



Absorption Kinetics of Sulfur Dioxide in Flint Corn during Steeping

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A mathematical model was proposed to simulate simultaneous sulfur dioxide absorption and reaction during corn steeping. The parameters of the model, the diffusion coefficient and the reaction rate constant, were determined. The reaction rate of sulfur dioxide in corn grain followed first-order kinetics, with rate constants in the range 1.49×10^{-7} to $9.78 \times 10^{-7} \text{ s}^{-1}$ for steeping temperatures from 25 to 55 °C. The Arrhenius activation energy for sulfur dioxide loss was 60.5 kJ/mol. The fitting of the mathematical model to the absorption curves determined the diffusion coefficient of sulfur dioxide in corn kernels. These varied from 1.17×10^{-11} to $5.18 \times 10^{-11} \text{ m}^2/\text{s}$ for the temperature range 25–55 °C. Temperature-dependent diffusion coefficients was shown to obey the Arrhenius equation with an activation energy of 39.8 kJ/mol.

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Introduction

The major source of starch for industry is corn wet-milling, steeping being the key step of the process. During this process, whole kernels are steeped at 45–55 °C in a weak solution of sulfurous acid, 0.1–0.3 g/100 g, for 30–55 h. A complex process takes place, which consists of sulfur dioxide entering through the endosperm of corn kernel, and disrupting the protein matrix that surrounds and binds starch granules (Cox *et al.*, 1944). The efficacy of the steeping operation depends on the rate of diffusion of steep water. The disintegration of the protein network cannot occur without the entrance of sulfur dioxide carried by water.

Some investigations have been carried out in the past to evaluate the rate of water absorption by cereal grains with and without sulfur dioxide in steepwater (Fan, *et al.*, 1961; 1963; 1965; Haros *et al.*, 1995). Fan *et al.*, (1965) studied the diffusion of water into corn kernels in plain water and sulfur dioxide solution and found that the presence of sulfur dioxide in steepwater accelerates the water uptake. They also found that water uptake follows the diffusion equation based on Fick's law. This equation

has been widely used to describe the absorption and desorption of water in cereal grains (Muthukumarappan & Gunasekaran, 1990; Parti & Dugmanics, 1990).

A diffusive model based on Fick's law has also been used to describe the sulfur dioxide absorption in pre-peeled potatoes (Rodriguez & Zaritzky, 1986) and apricots (Rossello *et al.*, 1993). Eckhoff and Okos (1990) investigated the absorption rates of gaseous sulfur dioxide in yellow dent corn. The authors simulated the process assuming that the gas is absorbed by one end of the kernel (tip cap) and diffuses from there into the kernel. The diffusion equation for a semiinfinite cylinder was adopted and the diffusion coefficient of sulfur dioxide in corn kernel was calculated at several temperatures.

Presently, there is no information on the rate of absorption of sulfur dioxide during corn steeping. The objective of this study was to propose a mathematical model based on the mass transfer equation for simultaneous diffusion and reaction on a homogeneous solid to evaluate the concentration profiles and mean concentrations of unreacted sulfur dioxide in corn kernels. The results were tested for different steeping conditions, including those corresponding to industrial operating conditions. Additionally, the present work includes an experimental determination of the reaction rate constant of sulfur dioxide loss in corn, as well as its dependence on the steeping temperature.

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Mathematical Model

The migration of sulfur dioxide into corn kernel during steeping is described as a simultaneous process of mass transfer and chemical reaction. The following assumptions were performed: (1) Fick's law holds for the diffusion of unreacted sulfur dioxide; (2) the disappearance of sulfur dioxide in the kernel follows a first order irreversible reaction; (3) the kernel is a solid of spherical shape whose dimensions remain unchanged, i.e. the swelling of the kernel is neglected; (4) during steeping, corn kernel is isothermal, i.e. heat transfer equation is neglected; (5) the diffusion coefficient and reaction rate constant are independent of sulfur dioxide level.

The validity of the first assumption was tested for gaseous sulfur dioxide absorption by corn (Eckhoff & Okos, 1990) as well as for the absorption of sulfurous acid in fruits and vegetables (Rodriguez & Zaritzky, 1986; Rossello *et al.*, 1993). Little experimental evidences exist in relation to assumption (Fan *et al.*, 1961). Eckhoff and Okos (1986) found that the loss of sulfur dioxide in yellow dent corn follows first-order kinetics. Similar results were found for sulfur dioxide loss in other food materials (Legault *et al.*, 1949; Bolin & Boyle, 1972). In the present work, the reaction kinetics of sulfur dioxide in corn was experimentally determined, as described in Materials and Methods.

Although corn kernel is irregular in shape, it was assumed a simple configuration to facilitate the calculus. In fact, corn has been previously assumed to be a sphere to simulate drying or absorption processes (Fan *et al.*, 1965; Parti & Dugmanics, 1990; Haros *et al.*, 1995).

Finally, the hypothesis of isothermal was frequently used in the literature to simulate the soaking process of various cereal grains (Becker, 1960; Fan *et al.*, 1963).

Based on the above considerations, the partial differential equation describing the simultaneous diffusion and first-order chemical reaction process (Crank, 1957) is:

$$D \left\{ \frac{\partial^2 c}{\partial r^2} + 2 \frac{\partial c}{\partial r} \right\} - kc = \frac{\partial c}{\partial t} \quad \text{Eqn [1]}$$

In Eqn [1], c represents the local concentration of sulfur dioxide in corn kernel free to migrate, t is the time, r the radius, D the diffusion coefficient and k the reaction rate constant.

To solve Eqn [1]; it was assumed that the concentration of sulfur dioxide at the surface of the solid is in equilibrium with that of the medium and, therefore, the mass transfer resistance in the steeping solution was ignored. The boundary and initial conditions used to solve Eqn [1] were:

$$t = 0; \quad c = 0; \quad 0 < r < R \quad \text{Eqn [2]}$$

$$r = R; \quad c = c_e; \quad t > 0 \quad \text{Eqn [3]}$$

$$r = 0; \quad \frac{\partial c}{\partial r} = 0; \quad t > 0 \quad \text{Eqn [4]}$$

where R is the equivalent radius of the sphere whose volume is equal to the volume of the kernel and c_e represents the equilibrium concentration of sulfur dioxide at the surface of the kernel. Equations [1] to [4] were

solved numerically to obtain the concentration profiles and mean concentration of unreacted sulfur dioxide in corn kernel as a function of time.

Materials and Methods

Materials

Flint corn, Cargill T-42 hybrid, provided by INTA Pergamino (Buenos Aires) was used in this work. The grains were harvested with a moisture content of 13.51 g water/100 g dry solid and stored at -18°C before their use.

Corn grains used for tests were screened to obtain samples of more uniform size (fraction retained between screens 8.5–9.5 mm was used). Volumetric measurements of grains were performed using cyclohexane displacement. An average grain volume (20 kernels with harvested moisture content were used) was determined from these measurements, giving an equivalent spherical radius of 4.5 mm.

Steeping tests

The experimental methodology for determining sulfur dioxide concentration in corn kernels during steeping included the following steps.

Preparation of the steeping solution by dissolution of sodium bisulfite in distilled water. The bisulfite was added to give a sulfur dioxide concentration of 0.25 g/100 g (the pH of the solution was 4.1).

Temperature adjustment of the steeping solution to predetermined values: 25, 35, 45 and 55°C .

Weighed samples (about 10 g) were placed in separate flasks, hermetically sealed, and totally filled with steepwater to minimize oxidation of sulfur dioxide. The flasks were immersed in a constant-temperature bath (Vicking, Model Dubnoff-Senedic) controlled $\pm 0.5^\circ\text{C}$ of the testing temperature. The grains were agitated at a rate of 70–80 rpm to reduce the mass transfer resistance of sulfur dioxide in steepwater.

Determination of unreacted sulfur dioxide in corn kernels. At regular intervals the flasks were removed from the bath for sulfur dioxide determination (the procedure is described below). The amount of unreacted sulfur dioxide was expressed on dry weight basis, as mg of sulfur dioxide per kg of dry corn kernels.

The saturation concentration of sulfur dioxide for each steeping temperature was obtained after 50 h of steeping. After that, sulfur dioxide in corn kernels was considered to have reached near-equilibrium with steeping solution.

Reaction kinetics of sulfur dioxide

Samples of ground corn were used to evaluate sulfur dioxide loss by chemical reaction. The grains were ground in a coffee-type grinder and the product placed in an Erlenmeyer flask to which a weighed amount of aqueous solution containing 0.185 g/100 g of sulfur dioxide (the solution was previously heated to the temperature of the kinetics tests) was added. A subsample was separated

to determine the moisture content of the ground corn. This was done according to the AOAC Method no. 14.014 (AOAC, 1980). After that, subsamples of about 15.0 g were transferred to tared small screw-tap culture tubes with Teflon-lined caps to prevent sulfur dioxide loss. The tubes were weighed and stored in a convection air-forced oven for different time intervals one to four days, approximately, depending on the storage temperatures investigated, which were 25, 35, 45 and 55 °C. Simultaneously, one of the samples was separated and used to evaluate the initial concentration of sulfur dioxide in the ground corn. The reaction rate was followed until the concentration of sulfur dioxide was about 25% of the initial concentration.

The tubes were periodically removed from the oven for sulfur dioxide determination. All the tests were performed in triplicate and average was taken (standard deviation ± 30 mg/kg). Control tubes containing only the sulfurous acid solution were used to verify that the tubes with Teflon-lined caps did not allow escape of the sulfur dioxide from solution.

Analytical method

Total sulfur dioxide content in corn samples was determined by modification of the method of Ponting and Johnson (1945). The method combines an extraction procedure at room temperature with titration by iodine. In corn, the method allowed to detect a limit of about 1 mg/kg. The method was used to evaluate sulfur dioxide content in corn samples, ground or whole, obtained from the respective reaction kinetics and steeping tests. The

kernels resulting from the steeping tests were first ground in a Waring-type grinder for three minutes in 250 mL of aqueous solution of NaCl, 20 g/100 g, to which 10 mL of 0.5 mol/L tartrate buffer at pH = 4.5 (tartaric acid and NaOH) was added. The samples resulting from the kinetics tests were also blended by mechanical agitation for three minutes with a solution similar to one described above. The slurry was alkalized by adding 2 mL of NaOH solution 1 mol/L; after 45 min, NaCl solution (20 g/100 g) was added to complete the volume to 500 mL. The slurry was then filtered through four-fold gauze. A volume of 100 mL of filtrate was pipetted into Erlenmeyer flask and acidified with 2 mL of 6 mol/L hydrochloric acid. One milliliter of 1 g/100 mL starch solution was added and treated rapidly and continuously with iodine solution 0.1 mol/L.

A blank was prepared by blending 10 g of ground corn with 100 mL of distilled water, 10 mL of buffer solution, 250 mL of NaCl solution and 1 mL of formaldehyde solution (38 g/100 g). Sulfur dioxide content was calculated as mg of sulfur dioxide per kg of corn on a moisture-free basis.

Results and Discussion

Estimation of the reaction rate constant

In **Fig. 1**, it is observed that sulfur dioxide loss by chemical reaction follows a first order kinetics. The reaction rate constants and intercepts values, c_i , for each of the temperature investigated are given in **Table 1**, together with the correlation coefficients obtained from the linear

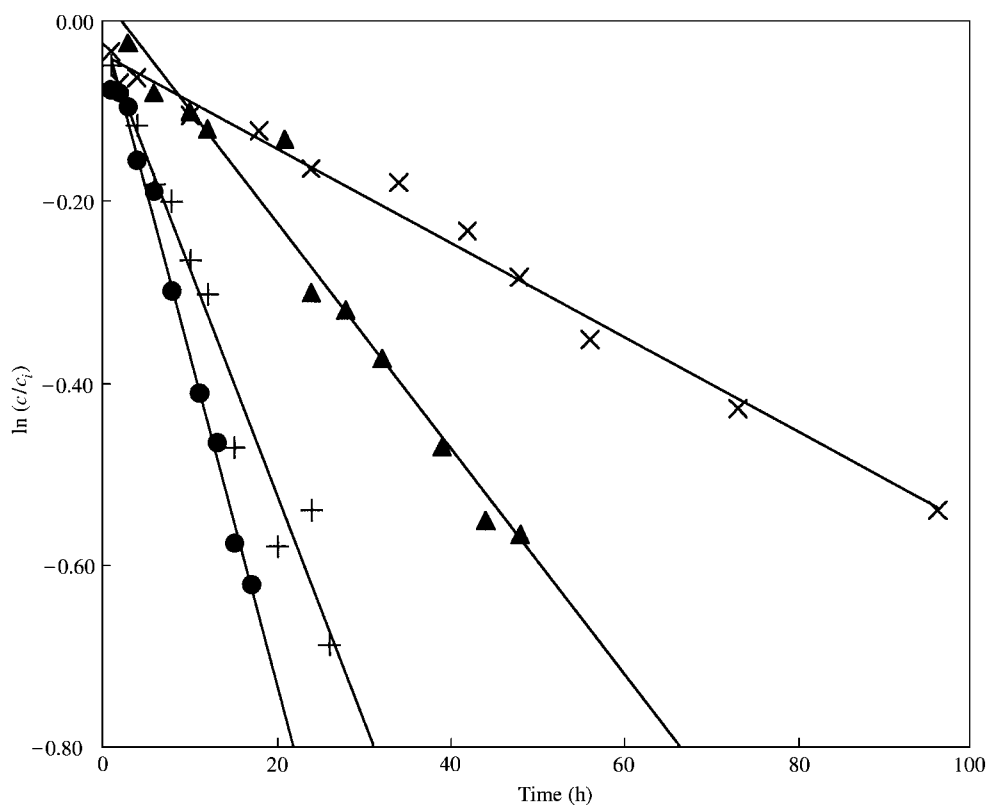


Fig. 1 Reaction kinetics of sulfur dioxide in flint corn (ground) at different temperatures: (x) 25 °C; (▲) 35 °C; (+) 45 °C; (●) 55 °C

Table 1 Intercept values, c_i , and first order rate constants, k , for SO_2 reaction in flint corn at different temperatures

Temperature (K)	c_i (mg/kg)	$k \times 10^7 \text{ s}^{-1}$	Correlation coefficient
298	1382	1.49 ± 0.22	0.988
308	1366	3.28 ± 0.19	0.964
318	1394	6.88 ± 0.38	0.960
328	1387	9.78 ± 0.47	0.991

regression analysis. The intercept values were slightly lower than the initial concentration of sulfur dioxide in the ground corn (1425 mg/kg).

The first-order rate constants in **Table 1** are one to two orders of magnitude larger than that reported by Eckhoff and Okos (1986) for yellow dent corn. Such differences in the reactivity of sulfur dioxide may be attributed in part to differences in composition of kernel endosperm. As flint corn has a larger proportion of corneous endosperm than dent corn, the amount of protein present in the former is also much larger (Robutti, 1980). Haros and Suárez (1998) found that for two local hybrids of dent and flint corn, the protein contents were 6.2 g/100 g and 9.3 g/100 g, respectively. Apparently, not only the amount but also the type of proteins present in the endosperm is responsible for the characteristics of the kernel. Robutti *et al.* (1997) found that corn endosperm hardness is influenced by the different proportion of zein fractions and that the zein compositions of corneous and floury endosperm are different.

Another factor that may contribute to the disappearance of sulfur dioxide is the moisture content of the grains. Eckhoff and Okos (1986) reported a 3-fold increase in the reaction rate of sulfurous acid when the moisture content of corn grains was increased from 13 g/100 g to 27 g/100 g, dry basis. A similar effect was also observed during the kinetics studies of sulfur dioxide loss in various vegetables (Legault *et al.*, 1949). In all cases, a marked increase in the rate of disappearance of sulfur dioxide with moisture content was observed. In the present study, the reaction rate constants were evaluated at moisture content of 69 g/100 g which, on average, corresponded to the moisture content reached by corn kernels at the end of the steeping process.

It is interesting that the reaction rate constant of sulfur dioxide in corn was several orders of magnitude less than sulfite oxidation in aqueous solution. Some volatile compounds such as alcohol and aromatic compounds have been shown to inhibit sulfite oxidation (Schreoter, 1966). Maga (1978) has determined a wide variety of volatile compounds in corn and other cereals, many of which may act as inhibitors of sulfite oxidation.

To evaluate the effect of temperature on the first-order rate constant, the values of k given in **Table 1** were correlated by Arrhenius equation and the activation energy calculated by linear regression. The resulting value was 60.5 kJ/mol, which is less than the value of 85.6 kJ/mol reported by Bolin and Boyle (1972) for dried apple and markedly different from the activation energy

of 163 kJ/mol reported by Legault *et al.* (1949) for dried vegetables.

Estimation of the diffusion coefficient

The diffusion coefficient of sulfur dioxide in corn for the different steeping temperatures investigated was calculated as follows. Equations [1] to [4] were numerically solved by finite differences method. The resulting equations were programmed on a digital computer and the value of D was calculated by non-linear regression method. The optimization technique varies D until the sum of squares of deviation between observed and estimated values of sulfur dioxide mean concentrations in corn kernels are minimum. The estimated mean concentration of unreacted sulfur dioxide at each time interval was calculated by numerical integration of the corresponding sulfur dioxide concentration profiles obtained from the numerical procedure. The concentration of sulfur dioxide was expressed as mg of sulfur dioxide per kg of corn (dry basis).

In **Fig. 2**, the results of the calculation procedure for the four steeping temperatures investigated are compared. It is observed that when absorption took place at the temperatures of 25, 35 and 45 °C the model predicts lower concentrations of sulfur dioxide than the experimental values during the initial steeping. This may be due to a phenomenon other than diffusion. In fact, the water absorption by some cereal grains is accelerated by the presence of natural capillaries and structural cracks in the kernels (Becker, 1960).

The values of D together with the equilibrium concentrations of sulfur dioxide experimentally determined are reported in **Table 2**. The diffusion coefficient increased from $1.17 \times 10^{-11} \text{ m}^2/\text{s}$ at 25 °C to $5.18 \times 10^{-11} \text{ m}^2/\text{s}$ at 55 °C. Eckhoff and Okos (1990) found that the diffusion coefficient of gaseous sulfur dioxide in yellow dent corn at 25 °C was $2.7 \times 10^{-8} \text{ m}^2/\text{s}$, almost three orders of magnitude larger than the value determined here for the same temperature. The value of D reported by Eckhoff and Okos (1990) is within the range of diffusivities for bulk diffusion of a gas in a liquid, i.e. at 25 °C, the diffusion coefficient of sulfur dioxide in water is $1.7 \times 10^{-9} \text{ m}^2/\text{s}$ (Schreoter, 1966). Several factors may contribute to the differences above mentioned including the structural differences between dent and flint corn. Flint-type kernels contain higher concentration of starch around the periphery of the endosperm than in the center, giving the endosperm hard external layers. The hardness of the endosperm is the result of the strong compactation of the starch granules in comparison to the loosely held granules of the floury endosperm (Robutti, 1980). Also, the ratio of corneous to floury endosperm for dent corn is less than for flint corn. Based on these considerations it must be expected that flint corn is more impervious to the absorption of substances than dent corn. In fact, Haros *et al.* (1995) found that the rate of water absorption for flint corn was much lower than for dent type.

The large differences in the rate of sulfur dioxide uptake by corn grains in comparison with other food products

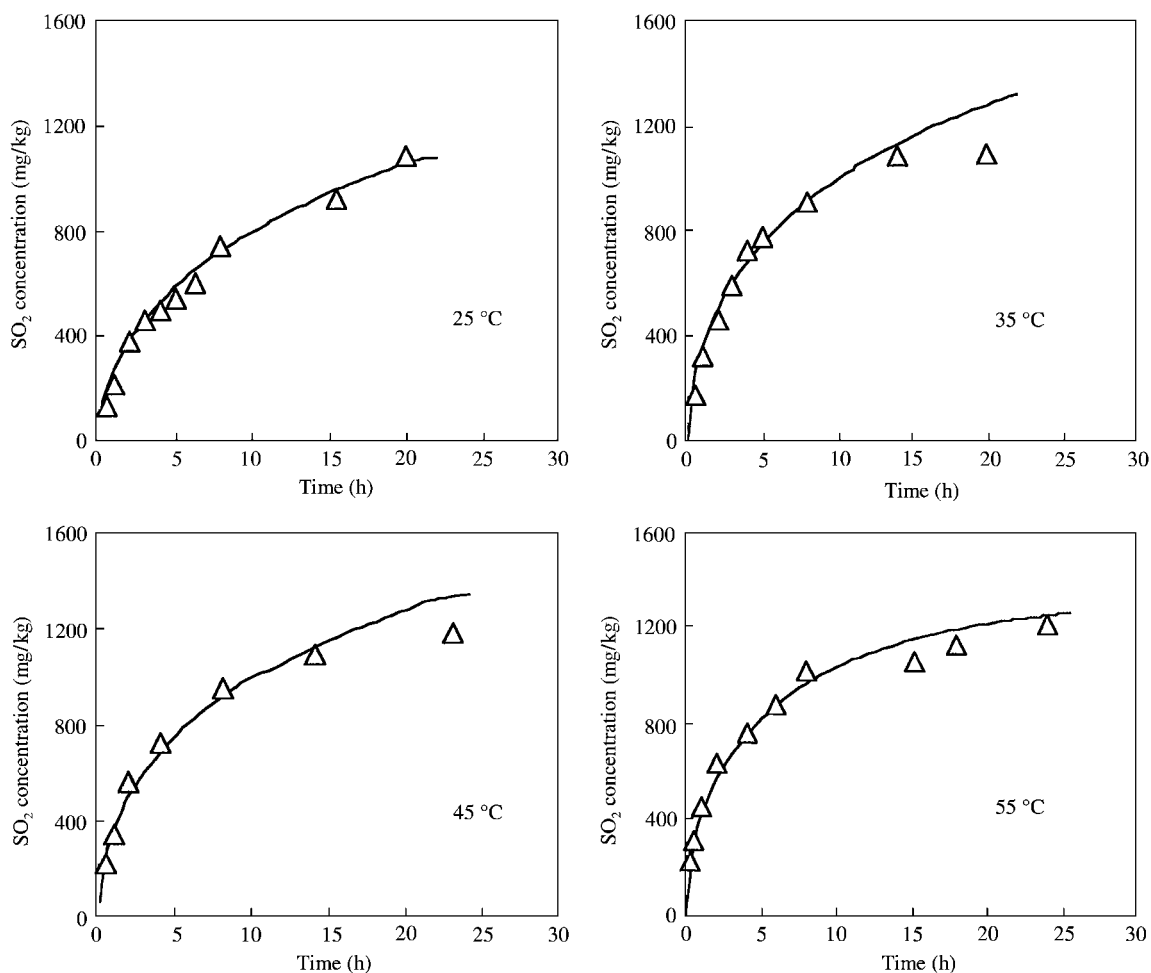


Fig. 2 Change of unreacted sulfur dioxide concentration in corn kernels at different steeping temperatures: (Δ) experimental; (—) predicted

Table 2 Equilibrium concentrations, c_e , and diffusion coefficients, D , of SO_2 in flint corn during steeping

Temperature (K)	c_e (mg/kg)	$D \times 10^{11}$ (m^2/s)
298	1900	1.17 ± 0.13
308	1800	2.05 ± 0.17
318	1500	3.12 ± 0.23
328	1350	5.18 ± 0.11

were noteworthy. Rodríguez and Zaritzky (1986) found that the diffusion coefficient of sulfur dioxide in potatoes was $1.08 \times 10^{-9} \text{ m}^2/\text{s}$ at 25°C , quite similar to the value of the diffusion coefficient of sulfur dioxide in water. For the same temperature, a value of $D = 1.05 \times 10^{-8} \text{ m}^2/\text{s}$ for sulfur dioxide diffusing in apricots was reported (Rossello *et al.*, 1993).

The relationship between diffusion coefficient and temperature was established using Arrhenius equation. The logarithm of D versus the inverse of the absolute temperature, T , was correlated and the activation energy, E_D , for the diffusive process calculated by linear regression. The resulting equation was:

$$D(\text{m}^2/\text{s}) = 2.7289 \times 10^{-5} \exp\left(-\frac{3673.85}{T(\text{K})}\right) \quad \text{Eqn [5]}$$

From Eqn [5], the activation energy for the diffusive process of sulfur dioxide in flint corn was equal to 39.8 kJ/mol. The value of E_D for absorption of gaseous sulfur dioxide in dent corn was 30.9 kJ/mol (Eckhoff & Okos, 1990); the discrepancy with other food products is even larger. For sulfurous acid diffusing in apricots, Rossello *et al.* (1993) found $E_D = 78.3 \text{ kJ/mol}$.

Comparing with the diffusion process of sulfur dioxide in water, which gives an activation energy of 27.9 kJ/mol (Perry & Chilton, 1973), it is possible to conclude that the mechanism of sulfur dioxide migration in corn differs from the relatively simple water-sulfur dioxide system. Factors such as moisture content variation and swelling of corn kernels during the steeping process which were not taken into consideration in the diffusional model, might have an important effect on the mechanism of sulfur dioxide diffusion. In fact, the rate of gaseous sulfur dioxide by corn was found to increase as the moisture content of the grain increased (Eckhoff & Okos, 1990). During steeping, the moisture content of corn kernels may vary from an initial value of about 13 g/100 g to 70 g/100 g, dry basis (Haros *et al.*, 1995), that may cause dramatic changes in the structure of the kernels. This will vary from a relatively closed and rigid structure at low moisture levels, where the mobility of the polymeric chains is reduced and water

molecules are strongly influenced by them, to another more open structure where water molecules are mainly surrounded by other water molecules and the effect of the substrate is considerably reduced. Under such circumstances, it is expected that sulfur dioxide migration will pass through various stages, since a first one, mainly influenced by the structure of the kernel, to the relative simple mechanism of sulfur dioxide diffusion in liquid water.

Conclusions

A mathematical model based on the mass transfer equation for simultaneous diffusion and reaction in porous medium was proposed to describe the absorption kinetics of sulfur dioxide in corn kernels. The proposed model is able to simulate sulfur dioxide uptakes by corn kernels in bisulfite solutions and the effect of the operating variables during the steeping process.

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