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## Modeling and Simulation of Wheat Grains Ozonation

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### Abstract

In order to improve the ozonation process efficiency, reliable modeling and simulation tools are needed. In this study, wheat grains were exposed to ozone gas for 45 min at an applied dose of 3.5 g O<sub>3</sub>/kg of wheat in a semi-industrial fixed-bed reactor. The consumed and the residual ozone were measured continuously. These values were fit, at a first step, to a two consecutive reactions kinetic model. A second part of our work led us to develop and assess a new simplified one reaction model. Cuticle layer hypothesis was later adopted to improve our original model. Using Matlab<sup>®</sup>, the reactions kinetics and mass transfer coefficients of these models were determined. Our kinetic model predicted the experimental data with high accuracy. In addition, a computational fluid dynamics (CFD) model was developed in COMSOL<sup>®</sup> v.5.3a in order to study the gas flow inside the reactor. This new approach is a first step to develop a comprehensive coupled model involving the ozonation reactions as well as the ozone diffusion in a porous medium.

*Key words:* Ozone, wheat grains, ozonation process, mathematical modeling, reaction kinetics, simulation, semi-industrial scale, CFD.

### Introduction

Ozone is a highly reactive oxidizing agent and it is also recognized by U.S. Food and Drug Administration (FDA) as GRAS (i.e. Generally Recognized as Safe) in 1997. The major advantage of ozone, compared to other oxidizing chemical agents, is that it finally breaks down into oxygen and leaves no residue of hazardous chemicals (McDonough et al., 2011). Ozone, therefore, has

found increasing applications in many fields. The use of ozone in food processing was widely reviewed in the literature (Cullen et al., 2009). Among these applications, gaseous ozone has been studied as a treatment for cereal grains (Tiwari et al., 2010). However, due to its strong oxidizing properties, ozone is not universally beneficial. Ozone can promote molecular alterations and/or oxidation of chemical constituents (i.e. proteins, lipids, minerals) present in food (Sandhu et al., 2011). Gozé et al. (2017) evaluated the effects of gaseous ozone on the molecular properties of wheat grain proteins and their consequences on the bread-making quality of the resulting flours (Gozé et al., 2017). The results demonstrate a reduction in the Sodium dodecyl sulfate (SDS) solubility of the wheat prolamins and an increase in the compactness of the protein polymers which leads to important modification in the rheological properties (i.e. increase in the tenacity and a great limitation of the extensibility) of the flours and/or dough obtained. Therefore to optimize ozonation treatments, it is necessary to understand the factors which govern the reaction. In other words, ozonation of foods requires a reliable model which should be able to incorporate the various process parameters and then predict appropriate treatment conditions to achieve desirable results.

According to our best knowledge, although many works have been conducted on the ozonation treatment of grains, there is still a limited number of studies on the mathematical modelling of this reaction. Lignocellulosic biomass ozonation was numerically studied. Recently, Bhattarai et al. (2015) introduced an original model for predicting ozone lignin degradation. This model, basically developed from the work of (García-Cubero et al., 2012), includes an outer mass transfer barrier or “cuticle” region where ozone mass transport is reduced in proportion to the mass of unreacted substrate (Bhattarai et al., 2015). The model presented by García-Cubero et al. (2012) did not take into account the residual insoluble lignin that remained unaffected even with a prolonged ozone exposure as proved by the experimental results.

In this study, three models of ozonation reaction were presented. Each model was fit to experimental ozone consumed and the reaction kinetics were estimated. The effectiveness of each model was also analyzed. The simulation is an indispensable tool to understand the effects of operating conditions and to evaluate the reactor performance, which could prove to be very helpful to improve the process. For this reason, a CFD (Computational Fluid Dynamics) model was developed to simulate the ozone flow behavior in a fixed bed reactor.

## **Material and Methods**

### **Wheat Grains Ozonation:**

6% (w/w) of water was added to 10 kg wheat (CREEK, Agora, France) and homogenized for 20 min. The grains mass was then fed into a cylindrical fixed-bed reactor and ozonized during 45 min. The operating pressure was maintained at 0.5 bar. Ozone was fed into the bottom of the

reactor at a flow rate of 2.0 m<sup>3</sup> NTP. h<sup>-1</sup>. An ozone generator (OZAT<sup>®</sup> CFS-2G, Ozonia, France) was used to produce the ozone at a concentration of 90 gO<sub>3</sub>.m<sup>-3</sup> NTP from oxygen (99.5%, Messer, France) as a feed gas. Inlet and outlet ozone concentrations were measured and recorded (Ozone Analyzer BMT 964) during the test. The ozone off gas was destroyed by a thermal destruction unit at 350 °C. The results are the average of three independent experiments.

### Ozonation Modeling:

In this study, the consumed ozone was fit to three different kinetic models. The first model is based on the model developed by Garcia-Cubero et al. (2012), which supposed the oxidation of the substrate by two consecutive steps. Secondly, a simplified one global reaction model was originally proposed in this paper. It was hypothesized that the ozone will react principally with one major substrate in wheat grain (more precisely: the protein) and all other secondary reactions were neglected. As previously reported (Gozé et al., 2017, 2016), the starch remains untouched during the ozonation treatment, and the proteins were the major constituents of wheat grain affected by the ozone reaction. However, these two models do not take into account the residual substrate that remained untouched by the ozone. They predict that prolonged ozone treatment removes completely the substrate which contradicts experimental data. As a consequence, a third model, which takes into account the residual substrate, was proposed. The principle of this model was initially published by Bhattarai et al. (2015), including an outer mass transfer barrier or “cuticle” layer where ozone mass transport is reduced in proportion to the mass of remaining substrate. The following simplification assumptions have been considered:

- The wheat grains are assumed as a continuous homogenous porous medium.
- Ozone decomposition reactions are negligible.
- The ozone consumption is considered as irreversible and as a second-order reaction.
- The ozone reactions are slow compared to mass transport in gas and solid phases. The ozone concentration in the gas phase is equal to the inlet ozone concentration. As a consequence, the equations (5, 11, and 18) were ignored to simplify the models.

The reaction equations and their kinetic expressions are listed below, based on early results detailed in results and discussion.

1- Two consecutive reactions model:



$$R_1 = k_1 [O_{3S}] \cdot [S_1] \quad [3]$$

$$R_2 = k_2 [O_{3S}] \cdot [S_2] \quad [4]$$

$$\varepsilon \frac{\partial O_{3G}}{\partial t} + u \frac{\partial O_{3G}}{\partial z} = -K_{OGS} a_p (O_{3G} - \frac{\rho_s O_{3S}}{1-\varepsilon}) \quad [5]$$

$$\frac{\partial O_{3S}}{\partial t} = \frac{K_{OGS} a_p}{(1-\varepsilon)} \left( \frac{O_{3G}}{\rho_s} - O_{3S} \right) - \alpha_1 R_1 - \alpha_3 R_2 \quad [6]$$

$$\frac{\partial S_1}{\partial t} = -\alpha_2 R_1 \quad [7]$$

$$\frac{\partial S_2}{\partial t} = \alpha_3 R_1 - \alpha_5 R_2 \quad [8]$$

2- One global reaction model:

$$\alpha_1 O_{3S} + \alpha_2 S \rightarrow P \quad [9]$$

$$R = k [O_{3S}] \cdot [S] \quad [10]$$

$$\varepsilon \frac{\partial O_{3G}}{\partial t} + u \frac{\partial O_{3G}}{\partial z} = -K_{OGS} a_p (O_{3G} - \frac{\rho_s O_{3S}}{1-\varepsilon}) \quad [11]$$

$$\frac{\partial O_{3S}}{\partial t} = \frac{K_{OGS} a_p}{(1-\varepsilon)} \left( \frac{O_{3G}}{\rho_s} - O_{3S} \right) - \alpha_1 R \quad [12]$$

$$\frac{\partial S}{\partial t} = -\alpha_2 R \quad [13]$$

3- One global reaction with cuticle layer model:

$$\alpha_1 O_{3c} + \alpha_2 S_c \rightarrow P \quad [14]$$

$$\alpha_1 O_{3S} + \alpha_2 S_S \rightarrow P \quad [15]$$

$$R_c = k [O_{3c}] \cdot [S_c] \quad [16]$$

$$R_S = k [O_{3S}] \cdot [S_S] \quad [17]$$

$$\varepsilon \frac{\partial O_{3G}}{\partial t} + u \frac{\partial O_{3G}}{\partial z} = -K_{OGC} a_p (O_{3G} - \frac{\rho_s O_{3c}}{1-\varepsilon}) \quad [18]$$

$$\frac{\partial O_{3c}}{\partial t} = \frac{K_{OGC} a_p}{(1-\varepsilon) \lambda_c} \left( \frac{O_{3G}}{\rho_s} - O_{3c} \right) + \frac{K_{OCS} a_p}{(1-\varepsilon) \lambda_c} (O_{3S} - O_{3c}) - \alpha_1 R_c \quad [19]$$

$$\frac{\partial S_c}{\partial t} = -\alpha_2 R_c \quad [20]$$

$$\frac{\partial O_{3S}}{\partial t} = \frac{K_{OCS} a_p}{(1-\varepsilon)(1-\lambda_c)} (O_{3C} - O_{3S}) - \alpha_1 R_S \quad [21]$$

$$\frac{\partial S_S}{\partial t} = -\alpha_2 R_S \quad [22]$$

$$K_{OGC} a_p = \frac{D_{O_3} a_p}{d_p} \frac{[S_c]}{([S_{c0} - S_c] \lambda_c + [S_c])} \quad [23]$$

$$K_{OCS} a_p = \frac{D_{O_3} a_p}{d_p} \frac{[S_c]}{[S_{c0}] \lambda_c} \quad [24]$$

Where  $O_{3G}$ ,  $O_{3S}$  and  $O_{3C}$  are the ozone treatment rate in gas, solid and cuticle phases respectively (g  $O_3$ / kg of wheat).  $P$  is the final reaction product (g/ kg of wheat).  $\alpha_1$ ,  $\alpha_2$ ,  $\alpha_3$ ,  $\alpha_4$ ,  $\alpha_5$  are stoichiometric ratios (dimensionless). For two reactions model,  $R_1$  and  $R_2$  are elementary bimolecular reaction rates (g  $O_3$ / s. kg of wheat) for ozone reacting with the first  $S_1$  and second  $S_2$  substrate (g of substrate/ kg of wheat) respectively. For one reaction cuticle model,  $R_C$  and  $R_S$  are the reaction rates for ozone reaction with cuticle layer substrate  $S_C$  and solid phase substrate  $S_S$  respectively.  $k_1$ ,  $k_2$  and  $k$  are kinetic rate constants (kg of wheat / s. g of substrate).  $\varepsilon$  is the void fraction (dimensionless),  $u$  is the superficial velocity in the porous phase (m/s),  $K_{OGS}$  is an overall mass transfer coefficient between the gas and solid phase ( $s^{-1}$ ),  $a_p$  is the interfacial area of the solid phase per unit volume of packed bed ( $m^2/m^3$ ).  $K_{OGC}$  is the mass transfer coefficient from the gas phase to the cuticle phase ( $s^{-1}$ ) and,  $K_{OCS}$  is the mass transfer coefficient from the cuticle phase to the solid phase ( $s^{-1}$ ).  $\lambda_c$  is the fraction of the particle that makes up the cuticle layer (dimensionless),  $D_{O_3}$  is the diffusion coefficient of ozone inside the cuticle layer ( $m^2/s$ ) and  $d_p$  is the diameter of the wheat straw particle (m).

The model parameters were estimated using the Levenberg–Marquardt optimization method (lsqnonlin function in Matlab<sup>®</sup> software) with the stiff ordinary differential equation solver, ODE15s. The minimize criterion was defined as the sum of the squares of the differences between the experimental values of consumed ozone and the model predictions.

$$Z = \sum_i (O_{3 \text{ consumed exp}} - O_{3S \text{ model}})^2 \quad [25]$$

In the case of the cuticle model the objective function was defined as:

$$Z = \sum_i (O_{3 \text{ consumed exp}} - [(1 - \lambda_c)O_{3S \text{ model}} + \lambda_c \times O_{3C \text{ model}}])^2 \quad [26]$$

In order to reduce the possibility that the optimization procedure stops on a local minimum, the optimization algorithm was tested with different initial values, therefore, a random number generator was programmed to select the initial values. 5,000 iterations were tested for each model.

The optimized parameters 95% confidence interval was calculated using *nlparci* function in Matlab<sup>®</sup> software.

### Computational Fluid Dynamics (CFD) Model:

Based on a finite element method, the three-dimensional numerical model was solved in COMSOL<sup>®</sup> Multiphysics 5.3a to study the ozone flow behavior in the reactor. The free and porous media flow physics in the CFD module was used. This module integrates the functionality of the laminar flow physics with the Brinkman equations physics. The gas flow in the empty spaces in the reactor is modeled by the Navier–Stokes equations:

$$\rho (u \cdot \nabla u) = \nabla \cdot [ -P\mathbf{I} + \mu (\nabla u + (\nabla u)^T) ] \quad [27]$$

$$\rho \cdot \nabla u = 0 \quad [28]$$

Where  $\rho$  is the fluid density ( $\text{kg}\cdot\text{m}^{-3}$ ),  $u$  is the vector velocity in the open reactor outside the packed bed ( $\text{m}\cdot\text{s}^{-1}$ ),  $P$  is the dynamic pressure (Pa),  $\mathbf{I}$  is the unit tensor,  $\mu$  is the dynamic viscosity (Pa.s) and  $T$  is the absolute temperature (K). Within the grains mass, the Brinkman equations describe the gas flow:

$$\frac{\rho}{\varepsilon_p} \left( (u \cdot \nabla) \frac{u}{\varepsilon_p} \right) = \nabla \cdot \left[ -P\mathbf{I} + \frac{\mu}{\varepsilon_p} (\nabla u + (\nabla u)^T) \right] - \left( \frac{\mu}{\kappa_b} + \beta_F |u| \right) u \quad [29]$$

$$\rho \cdot \nabla u = 0 \quad [30]$$

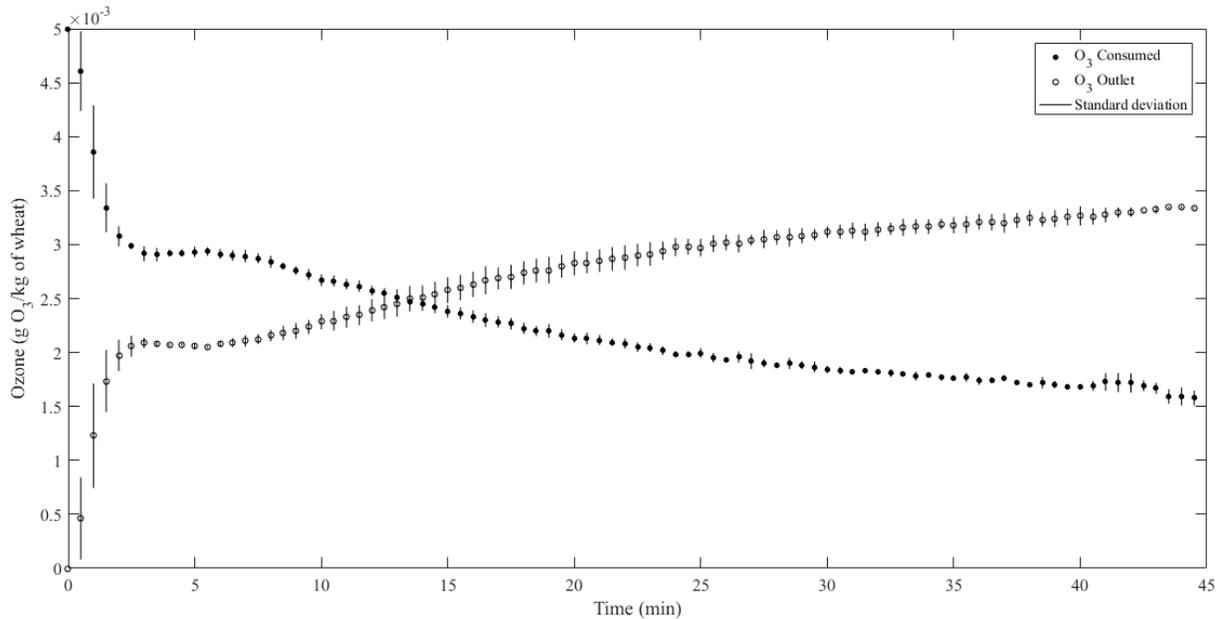
Where  $u$  is the superficial velocity ( $\text{m}\cdot\text{s}^{-1}$ ) in the packed bed,  $\varepsilon$  is the grains mass porosity,  $\kappa_{br}$  is the permeability ( $\text{m}^2$ ), and  $\beta_F$  is the Forchheimer drag coefficient ( $\text{kg}/\text{m}^4$ ) (Whitaker, 1996). The boundary conditions on the reactor surfaces are: no slip on the reactor walls,  $u = u_{\text{inlet}}$  ( $10 \text{ m}\cdot\text{s}^{-1}$ ) no normal stresses act on the outlet flow. The flow is laminar in the reactor. The pressure drop across the grains mass was neglected.

## Results and Discussion

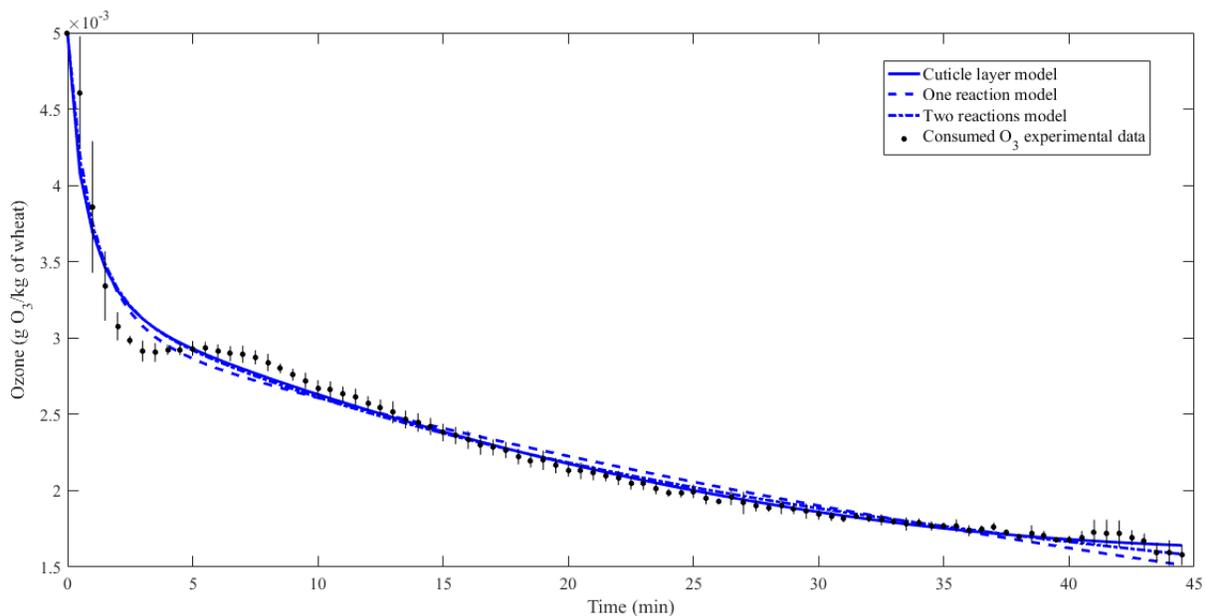
### Modelling Study:

The consumed and outlet ozone were measured continuously during the treatment, the experimental values are presented in figure (1). The points present instantly the quantity of applied ozone per kilogram of wheat. The results showed a high ozone consumption at the beginning of the reaction. This consumption dropped quickly to reach a more stable phase where it only

decreased gradually. It was hypothesized that there are two reactions. The ozone seems to react first rapidly with available and easily accessible substrate (i.e. protein of damaged grains for example) and then slowly with the whole grains substrate.



**Figure 1.** The experimental consumed (●) and outlet (○) ozone with confidence intervals (|)



**Figure 2.** Experimental consumed O3 with confidence intervals and numerical fitted data using the three models

The consumed ozone was fitted to three different models using Matlab<sup>®</sup> R2016b to determine the unknown parameter values. It was observed that the optimal parameter values were a function of the initial guesses. In order to overcome this problem, the Matlab<sup>®</sup> program completed 5000 iterations using sequentially different initial guesses to find the minimum of the objective function (Z). The smallest Z value obtained through the iterative process indicated the global minimum and yielded the best-fit parameter estimates. The stoichiometric parameters  $\alpha_1$ ,  $\alpha_4$  are reference parameters, their values were taken as 1. The reactions kinetics and mass transfer coefficients of the mathematical models to predict the consumed ozone were determined. For the three models, the simulation results were in good agreement with the experimental data. The best-fit parameter estimates are presented in Table I, and the resulting fit of the three models to the experimental data are shown in figure (2).

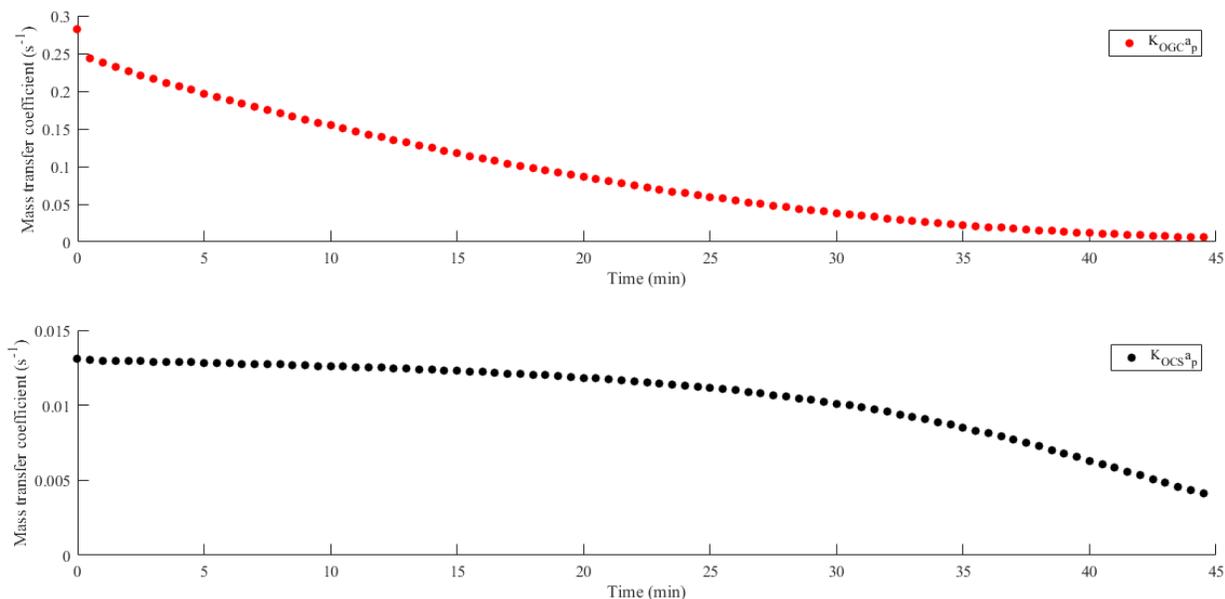
The Garcia-Cubero et al. (2012) model was originally published to describe the ozonation of lignin in lignocellulosic materials where the lignin degradation takes place in two steps. This model was the first model applied to our results in this study. The estimated parameters of two reactions model show that the second reaction is about 155 times faster than the first one which will practically determine the rate of ozonation reactions. For this reason, and due to the high 95% confidence intervals on the fitted parameters, a one reaction model was then proposed and successfully programmed. However, as well as the two reactions model, the one reaction model does not consider the residual substrate which remains intact even after a prolonged treatment. As a consequence, a third model, based on the cuticle hypothesis, was developed. In this model, the mass transfer coefficient between gas phase and wheat is gradually slowed down thanks to an outer cuticle layer. The last model gives the best results according to objective function Z value along with confidence intervals values. From a mathematical point of view, this modified one reaction cuticle layer model is enough to describe with high accuracy the ozonation of wheat grains, even though the real chemistry phenomena might be more complicated.

Mass transfer coefficients between gas phase and the cuticle outer layer and between cuticle and the internal layers of wheat are presented in figure (3). The results showed that the mass transfer between the gas phase and the outer layer of wheat controls the global ozone transfer in opposition to the interlayer transfer which remained relatively constant during the first 25 minutes of the treatment. Those results are in line with a decrease of the reactivity caused by a progressive transfer limitation of the outer layer. Enhancing gas-outer layer mass transfer coefficient will improve the overall ozone transfer to the substrate. It is now of common knowledge that adding a thin layer of water over the wheat surface will highly increase the gas transfer efficiency, see for example (Cogo et al., 1999).

**Table I.** MODELS BEST FIT PARAMETER ESTIMATES, THE 95% CONFIDENCE INTERVALS AND THE DIFFERENCE (%) FROM THE BEST ESTIMATES. THE MINIMUM VALUES OF THE OBJECTIVE FUNCTION ARE PRESENTED

Parameter (units)	Best-fit estimate <sup>♦</sup>	± 95% Confidence interval	%
<b>1- Two consecutive reaction model</b>			
$k_1 \left( \frac{\text{kg of wheat}}{\text{s.g of substrate}} \right)$	0.000182510091882581	0.0000080752510259	4.424550414
$k_2 \left( \frac{\text{kg of wheat}}{\text{s.g of substrate}} \right)$	0.028001484382880006	0.0150035097588981	53.58112289
$K_{OGS} a_p \text{ (s}^{-1}\text{)}$	0.000012615091716077	0.0008118002117550	6435.151087
$\alpha_2 \text{ (-)}$	49325.60653544618300	6239.1835183012000	12.64897475
$\alpha_3 \text{ (-)}$	16.58594650668911900	8.8667072860068000	53.45915762
$\alpha_5 \text{ (-)}$	9847.267352204062300	5262.9731576009400	53.44602690
Z value	0.000000622447544741		
<b>2- One global reaction model</b>			
$k_1 \left( \frac{\text{kg of wheat}}{\text{s.g of substrate}} \right)$	0.003084447633611548	0.0000052490356162	0.170177492
$K_{OGS} a_p \text{ (s}^{-1}\text{)}$	0.009646268421245774	0.0000061591740279	0.063850328
$\alpha_2 \text{ (-)}$	73209.62378386977100	26.673874313506500	0.036434929
Z value	0.00000077813020382		
<b>3- One global reaction with cuticle layer model</b>			
$k_1 \left( \frac{\text{kg of wheat}}{\text{s.g of substrate}} \right)$	0.002860297095135403	0.0000027361662138	0.095660210
$\frac{D_{O_3} a_p}{d_p} \text{ (s}^{-1}\text{)}$	0.013089005379067627	0.0000131797188107	0.100693051
$\lambda c \text{ (-)}$	0.046397738386247729	0.0000132132883316	0.028478303
$\alpha_2 \text{ (-)}$	87700.26810971219700	49.336476428405100	0.056255787
Z value	0.000000626662064529		

<sup>♦</sup> All digits are needed to reproduce the exact results

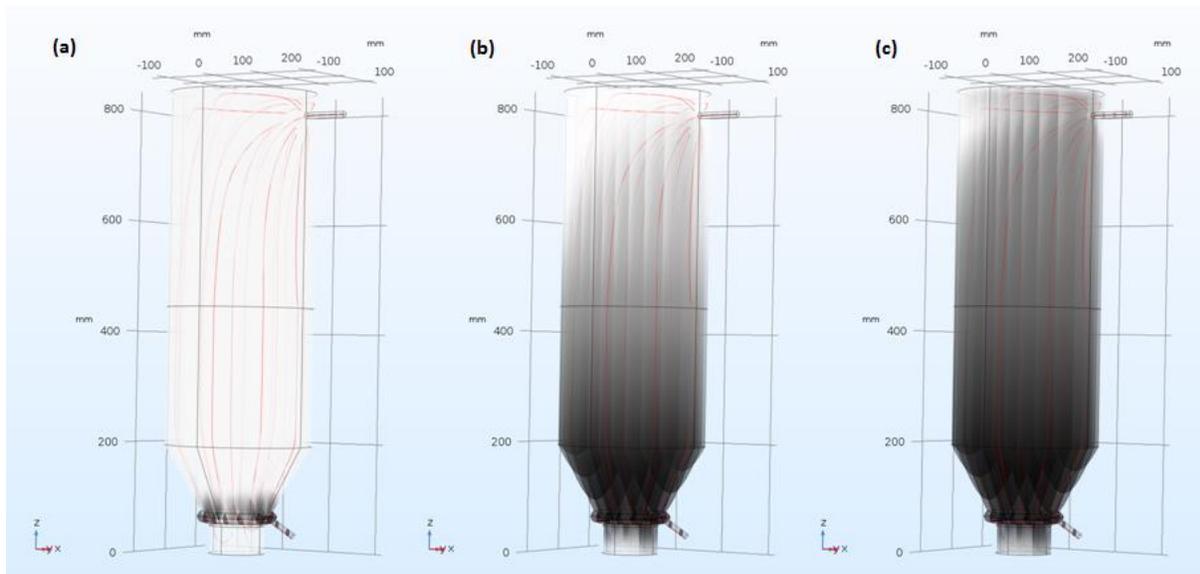


**Figure 3.** Mass transfer coefficients between gas phase and the cuticle ( $K_{OGC}^{a_p}$ ) and between cuticle and the internal layers of wheat ( $K_{OCS}^{a_p}$ ) as a function of ozonation time

### CFD Study:

To study the flow behavior of ozone inside the reactor, a CFD model was developed to obtain detailed flow fields in the reactor half filled with wheat. The operating parameters were incorporated to the CFD model. The overall real time simulated was 45 min. Figure (4) visualizes the ozone flow streamlines as well as the gas distribution inside the reactor as a function of time. According to these results, the gas was distributed relatively uniformly across the grains mass. The gas exit time was about 110 sec which was in good agreement with the experimental value. In order to simplify the previous ozonation models, it was supposed that the gas diffusion time is too small compared to the treatment time. However, the CFD model shows that this assumption was not very accurate. The ozone distribution time inside the reactor represents ~ 4.5 % of the entire treatment duration. Ozone diffusion needs to be considered to develop a more realistic model.

The CFD model shows that the zone under the inlet tube was not correctly exposed to gas (figure 4: b and c) which is in agreement with the experimental remarks (data not shown). Further optimization attempts will be necessary to improve the design of the existing reactor. The next step will be adding chemical reaction equations to complete the CFD model. The coupled model will describe completely the ozonation treatment and will be a very useful tool to understand and develop the process.



**Figure 4.** Velocity field inside the reactor (red lines). The black shading represents the moving gas phase distribution as a function of time (a: 1s, b: 50 s, c: 110 s).

## Conclusion

This work proposed three mathematical models and a CFD model to describe the ozonation of wheat grains in a fixed bed reactor. The results showed that the one reaction model was correctly fitted to the experimental data. In order to take into account the residual intact substrate for a prolonged ozonation treatment, the modified one reaction with cuticle layer is preferred. The CFD study revealed that the wheat was uniformly exposed to ozone, however, the gradual ozone diffusion inside the bed has to be considered in the future CFD model which will also include the reaction equations to model completely and optimize the ozonation treatment.

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