225.4	115.2
14	180.2	100.42
15	225.4	111.98

Source: The authors.

The quadratic model for ethanol production, in mg/g, containing the coded independent variables, where the regression coefficients were obtained from the matrix equation $\beta = (A'A)^{-1} A'B$ where A is the matrix of design containing linear, quadratic and interaction terms and B is the response vector. In the resulting equation (1):

$$Y = 109.20 + 11.99x_1 + 11.25x_2 + 23.91x_3 - 11.63x_1^2 - 13.90x_2^2 - 10.90x_3^2 - 19.19x_1x_2 - 10.15x_2x_3, \quad (1)$$

where the significant terms at the 5% level are represented by asterisks, Y represents the estimated value of ethanol production and the values of x_i represent, respectively, the concentration of H_2SO_4 used x_1, the autoclave time in minutes x_2 and the mesh of the sieve used x_3. As it has a value of $p = 0.93$, the term between x_1x_3 was removed from the predictive equation. The linear terms positively influence the increase in alcohol concentration and the quadratic and interaction terms negatively influence, indicating a certain antagonism.

The non-significant terms that are present in the model presented the p statistic varying from $0.05 \leq p \leq 0.12$, values very close to 0.05. In addition, the value of the coefficient of determination R^2 obtained was 0.80 which, according to Joglekar and May (1987), to obtain a good fit of the model to the experimental data, the value of R^2 needs to be equal to or greater than 0.80. In addition, the analysis of variance, Table 5, without the interaction term between the variables x_1 and x_3, which are the least significant, showed that the regression is significant, at a level of 5%, because the value of the calculated F was greater than the tabulated F value and the regression deviation value were considered non-significant, at the same level of significance, which reinforces the quality of the mathematical model obtained.

Table 5 – Analysis of variance of ethanol production.

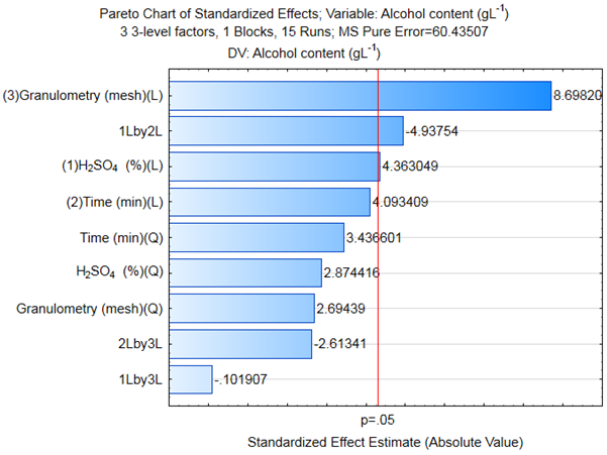
	G.L.	S.Q.	Q.M.	F calc.	F tab.
Regression	8	10057.19	1257.15	$20.80^{(S)}$	19.37
Lack of fit	4	2393.26	598.32	$9.90^{(NS)}$	19.25
Pure error	2	120.87	60.44		
Total	14	12571.32			

$^{(S)}$significant at 5% level and $^{(NS)}$not significant at 5% level.

Source: The authors.

The Pareto chart, Figure 2, containing all terms, shows the most significant variables in the construction of the quadratic model. The numbers in front of the rectangle represent the t-test values. It shows that the granulometry or mesh size and the concentration of sulfuric acid, in that order, are the variables that most influence the process of obtaining reducing sugars and, consequently, the greater production of ethanol. It also shows that the interaction between these variables is not important and can be disregarded in the mathematical model obtained.

Figure 2 – Pareto chart showing the most significant model variables.



Source: The authors.

Figure 3 shows the optimization of the different proportions of sulfuric acid concentration, hydrolysis time and granulometry used, taking into account the maximization of ethanol concentration. The optimization shows that the application of 5% sulfuric acid, from 10 to 22.5 minutes and a granulometry of 32 mesh, produce 128.14 mg of alcohol per 1 gram of cassava husk dry matter. The same figure shows that increasing the acid concentration reduces the hydrolysis time and that an increase in granulometry favors the production of glucose, consequently an increase in the production of alcohol.

A contour region is the projection of a three-dimensional surface onto a two-dimensional plane. The values of the surface, in terms of the response variable, can be represented by lines of various shades of color on a graph containing the independent variables. Next to it are the responses of the dependent variables, which vary from a minimum value to a maximum value (CHENDYNSKI *et al*., 2020; HILL; LEWICKI, 2006).

The response surfaces obtained through the mathematical model, equation (1), for the alcohol content in relation to the binary combination of the independent variables, are shown in Figures 4, 5 and 6. Figure 4 was obtained by fixing the granulometry in 32 mesh which, according to Figure 3, represents its optimal value. In the Figure 3 we can see that the increase in the acid concentration reduces the hydrolysis time.

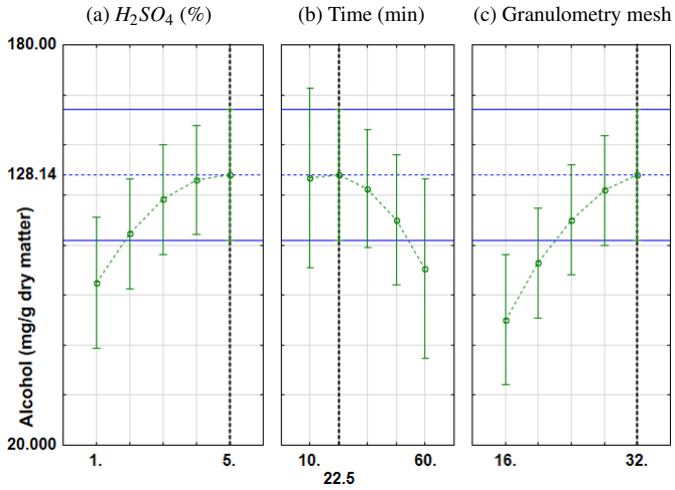
Because starch is not easily fermented by yeasts, the hydrolysis process is essential, because in this process the biopolymers that make up the starch granules break the glycosidic bonds, giving rise to dextrins, maltose and glucose, which have shorter chains (SILVA, 2019).

Figure 5 was obtained by setting the hydrolysis time to 22.5 minutes, which, according to Figure 3, represents its optimal value. It is possible to observe that the greater the granulometry of the cassava peel sample, the greater the concentration of sulfuric acid to promote hydrolysis with an increase in the glucose content and, consequently, an increase in alcohol production.

Cabral *et al*. (2015) used 2% H2SO4 for 96 hours to perform the acid hydrolysis and found that the acid hydrolysis method without heating in an autoclave is extremely time-consuming and of low efficiency in the release of fermentable sugars, not being viable at the industrial level.

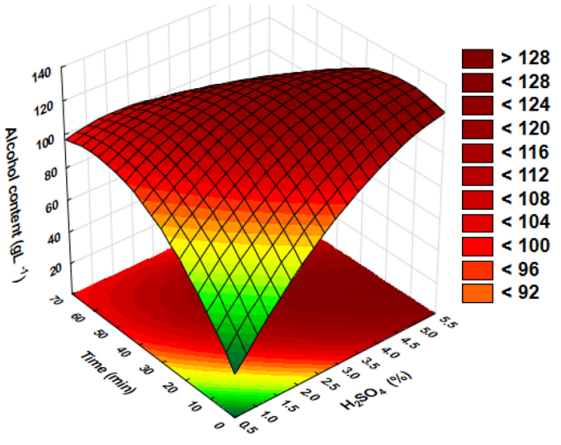
Loureiro *et al*. (2020) could infer that the hydrolysis of cassava processing residues (husks, rinds and tips) when sulfuric acid was used at a concentration of 0.25% for a period of 2 hours of heating at 120°C was effective in releasing fermentable sugars, resulting in (58.25 g/L), which were later used as a fermentation substrate and proved to be a viable process even with a low acid concentration.

Figure 3 – Optimization of predict values for percentage of sulfuric acid, autoclaving time (min.) and per mesh in obtaining alcohol.



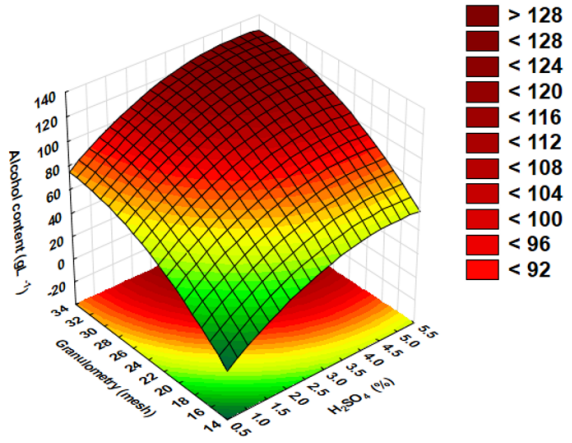
Source: The authors.

Figure 4 – Response surface for alcohol production setting the granulometry at 32 mesh.



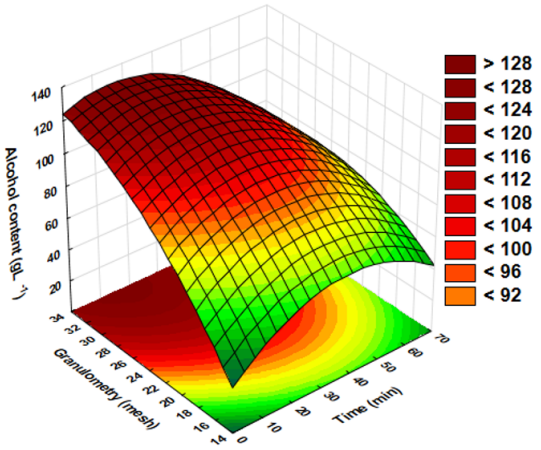
Source: The authors.

Figure 5 – Response surface for alcohol production, setting the hydrolysis time at 22.5 minutes.



Source: The authors.

Figure 6 – Response surface for alcohol production by setting the sulfuric acid concentration to 5%.



Source: The authors.

Figure 6 was obtained by setting the acid concentration to 5% which, according to Figure 3, represents its optimal value. It is possible to observe that at this acid concentration, the greater the granulometry of the cassava husk sample, the smaller the time required to complete the hydrolysis process and, consequently, an increase in alcohol production.

Zenatti (2015) reported in his work that he managed to obtain the conversion of sugars in 99.98 % and that the independent variables provided greater influence on the conversion of reducing sugars, and the variables time and temperature resulted in a more significant influence when using the sulfuric acid concentration of 0.10% and reach a temperature of 140°C for a period of time of 45 minutes.

Conclusion

It can be concluded that cassava husks, which are residues from the production of starch plants, can be used as a source of starchy biomass for the production of ethanol biofuel, in addition to contributing to environmental policies and generating added value to by-products and encouraging the operation of small and large biorefineries.

The result obtained in the optimization of the acid hydrolysis process, having as variants the sulfuric acid concentration, the autoclave time and the mesh of the sieve for the cassava husk, presented the best result of the optimum point with a 5% solution of sulfuric acid, sieve of 32 mesh and having 22.5 minutes of autoclaving. A production of 128.14mg of alcohol per 1g of dry matter of cassava husk can be estimated.

Acknowledgments

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