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KINETIC STUDY OF HYDRATED RED RICE USING A NON-ISOTHERMAL METHOD

ESTUDO CINÉTICO DO ARROZ VERMELHO HIDRATADO UTILIZANDO O MÉTODO NÃO ISOTÉRMICO

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ABSTRACT

The objective of this research was to evaluate through thermogravimetric analysis and its derivative (TG and DTG), the decomposition kinetics of the starch present on red paddy rice submitted to the hydration process at temperatures of 45°C, 55°C, 65° C and 75°C. The analysis of the experimental data of TG in the decomposition region was performed by obtaining the function g (ɑ) for the kinetic mechanisms of the Coats – Redfern (CR) model called unidimensional contraction (R1 model), one-dimensional diffusion (D1 model), random nucleation and growth, Avrami Erofe've Equation (A2 model), Random nucleation and growth, Avrami Erofe's Equation (A3 model) and First-order (random nucleation with one nucleus on the individual particle (F1 model). The results obtained show that in the kinetics of starch decomposition evaluated for red paddy rice, there was a loss of mass corresponding to 51.92- 57.07%, in the temperature range of thermal decomposition of starch of 240.16°C- 356.43°C. As well as the calculation of the function g (ɑ) showed that the first-order kinetic model (F1 model) fitted well to the experimental data, with a coefficient of determination above 99%, confirming that the mechanism that described the decomposition of the starch present in hydrated red rice is of first-order (n=1). With pre-exponential factor (ln A) that reduced the values with increasing hydration temperature. Activation energy and enthalpy values were reduced through the hydration process from 45°C to 75°C.

Keywords: Red rice; Decomposition; Thermogravimetry.

RESUMO

O objetivo desta pesquisa foi avaliar por meio de análise termogravimétrica e sua derivada (TG e DTG), a cinética de decomposição do amido presente no arroz vermelho em casca submetido ao processo de hidratação nas temperaturas de 45°C, 55°C, 65°C e 75°C. A análise dos dados experimentais de TG na região de decomposição foi realizada pela obtenção da função g (ɑ) para os seguintes mecanismos cinéticos: 1) modelo Coats–Redfern (CR), denominados de contração unidimensional (modelo R1); 2) difusão unidimensional (modelo D1); 3) nucleação e crescimento aleatórios, Equação de Avrami Erofe (modelo A2); 4) nucleação e crescimento aleatórios, Equação de Avrami Erofe (modelo A3); 5) Primeira ordem (nucleação aleatória com um núcleo na partícula individual (modelo F1). Os resultados obtidos mostram que na cinética de decomposição do amido avaliada para arroz vermelho em casca, houve perda de massa correspondente a 51,92- 57,07%, na faixa de temperatura de decomposição térmica do amido de 240,16°C- 356,43°C. O cálculo da função g (ɑ) mostrou que o modelo cinético de primeira ordem (modelo F1) se ajustou bem aos dados experimentais, com coeficiente de determinação acima de 99%, confirmando que o mecanismo que descreveu a decomposição do amido presente no arroz vermelho hidratado é de primeira ordem (n=1). Já o fator pré-exponencial (ln A) reduziu os valores com o aumento da temperatura de hidratação. Também verificaram que os valores de energia de ativação e entalpia foram reduzidos através do processo de hidratação de 45°C para 75°C.

Palavras-chave: Arroz vermelho; Decomposição; Termogravimetria.

INTRODUCTION

Rice is a source of energy for most of the human population. Consumer preference for this cereal is associated with economic, traditional, and cultural aspects. Brazil, the largest producer of this cereal in the West, rice is one of the components of greater participation in the diet of its inhabitants, predominating the production and consumption of the common white type, but the red type is also found, preferred by consumers of rice. some states in the Northeast (PEREIRA; DE MORAIS, 2014).

The parboiling process is a hydrothermal treatment where paddy rice is immersed in heated water, resulting in the breaking of the hydrogen bonds between amylose and amylopectin, providing water absorption in a uniform and irreversible way by the grain. In sequence, the grain passes through the stages of starch gelatinization and grain drying (BUGGENHOUT et al., 2013; OLI et al., 2014).

Buggenhout et al. (2013) and Balbinoti and De Matos Jorge (2018) explain that hydration is the main step in the rice parboiling process, as it promotes starch gelatinization. The granules are heated in water, they swell irreversibly, in a phenomenon called gelatinization, in which structural organization is lost (DENARDIN; SILVA, 2009). From a nutritional point of view, gelatinization is important, as it allows the fixation of nutrients transferred during hydration, such as vitamins and water-soluble minerals. Almost all lipid globules present in aleurone and germ cells are destroyed, becoming dispersed in the starchy endosperm of the caryopsis (DENARDIM; SILVA, 2009 and PAIVA et al., 2016).

Corradini et al. (2007) in their studies, evaluated that among the phenomena that change the properties of starch, one of them is gelatinization, the process of transforming granular starch into a viscoelastic paste. To obtain a starch-based thermoplastic, its granular structure must be destroyed to give rise to a homogeneous and essentially amorphous polymeric matrix. Azevedo et al. (2018) report the main features and progress in the use of starch as a polymer in biodegradable films and the interest of the scientific and industrial community in this technology. They also show the rise since 1990 of technology to obtain biopolymers being intensively improved by countries such as China and the USA.

Until the present moment, it was possible to evaluate the mechanism of thermal degradation of different sources of starch through non-isothermal methods according to Guinesi et al. (2006); Pineda – Gómez et al. (2014); Merci et al. (2019); Piglowska et al. (2020). Given these studies, the kinetics of thermal degradation of the starch present in red rice, in the region of occurrence of decomposition and depolymerization of the starch polymeric chains, has not yet been studied.

According to Piglowska et al. (2020) between 210°C and 220°C, the amorphous region is formed and the birefringence of the starch molecules is destroyed in this temperature range. As reported by Miano and Augusto (2018), hydration of cereal grains is an important step during their processing. Because it is a step that precedes other commonly used processes such as germination, cooking, extraction, fermentation, etc. Although there are numerous publications on the study of the mechanisms that involve hydration, there are several aspects that are poorly understood of the complex hydration process of cereal grains (MARTINS et al. 2020, MIANO AND AUGUSTO, 2018).

Given the above, the main objective is to evaluate the kinetics of thermal degradation of hydrated red rice in the temperature range from 45°C to 75°C, determining the kinetic parameters: the pre-exponential factor (A) and the constant (n); determining the kinetic model g (α) for the kinetic mechanisms of the Coats – Redfern (CR) model called unidimensional contraction mechanisms (R1 model), unidimensional diffusion (D1 model), nucleation and random growth (A2 model), nucleation and random growth (A3 model) and random nucleation with one nucleus on the individual particle (F1 model)

MATERIALS AND METHODS

This research was carried out in the Plant Physiology Laboratories, belonging to the Academic Unit of Agronomy, as well as in the Food Engineering Laboratories of the Federal University of Campina Grande (UFCG). The thermogravimetry (TG) and its derivative

(DTG) analyzes were performed at the Nanotechnology Laboratory of the Northeast Strategic Technologies Center – Cetene.

Materials

Red paddy rice produced by the State of Paraíba was used for the tests. The sample field was a batch of 1 kg. Initially, the material was manually pre-benefited to eliminate defective grains and foreign materials. Then the samples were stored in the absence of light, at room temperature, in the quantities necessary for each experiment. The initial water content was determined by the standard oven method at 105 ± 1°C, for a period of 24 h, using five subsamples of 10 g of red rice grains, as described by the Rules for Seed Analysis (BRASIL, 2009).

Samples were weighed using an analytical balance (Bel Enginneering®) with a precision of 0.001 g. The water content was calculated on a wet basis by applying Equation (1), with the final result expressed as the arithmetic mean of the subsamples.

$$Water\ content = \frac{(m_i - m_f)}{(m_f - t')} \times 10 \qquad (1)$$

where: m_i - initial mass, the mass of the crucible plus the mass of wet grains, g; m_f -final mass, crucible mass plus dry seed mass, g; t' – a mass of empty crucible, g.

Pre-drying

A 1kg mass of red rice was pre-dried in an oven (Marqlabor brand) with air circulation at a temperature of 50°C until reaching a water content below 10% on a wet basis. Subsequently, the 1 kg mass was vacuum packed in a Selovac® sealer and stored in B.O.D at a temperature of 8°C.

Hydration process

The methodology followed was based on studies of the hydration process carried out by Miano et al. (2015) and Martins et al. (2020). In the hydration process, samples of 200 g of red rice in husk were used, which were hydrated with distilled water in a beaker in a ratio of two volumes of water to one of the grains. The red rice in the husk was hydrated in a thermostatic bath (Quimis®) at temperatures of 45, 55, 65, and 75 °C, for seven hours, with three replications per temperature.

During the hydration period, the samples were periodically weighed on a digital balance accurate to 0.001 g (Bel Engineering®). For this, the samples were removed from the immersion and left for three minutes exposed to ambient air, so that the surface water was evaporated, weighed, and later returned to the immersion. The water content for a given time after the start of the experiment was calculated based on the mass increase of the samples about the initial mass.

Characterization of red rice

Red rice in nature, rice submitted to pre-drying, and hydrated rice was characterized in terms of thermal behavior using thermogravimetry and its derivative (TG/DTG). The preparation of red rice in natural and red rice without husk were dried in a circulating oven, brand Marqlabor, at a temperature of 50°C, for 24 hours, until reaching a water content below 10% based on wet. Then, the grains of the rice samples without husk were placed in Eppendorf tubes and submitted for analysis.

Thermogravimetric Analysis (TG/DTG)

The thermal stability of decomposition of natural rice, rice submitted to pre-drying and hydrated rice, and the thermal events were evaluated through the thermogravimetric technique (TG) and its derivative (DTG). Thermogravimetric analyses by TG/DTG were performed in a simultaneous thermogravimetric TG/DTG analyzer, model Netzsch STA 449F3 - Jupiter. In each analysis, 15 mg of the rice sample was used in an atmosphere of N2, a flow of 20 mL/min, and in a sample port of Al2O3, with a range from 40- 600°C, with a heating rate of 10 °C/min (beta).

Methods Adopted to Obtain the Kinetics

To determine the kinetics of starch decomposition, the TG/DTG data were used. The fundamental equation, used in all TG/DTG calculations, is described as follows:

$$\frac{d\alpha}{dt} = kf(\alpha) \qquad (2)$$

The conversion rate is given by the following equation:

$$\alpha = \frac{m_i' - m_t'}{m_i' - m_f'} \qquad (3)$$

where m_i' is the initial mass; m_t' is the mass in time t, constant in given temperature: K; m_f' and is the final mass.

The generally occurring Arrhenius equation, and thus the rate constant k, has the following formula:

$$k = A\,exp\left(\frac{-Ea}{RT}\right) \qquad (4)$$

The heating rate is given by the following formula:

$$\beta = \frac{dT}{dt} \qquad (5)$$

To obtain the reaction rate constant, the modified Arrhenius equation, and thus the combination of Equations (2), (4), and (5), is recommended:

$$k = \frac{A}{\beta} exp\left(\frac{-Ea}{RT}\right) f(\alpha) \qquad (6)$$

where k(t) is the reaction rate constant(min-1), f (α) is a function describing the transition (reaction model), Ea is the activation energy (J.mol-1K-1), A is the pre-exponential factor(min-1), T is the temperature of heating (K), and R is the gas constant reaching the value of 8.314 J.mol-1.K-1.

According to Liu et al. (2013) and assuming the non-isothermal conditions of hydrated, for the data analysis, the form of function with infinite limits g(α), given by the equation below, was used:

$$g(\alpha) = \int \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta}\int exp\,(\frac{-Ea}{RT}) f(\alpha) \qquad (7)$$

where the g(α) and f (α) functions for suitable mechanisms are presented in Table 1.

For the graph, the best fits, as well as the slope and intercept values, are used to determine Ea and A parameters from the equation (Coats–Redfern (CR)):

$$ln\left(\frac{g(\alpha)}{T^2}\right) = ln\left(\frac{AR}{\beta Ea}\left(1 - \frac{2RT_{max}}{Ea}\right)\right) - \frac{Ea}{RT} \qquad (8)$$

According to Pigłowska et al. (2020) algebraic expression and plot are recommended for the adopted kinetic model with plots:

$$ln\left(\frac{g(\alpha)}{T^2}\right) = f\left(\frac{1}{T}\right) \qquad (9)$$

The graphs and calculations of linear and non-linear regressions were performed using the ORIGIN® and STATISTICA® computer programs.

Table 1 brings together the kinetic models for the Coats–Redfern (CR) mechanism used in the present work (MERCI et al. 2019).

Thermogravimetric Analysis

Figures 1 to 2 show the thermogravimetry analysis (TG) and its derivative (DTG) of in-natural red rice, red rice submitted to pre-drying, and red rice hydrated at temperatures of 45°C, 55°C, 65°C, and 75°C.

Mechanism	g(α)	f (α)
A2, Random nucleation and growth, Avrami Erofe've Equation	$\sqrt{(-ln\,(1-\alpha)\,)}$	$2(1-\alpha)\sqrt{(-\,ln\;(1-\alpha)\,)}$
A3, Random nucleation and growth, Avrami Erofe've Equation	$\sqrt[3]{(-ln(1\,-\alpha))}$	$3(1-\alpha)(-ln\,(1-\alpha))^{2/3}$
R1,Unidimensional contraction	α	1
D1, One-dimensional difusion	α^2	$\frac{1}{2}\alpha^{-1}$
F1, First order (random nucleation with one nucleus on the individual particle)	$-ln(1-\alpha)$	$(1-\alpha)$

Source: Adapted from Merci et al. (2019)

Figure 1. Thermogravimetric analysis and its derivative (TG-DTG) for in-natural red rice, pre-dried rice at 50ºC, and hydrated rice at temperatures from 45ºC to 75ºC.

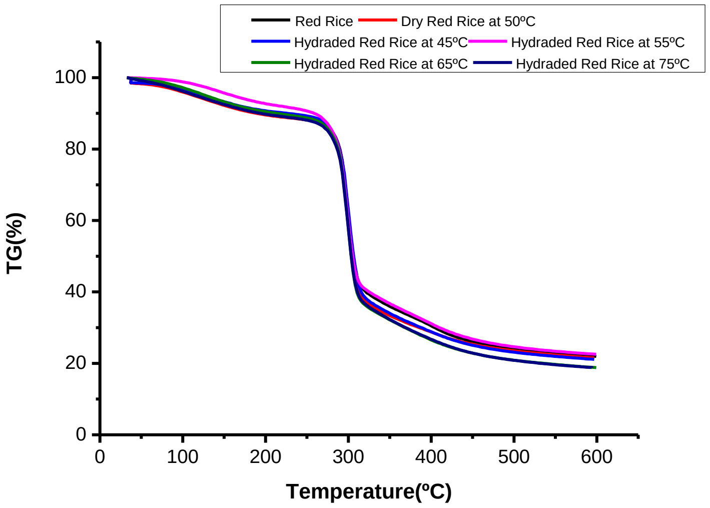
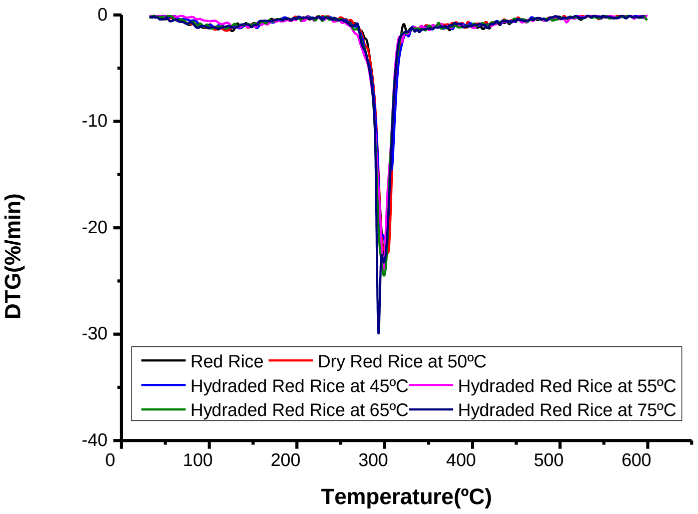


Figure 2. Analysis of the thermogravimetric derivative (DTG) for in-natural red rice pre-dried rice at 50ºC and hydrated rice at temperatures from 45ºC to 75ºC.



From the TG and DTG results visualized in Figures 1 and 2, three stages of decomposition can be seen. In the first thermal event, there was a mass loss of 8.87% to 10.65% rice in the range from 54.13˚C to approximately 258.31˚C. This thermal event corresponds to the dehydration of the samples. The start temperature of the second thermal event related to the step of decomposition and depolymerization of the polymeric chains of the starch present in the rice samples, as seen in Figure 1, occurred from 240.16˚C to 356.43ºC. At these temperatures, the maximum rate of degradation occurs with mass loss from 51.92% to 57.07%. Subsequently, in the temperature range from 340.55ºC to 551.54ºC, the mass continues to decrease, with a loss of 11.72% to 13.13% for the rice samples. In this temperature range, the elimination of carbonaceous products occurs. Similar results to the present study were seen in the studies by Miano et al. (2019) and Pigłowska et al. (2020) in different sources of starch (rice, potato starch, corn, and wheat).

Determination of the kinetic model, pre-exponential factor, and calculation of activation energy

From the data extracted from the thermograms in Figure 1, the information on mass loss and decomposition temperature can be organized in Table 2. Thus, it was found that for the red rice samples analyzed, the decomposition region occurred between 240.16-356.43°C and with a mass loss of 51.92-57.07%, showing that thermal decomposition is the main thermal event that occurred.

Inside the decomposition region, the kinetic models that are displayed in Table 1 were tested (models R1, D1, A2, A3, and F1), so the kinetics of thermal decomposition can be verified through the curves of ln[g(α)/T^2] versus 1/T and with the values of the coefficient of determination (R2) determined, the closer to 1, it indicated a more adequate kinetic model to explain the kinetics of thermal decomposition of the starch present in the red rice samples analyze.

Therefore, it can be seen from the data contained in Table 2 that the kinetic model with first-order reaction (F1) was the best to describe the kinetics of thermal decomposition with coefficients of determination greater than 99% in a temperature range of thermal decomposition greater than 240.16°C and less than 356.43°C. The mass loss of 51.92-57.07%, shows that the highest percentage of mass loss occurred in the region of the main thermal event (thermal decomposition region).

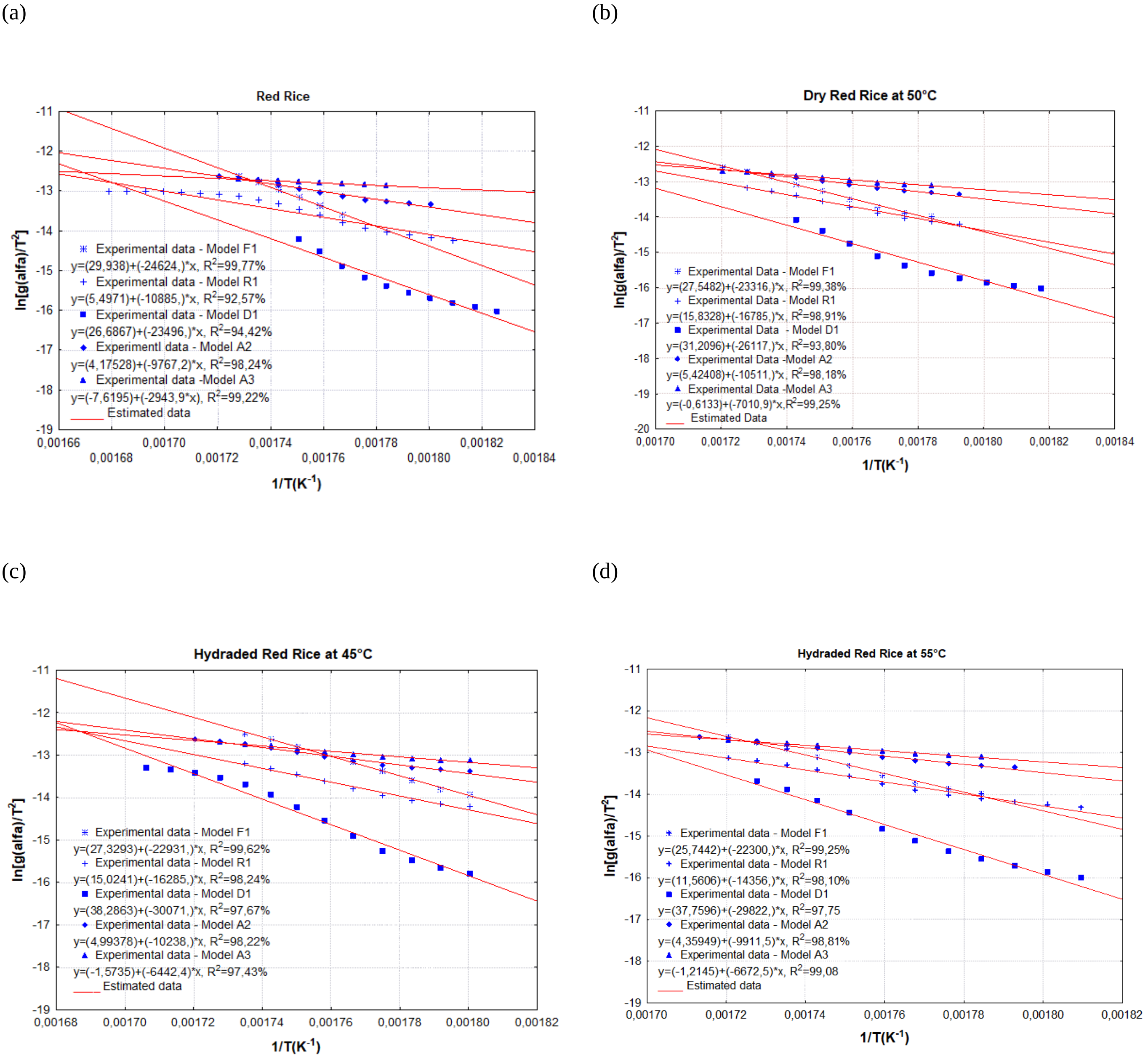
Table 2. Analysis of the kinetic model in the range of thermal decomposition of *in-natural* red rice, rice submitted to pre-drying at 50°C, and hydrated rice at temperatures of 45, 55, 65, and 75°C.

Red Rice	**Temperature Range (°C)**	**Peak temperature**	**Weight loss (%)**	**R1 R^2 (%)**	**D1 R^2 (%)**	**A2 R^2 (%)**	**A3 R^2 (%)**	**F1 R^2 (%)**
In-Natura	251,51-347,93	289,93	51,92	92,57	94,42	98,24	99,22	99,77
Pre drying	252,07-340,55	290,22	53,77	98,91	93,80	98,18	99,25	99,38
Hydrated to 45ºC	246,97-356,43	290,45	56,77	98,24	97,67	98,22	97,43	99,62
Hydrated to 55ºC	258,31-350,20	292,00	54,93	98,10	97,75	98,81	99,08	99,25
Hydratedo to 65ºC	258,31-349,06	293,14	53,74	98,67	98,15	98,77	98,62	99,00
Hydrated to 75ºC	240,16-349,63	294,05	57,07	98,87	97,48	98,73	98,82	99,31

Figures 3 (a-f) contain the curves of ln (g(α)/T^2) versus (1/T) with data on mass loss in the region of decomposition of samples of red rice in nature, red rice submitted to pre-drying and rice submitted to hydration at temperatures from 45°C to 75°C.

The observation of the data obtained from ln (g(α)/T^2) versus 1/T confirms that the reaction that describes the thermal decomposition of the starch present in the analyzed red rice samples is of the first order. The data contained in Figures 3 (a) - (f) confirm the linear behavior for the data fitted to the first-order model (F1 model), confirmed by the coefficient of determination that was greater than 99%.

Figure 3. ln[g(alpha)/T^2) versus 1/T responses for in-natural red rice (a), pre-dried rice (b), hydrated red rice at 45°C(c), hydrated red rice at 55°C(d), red rice hydrated at 65°C(e) and red rice hydrated at 75°C (f).


(a) (b) (c) (d)

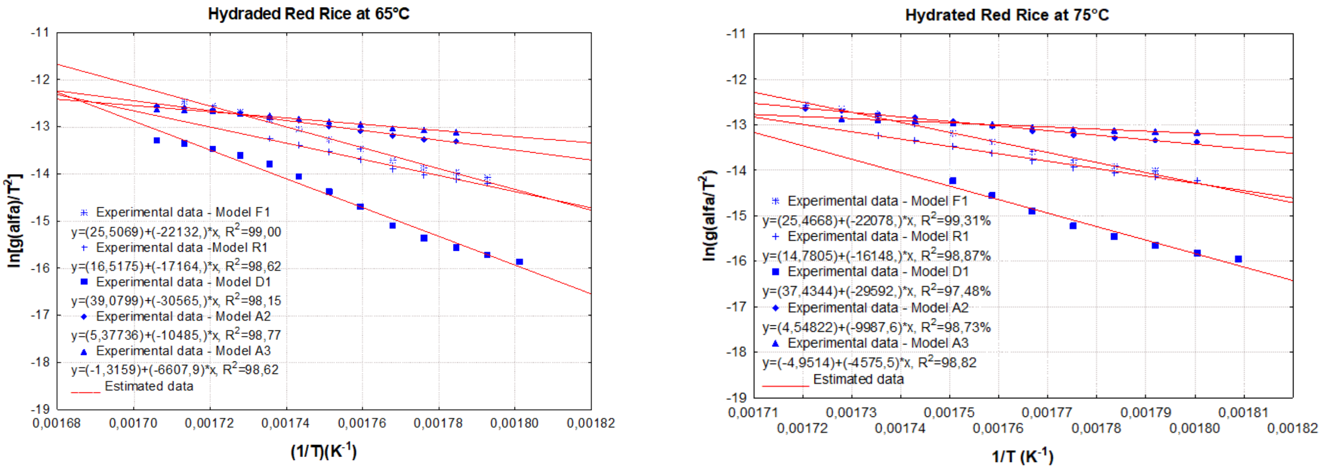

(f) (g)

Table 3 contains the calculated data of the kinetic parameters of the first-order reaction, the pre-exponential parameters, and the activation energy values of the analyzed red rice samples.

Table 3. Kinetic parameters of red rice decomposition for the first-order reaction mechanism (Model F1).

Rice	Ea(kJ/mol)	A(min^{-1})	lnA	k(min^{-1})
Red rice	200,292	2,552x10^{18}	42,383	0,02479
Dry Red rice at 50°C	189,652	2,221x10^{17}	39,942	0,02211
Hydrated Red Rice at 45°C	186,521	1,757x10^{17}	39,707	0,03439
Hydrated Red Rice at 55°C	181,388	3,509x10^{16}	38,097	0,02113
Hydrated Red Rice at 65°C	180,022	2,744x10^{16}	37,851	0,02222
Hydrated Red Rice at 75°C	179,582	2,632x10^{16}	37,809	0,02347

By analyzing the data contained in Tables 2 and 3, it is observed that the hydration process promoted a change in the chemical structure of red rice since above 280°C the amorphous region is produced and the starch granules lose birefringence, so the rice granules have characteristic of an amorphous structure, ceasing to be a semi-crystalline structure.

It was also observed by thermograms that in the starch decomposition region, the analyzed samples have the same mass loss behavior; however, it is observed that for the lowest peak temperature (Tp=289.93 °C) it presented the highest value for activation energy (Ea=200.292 KJ/mol), while the highest peak temperature value high (Tp=294.05 °C) had the lowest activation energy (Ea= 179.582 KJ/mol). This occurred because during the hydration of red rice, in the region of decomposition of the starch, it promoted changes in the structure of the amylose chains and the ramifications of the amylopectin present in the amorphous region formed.

Thus, the starch present in-natural rice degraded well before the samples of hydrated red rice. As confirmed by the peak temperature of the calculated thermograms. In this way, it was verified that the crystallinity was reduced by the hydration of red rice, showing that the samples of hydrated red rice have characteristics of amorphous structures (DENARDIM; SILVA, 2009; PAIVA et al., 2016 and MARTINS et al. 2020).

The fact that in-nature red rice requires higher activation energy of Ea=200.292 KJ/mol than samples of hydrated rice whose activation energy values are between the values of 186.521 KJ/mol to 179.582 KJ/mol. It can be said that the speed of starch decomposition occurs more quickly for hydrated red rice, especially for hydrated red rice at 75°C with an enthalpy of 179.582 KJ/mol.

Regarding the kinetic parameters: pre-exponential factor (lnA) and the kinetic decomposition constant (k) it was verified by the data in Table 3 that the hydration of red rice above 55°C favored faster decomposition kinetics compared to in-natural red rice, because the lnA values decreased from 42.383 to 38.097, respectively. The values of the kinetic constant k reduced from 0.02479 min-1 to 0.02113 min-1, respectively.

In short, the activation energy values presented in the present study of 200.292-179.582 KJ/mol were higher values than the rice activation energy value studied by Piglowska et al. (2020) at 123.733 KJ/mol. Also, the values of the kinetic parameters ln A between 42.383 - 38.097 and the kinetic constant of the first-order reaction (k) between 0.0247- 0.02113 min-1 data from the present research were higher than the values of rice studied by Piglowska et al. (2020), with values equal to lnA=24.110 and k=1.301x10-2 min-1.

In this manuscript, the commercial chickpea hydration process was evaluated in order to reduce and/or eliminate the antinutritional factors present in the grains, before cooking. To help with this characterization, thermogravimetry was used to evaluate the thermal degradation kinetics of the starch present in red rice, in the region where the decomposition and depolymerization of the starch polymer chains occurred. It was found that above 65°C, the grains had starch gelatinized as a pre-cooking, being an alternative to improve the protein intake of its consumers. From the results of the thermogravimetric analysis of hydrated red rice at a temperature above 65°C, the consumption of starch gelatinization. From a nutritional point of view, gelatinization is important, as it allows the fixation of nutrients transferred during hydration, such as water-soluble vitamins and minerals. Almost all the lipid globules present in the aleurone and in the germ cells are destroyed, being dispersed in the starchy endosperm of the caryopsis (GUINESI et al. (2006); PINEDA – GÓMEZ et al. (2014); MERCI et al. (2019); PIGLOWSKA et al. (2020)).

CONCLUSION

From the results of thermogravimetric analysis and its derivative (TG/DTG) of in-natural red rice and hydrated red rice at temperatures from 45°C to 75°C, it was possible to analyze that between 289.93 – 294.05°C, the decomposition and depolymerization of the starch polymer chains present in the rice samples occurred. This reaction by thermal decomposition occurred with a loss of mass between 51.92- 57.07%. The kinetic mechanism was well described by the first-order kinetic model (F1 model) with coefficients of determination above 99%. With pre-exponential factor (ln A) that ranged from 42.383 – to 38.097. And, the model constant (n) equals 1, because the kinetic model is first order. Therefore, red paddy rice subjected to hydration above 65°C is likely to reach the gelatinization temperature of the starch present in its samples. Differential Scanning Calorimetry (DSC) and Fourier Transform (FTIR-ATR) analyses can more accurately confirm the occurrence of starch gelatinization present in hydrated red rice.

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