DECOMPOSITION KINETICS OF GASEOUS OZONE IN PEANUTS[1]

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ABSTRACT: This study was conducted to evaluate the decomposition kinetics of gaseous ozone in peanut grains. This evaluation was made with 1-kg peanut samples, moisture contents being 7.1 and 10.5% wet basis (w.b.), placed in 3-liter glass containers. The peanut grains were ozonated at the concentration of 450 μg L^{-1}, at 25 and 35 ºC, with gas flow rates of 1.0 and 3.0 L min^{-1}. Time of saturation was determined by quantifying the residual concentration of ozone after the gas passed through the grains to constant mass. The decomposition kinetics of ozone was evaluated after the grain mass was ozone-saturated. For the peanut grains whose moisture content was 7.1% (w.b.), at 25 and 35ºC and with flow rates of 1.0 and 3.0 L min^{-1}, the values obtained for time of saturation of gaseous ozone ranged between 173 and 192 min; the concentration of saturation was approximately 260 μg L^{-1}. For the grains whose moisture content was 10.5% (w.b.), a higher residual concentration of gaseous ozone was obtained at 25 ºC, that of 190 μg L^{-1}. As regards the half-life of ozone, the highest value obtained was equivalent to 7.7 min for grains ozonated at 25 ºC, while for those with moisture content of 10.5% at 35 ºC, half-life was 3.2 min. In the process of ozone decomposition in peanut grains, temperature was concluded to be the key factor. An increase of 10 ºC in the temperature of the grains results in a decrease of at least 43% in the half-life of ozone.

KEYWORDS: concentration of saturation, time of saturation, half-life.

CINÉTICA DA DECOMPOSIÇÃO DO GÁS OZÔNIO EM AMENDOIM

RESUMO: Este trabalho foi desenvolvido com o objetivo de avaliar a cinética de decomposição do ozônio em grãos de amendoim. Para avaliar a cinética de decomposição do gás, utilizaram-se amostras de 1 kg de amendoim, com teores de água de 7,1 e 10,5% base úmida (b.u.), acondicionadas em recipientes de vidro com capacidade de 3 L. Os grãos de amendoim foram ozonizados na concentração de 450 μg L^{-1}, nas temperaturas de 25 e 35 ºC, e vazões do gás de 1,0 e 3,0 L min^{-1}. Determinou-se o tempo de saturação, quantificando-se a concentração residual do ozônio após a passagem do gás pela massa de grãos, até que a mesma se mantivesse constante. A cinética de decomposição foi avaliada depois da saturação da massa de grãos com o gás. Nessa etapa, obteve-se a concentração residual do ozônio, depois de períodos de repouso, durante os quais o gás reagia no meio poroso, e dessa forma, era decomposto. Um modelo cinético de primeira ordem foi ajustado aos dados da concentração residual em função do tempo, após linearização. A partir dos valores da constante da taxa de decomposição, definida como a inclinação da reta referente ao modelo cinético de primeira ordem linearizado, foi possível obter a meia-vida do ozônio em grãos de amendoim. Para os grãos de amendoim com teor de água de 7,1% (b.u.), nas temperaturas de 25 e 35 °C, e vazões de 1,0 e 3,0 L min^{-1}, os valores obtidos de tempo de saturação do gás permaneceram na faixa entre 173 e 192 min, com concentração de saturação de aproximadamente 260 μg. L^{-1}. Para os grãos com teor de água de 10,5% b.u., obteve-se maior concentração residual do gás na temperatura de 25 ºC, sendo igual a 190 μg. L^{-1}. No que se refere ao tempo de meia-vida do ozônio, o maior valor obtido foi equivalente a 7,7 min para os grãos ozonizados na temperatura de 25 ºC, enquanto para aqueles com 10,5% de teor de água, na temperatura de 35 ºC, foi de 3,2 min. Concluiu-se que, no processo de decomposição do ozônio em grãos de amendoim, o fator determinante é a temperatura. Observou-se que o aumento de 10 ºC na temperatura dos grãos implica decréscimo de, pelo menos, 43% no tempo de meia-vida do gás.

PALAVRAS-CHAVE: concentração de saturação, tempo de saturação, tempo de meia-vida.

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INTRODUCTION

Peanut (*Arachis hypogaea* Linn.) has high nutritional value and its grains are widely consumed. It is a rich source of oil and vegetal protein, especially folate, niacin, and essential fatty acids (YEH et al., 2002). As a result, it is crucial to produce peanuts that comply with quality standards because they are the ideal substrate for the development of toxigenic fungi. Such fungi not only degrade nutrients but also produce secondary metabolites which are toxic to both men and animals (SABINO et al., 1989). According to CHIOU (1997), fungal infection of peanuts is a matter of great concern for food producers and consumers because of the resultant quality decay and production of aflatoxins. Aflatoxins exert direct influence on the quality of peanut and its derivatives consumed by both people and animals (PRADO et al., 1999). Of the various types of mycotoxins, aflatoxins stand out as presenting high acute and chronic toxicity in animals, including human beings; they can also cause liver damage, such as cirrhosis and hepatocellular carcinoma, in addition to teratogenic effects (ABDULKADAR et al., 2000). In this context, ozone has been proposed as either a fungicide, preventing aflatoxins from being produced, or as a healing agent that can detoxify contaminated products (MCKENZIE et al., 1998; GÜZEL-SEYDIM et al., 2004; WU et al., 2006; YOUNG et al., 2006; ZOTTI et al., 2008).

Ozone is an unstable molecule formed by adding an atom of oxygen to the diatomic oxygen molecule. Among the compounds usually employed in food preservation, ozone is the second most powerful oxidant (2.07 mV), after fluoride (GUZEL-SEYDIM et al., 2004). Such feature makes ozone a strong anti-microbial agent with wide applicability in the food industry, and one of the most powerful sanitizers.

Under normal circumstances, low concentrations of ozone and short contact time are required to inactivate bacteria, fungi, yeasts, parasites and viruses. However, the susceptibility of those micro-organisms to ozone varies according to the physiological state of the culture, the pH of the medium, temperature, moisture content, organic load, presence of additives, among other factors (PUIA et al., 2004; SUSLOW, 2004; STUCKI et al., 2005; PASCUAL et al., 2007; YUK et al., 2007; AKBAS e OZDEMIR, 2008; SELMA et al., 2008). Another fact worth of mention is that ozone was classified as GRAS (Generally Recognized as Safe) in the United States and approved by the FDA (Food and Drug Administration) as an anti-microbial agent to be directly applied on foods, both in its gaseous and aqueous form (FDA, 2001).

One of the advantages of applying ozone is that it can be generated on-site and does not require storage containers, unlike other chemical products. Moreover, ozone is highly reactive and unspecific to a group of micro-organisms, and yields oxygen as the final product of its breakdown (NOVAK & YUAN, 2007).

The high reactivity of ozone is attributed to the electron configuration of the molecule (BELTRÁN, 2005). As an electrophilic agent, ozone lacks electrons in one of the orbital of the atoms of its molecule. When there are too many electrons in one of the orbital of the atoms of the oxygen molecule, the gas is a nucleophilic agent.

Ozone is highly reactive to food and, hence, breaks down into oxygen, leaving no toxic residue (CULLEN et al., 2009). In turn, breakdown of ozone in moisture is affected by factors such as pH and temperature. In high temperature or high pH, breakdown of ozone into a wide range of free radicals is accelerated (CSÉFALVAY et al., 2007; NOVAK & YUAN, 2007; ERSHOV & MOROZOV, 2009).

Although several studies have investigated ozone degradation in moisture as well as the effect of applying such technology to food preservation, there are few studies on issues such as time of saturation, that is, the time required for the applied concentration of gaseous ozone to remain constant, or on kinetics parameters in porous media, for example, half-life, which is the time required for the concentration of ozone to decrease by half.

These parameters are crucial to predict the distribution of ozone within a given porous medium, to evaluate the technical feasibility of the ozonation process, and to estimate the dimension of industrial systems by using gaseous ozone. Given the above scenario, the purpose of this study was to determine the concentration and the saturation time of ozone, and to evaluate the decomposition kinetics of ozone in peanut grains.

MATERIAL AND METHODS

To determine concentration and time of saturation, and to evaluate the decomposition kinetics of gaseous ozone, peanut grains (*Arachis hypogaea* Linn.) were used, moisture contents being 7.1 and 10.5% wet basis (w.b.), at 25 and 35 ºC. The moisture content of peanut grains was determined by the gravimetric method in a forced-air circulation oven and temperature of 130±1 ºC for 6 h (ASAE, 2007).

Gaseous ozone was obtained with an ozone generator based on the Dielectric Barrier Discharge method developed by the Department of Physics at the Aeronautical Institute of Technology (ITA), São José dos Campos, SP, Brazil. In the ozone generation process, moisture-free oxygen obtained from the Mark 5 Plus Oxygen Concentrator was used as input, with purity level of 90±3%.

Ozone concentration was determined by the iodometric method described by CLESCERL et al. (2000), which consists in bubbling ozone into 50 mL solution of potassium iodide (KI) 1 N, and Iodine (I_2) being produced. To ensure that the reaction shifts towards producing I_2, the medium had to be acidified with 2.5 mL sulphuric acid (H_2SO_4) 1 N. The solution was then titrated with sodium thiosulphate ($Na_2S_2O_3$) 0.005 N, and a 1% starch solution was used as an indicator.

Time of saturation and decomposition kinetics of ozone in peanut grains

To determine time of saturation of gaseous ozone in peanut grains, gas was injected at a concentration of 450 µg L^{-1} in 3.25-liter glass containers with 1 kg of grains (Figure 1). At this stage gas flow rates were 1.0 and 3.0 L min^{-1}. The residual concentration of the gas was determined after it passed through the product, at regular time intervals to constant concentration, for the different arrangements of moisture content, temperature and gas flow rate, according to the method proposed by SANTOS et al. (2007). To establish a relation between residual concentration of gaseous ozone and time, the sigmoidal equation was adjusted to the data obtained (Equation 1):

$$C = \left[\frac{a}{1 + e^{-(t-b)/c}} \right] \quad (1)$$

where,

C - concentration of gaseous ozone, µg L^{-1};

t - time, s, and

a, b and c - constants of the equation.

The values of the constants b and c, according to VENEGAS et al. (1998), enabled the time of saturation to be obtained for each arrangement of moisture content, temperature and gas flow rate according the equation 2:

$$t_{Sat} = b + 2c \quad (2)$$

where,

t_{Sat} - saturation time (s).

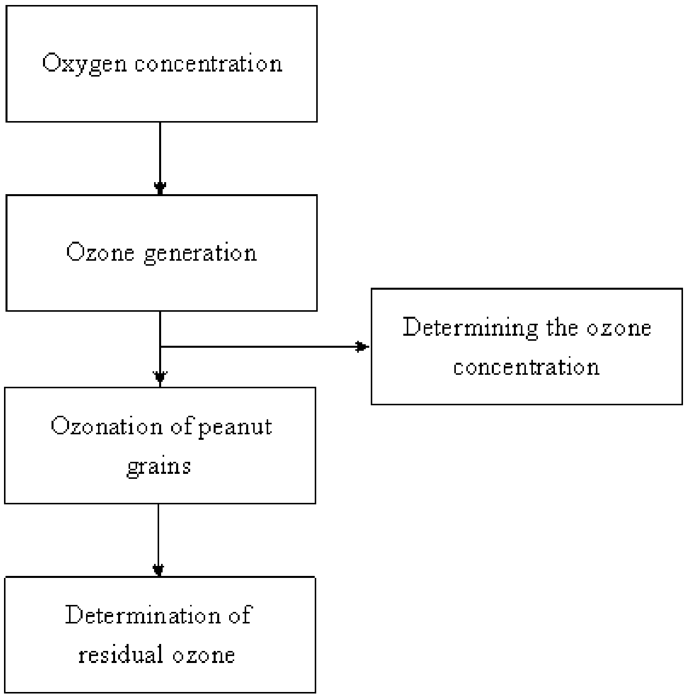

FIGURE 1. Schematic diagram of ozone treatment of peanut grains.

Decomposition kinetics was evaluated after the porous medium was saturated with ozone by quantifying the residual concentration of the gas, after time intervals during which ozone decomposition took place. After each time interval, the concentration of saturation was re-established. This procedure was repeated until the residual ozone could no longer be quantified by the iodometric method. For the evaluation of decomposition kinetics, only the gas flow rate of 1 L min^{-1} was used.

The first order kinetic model (Equation 3) was adjusted to ozone concentration data as a function of time (WRIGHT, 2004). The adjustment of the kinetic decomposition model, following linearization (Equation 4) was made by means of regression analysis. The decomposition rate constant (k) is given by the slope of the line after the adjustment of the integrated and the linearized models.

$$\frac{dC}{dt} = -kC \quad (3)$$

$$\ln C = \ln C_o - kt \quad (4)$$

where,

C - concentration of gaseous ozone, μg L^{-1};

t - time, s;

k - decomposition reaction constant, s^{-1}, and

C_o - ozone injected into the product mass at the initial time, μg L^{-1}.

The values of the decomposition rate constant enabled the half-life ($t_{1/2}$) of ozone to be obtained in peanut grains, which, for the first order kinetic model, is defined by Equation 5 (WRIGHT, 2004):

$$t_{1/2} = \frac{\ln 2}{k} \quad (5)$$

The experiment was conducted in a 2 x 2 x 2 factorial design, with two moisture contents (7.1 and 10.5%), two temperatures (25 and 35 ºC) and two gas flow rates (1.0 and 3.0 L min^{-1}), in randomized complete blocks, with three replicates. The Software SigmaPlot 2001 was used to obtain regression equations and plot the graphs related to time of saturation and decomposition rate.

RESULTS AND DISCUSSION

For peanut grains whose moisture content is 7.1% (w.b.), at 25 and 35 °C and flow rates of 1.0 and 3.0 L min^{-1} (Figure 2 A and Table 1), the values obtained for the saturation time of gaseous ozone were observed to range between 173 and 192 min. The concentration of saturation of gaseous ozone was approximately 260 µg L^{-1}, for all the arrangements of moisture content, temperature and gas flow rate. It should be noted that such value of residual concentration of ozone corresponds to 58% of the initial value, which was 450 µg L^{-1}.

For the grains whose moisture content was 10.5% (w.b.), at 25 ºC (Figure 2 B and Table 1), the saturation time obtained was 230 and 111 min, for gas flow rates of 1.0 and 3.0 L min^{-1}, respectively, with the residual concentration of the gas being approximately 190 µg L^{-1}. The obtained value corresponds to 42% of the initial concentration of gaseous ozone. For grains ozonated at 35 ºC, the residual concentration of the ozone obtained by saturation was approximately 135 µg L^{-1}, which represents 28% of the initial concentration, saturation times being 228 and 126 min, and gas flow rates being 1.0 and 3.0 mg L^{-1}, respectively.

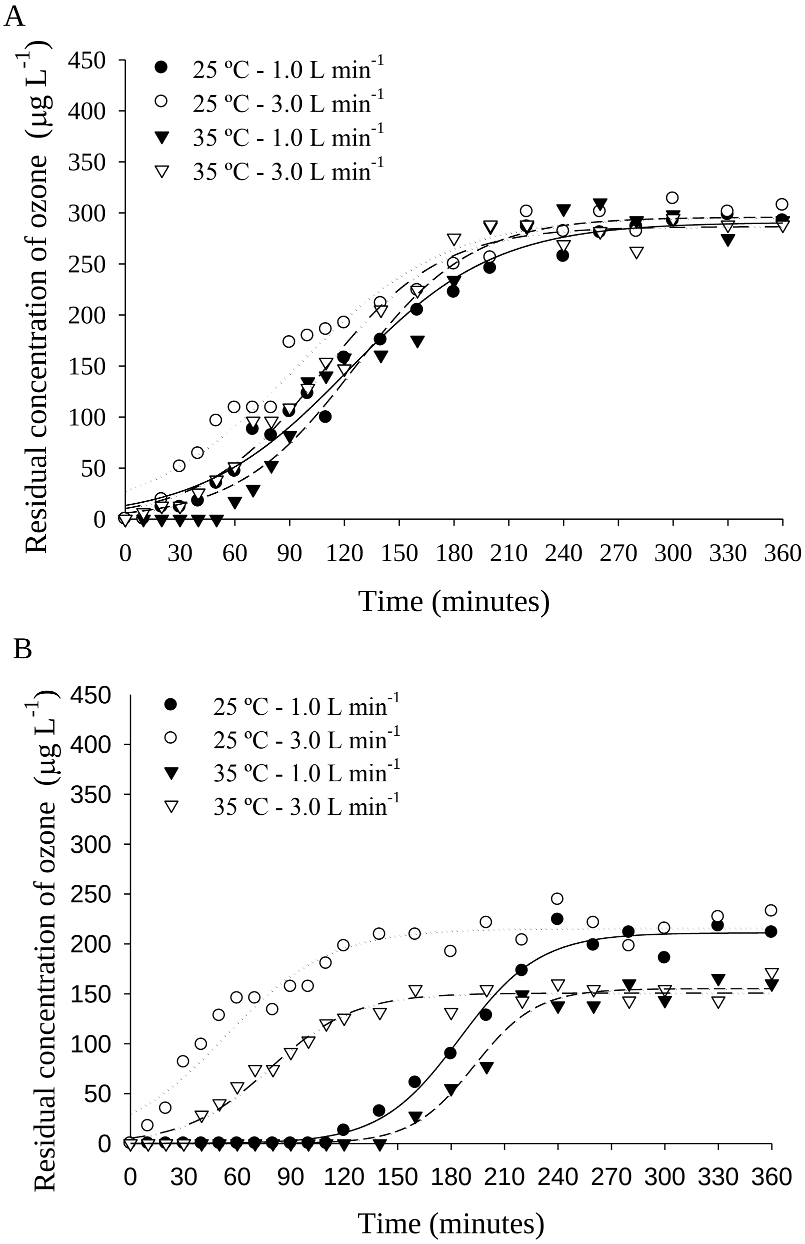

FIGURE 2. Residual concentration of ozone (µg L^{-1}) as a function of time during the saturation process of peanut grains, with moisture contents of 7.1 (A) and 10.5 (B) % (w.b.) and flow rates of 1.0 and 3.0 L min^{-1}, at 25 and 35 ºC.

TABLE 1. Adjusted regression equations and their respective coefficients of determination (R^2) for residual concentration of ozone (µg L^{-1}) during the saturation process of peanut grains with moisture contents of 7.1 (A) and 10.5 (B) % (w.b.) and flow rates of 1.0 and 3.0 L min^{-1}, at 25 and 35 ºC.

Water content (%)	Temp. (ºC)	Flow rate (L min^{-1})	Adjusted equations	R^2	t_{Sat} (min)	C_{Sat} (µg L^{-1})
7.1	25	1	$\hat{y} = \frac{290.83}{1+e^{-\left(\frac{x-121.92}{35.08}\right)}}$	0.98	192	256
	25	3	$\hat{y} = \frac{296.53}{1+e^{-\left(\frac{x-92.92}{40.82}\right)}}$	0.96	175	261
	35	1	$\hat{y} = \frac{295.89}{1+e^{-\left(\frac{x-125.39}{32.23}\right)}}$	0.97	190	260
	35	3	$\hat{y} = \frac{286.59}{1+e^{-\left(\frac{x-107.58}{32.74}\right)}}$	0.98	173	253
10.5	25	1	$\hat{y} = \frac{211.10}{1+e^{-\left(\frac{x-184.92}{22.58}\right)}}$	0.98	230	186
		3	$\hat{y} = \frac{215.82}{1+e^{-\left(\frac{x-52.58}{29.22}\right)}}$	0.93	111	190
	35	1	$\hat{y} = \frac{155.18}{1+e^{-\left(\frac{x-192.82}{17.57}\right)}}$	0.98	228	137
		3	$\hat{y} = \frac{150.80}{1+e^{-\left(\frac{x-78.32}{23.76}\right)}}$	0.97	126	133

t_{Sat} - saturation time; C_{Sat} - concentration time.

As a gas, ozone is 100 times as reactive as chlorine, temperature being one of the factors influencing its decomposition kinetics. According to NOVAK & YUAN (2007), at 20 ºC, the half-life of gaseous ozone is shorter than 20 min. In distilled moisture at 20 ºC, the half-life of dissolved ozone usually ranges between 20 and 30 min. Half-life lowers as the temperature rises, ranging between 8 and 10min at 35 ºC (CULLEN et al., 2009).

In this study, as regards concentration and time of saturation of ozone in peanut grains, the higher the moisture content of the grains, the greater the influence of temperature and gas flow rate, as shown in Figure 2. In grains whose moisture content is 7.1%, virtually the same trend was observed as regards concentration and time of saturation. On the other hand, for peanut grains whose moisture content is 10.5%, as temperature rose and gas flow rate decreased, there was longer time of saturation and lower residual concentration of ozone. The explanation for this trend lies in the fact that moisture content is a key factor in the process of gaseous ozone adsorption on surfaces (GRONTOFT et al., 2004). Thus, an increase in moisture content fostered the adsorption of ozone by the surface of peanut grains and, thus, increased the reactivity of the gas. It should be noted that moisture content has to be considered as a variable in the ionization process when gas, for example, is used as fungicidal agent.

According to RAILA et al. (2006) greater efficiency of ozone in fungal control is obtained in products with higher moisture content. Besides, values for time of saturation for peanut grains are higher than those observed by SANTOS et al. (2007) in all arrangements of moisture content, temperature and gas flow rate for maize grains. The authors obtained saturation time of 70 min for grains with moisture content of 12.8%, temperature of 25ºC and flow rate of 4.6 L min^{-1}.

The trend observed in the residual concentration of ozone in the peanut grains during the saturation process, in the different arrangements of moisture content, temperature and gas flow rate, corroborates the findings of STRAIT (1998), KELLS et al (2001) and MENDEZ et al. (2003). According to the authors, ozone movement through the grain mass is comprised of two phases. In phase 1, the ozone reacts with active sites on the surface of the product at the beginning of ionization; ozone is degraded and thus eliminated from such active sites. Once these elements are eliminated, the gas moves through the porous medium with a reduced degradation rate. When this state is reached, phase 2 takes place.

In turn, the first order kinetic model (Figure 3) was observed to adequately represent the decomposition of ozone in peanut grains as a function of time. Table 2 shows the decomposition rate constants (*k*), defined as the slope of the lines, in the different arrangements of temperature and moisture content.

The ozone decomposition process (Table 2) was faster as the temperature rose. For peanut grains ozonated at 35 ºC, the values for *k* were 0.156 and 0.215 min^{-1} for moisture contents of 7.1 and 10.2%, respectively.

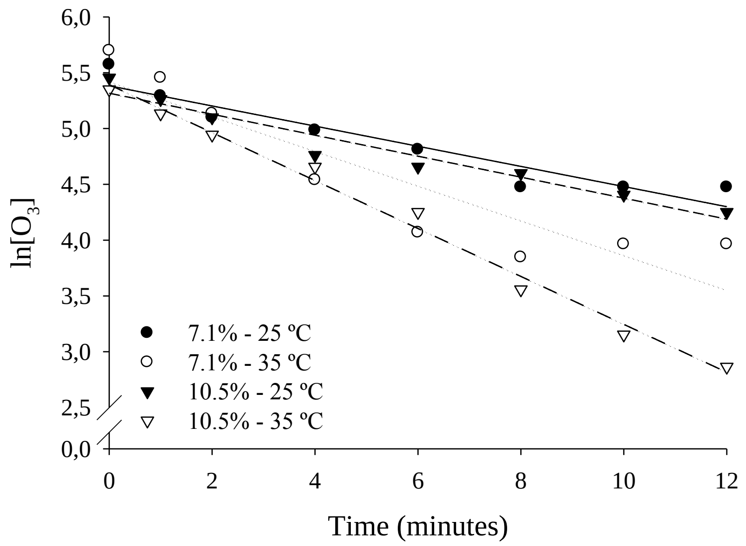

FIGURE 3. First order kinetic model adjusted to the observed data of residual concentration of ozone, following saturation, in peanuts with moisture content of 7.1 and 10.5% (w.b.), at 25 and 35 ºC.

TABLE 2. Regression adjusted equations as a function of time for the residual concentration of ozone in peanut grains in different arrangements of moisture content and temperature and its respective coefficients of determination (r^2).

Water content (%)	Temperature (ºC)	Adjusted equations	r^2
7.1	25	$\hat{y} = 5.383 - 0.090^{*} x$	0.91
	35	$\hat{y} = 5.420 - 0.156^{*} x$	0.83
10.5	25	$\hat{y} = 5.316 - 0.094^{*} x$	0.94
	35	$\hat{y} = 5.393 - 0.215^{*} x$	0.98

*Significant at 5% of probability by the F test.

As regards the half-life of ozone in peanut grains (Table 3), the highest values obtained occurred at 25 ºC, being equivalent to 7.7 and 7.4 min for moisture contents of 7.1 and 10.5%, respectively. The lowest half-life values were obtained for the grains ozonated at 35 ºC.

TABLE 3. Half-life of ozone after saturation of peanut grains in the different arrangements of moisture content and temperature.

Moisture content (%)	Temperature (ºC)	Half-life (min)
7.1	25	7.7
	35	4.4
10.5	25	7.4
	35	3.2

Temperature was a determinant as far as decomposition kinetics is concerned (Table 3). For grains whose moisture content is 7.1 and 10.5%, which were initially saturated, an increase of 10 ºC in the temperature resulted in an increase in the value for of the decomposition rate constant that is equivalent to 73.3 and 128.7%, respectively. Thus, the shortest half-life was obtained for the grains whose moisture content was 10.5% at 35 ºC. These results are in accordance with WRIGHT (2004), who claims that in many chemical reactions the decomposition rate constant increases as the temperature increases. A similar trend was evidenced in moisture by CSÉFALVAY et al. (2007), who observed that an increase of 10 ºC in the temperature of the moisture doubles the value of the reaction rate of ozone. SANTOS et al. (2007) obtained a half-life of 5.57 min for maize with moisture content of 12.8% at 25 ºC. Comparatively, this value of half-life for maize is lower than the ones obtained in the peanut grains with moisture content of 7.1 and 10.5%, at 25 ºC.

CONCLUSIONS

Data analysis and the interpretation of the results enable the conclusion that the concentration of ozone during the saturation process of peanut grains depends on the following parameters: moisture content, temperature and gas flow rate. As moisture content of the grains and the temperature are increased, lower values for the concentration of saturation of ozone are obtained.

Temperature is the major determinant in the process of ozone decomposition in peanut grains. An increase of 10 ºC in the temperature of the grains results in a decrease of at least 43% in the half-life of ozone.

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