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## MODEL-BASED OPTIMIZATION OF PERLITE EXPANSION VIA A RESPONSE SURFACE METHOD (RSM)

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**Keywords:** Perlite expansion, dynamic modeling, response surface methodology (RSM), process optimization.

**Abstract.** *Conventional perlite expansion suffers certain well-known shortcomings compromising its viability and the adherence of expanded perlite to modern technical specifications for high-quality insulation materials. A new perlite expansion process has been designed and a vertical electrical furnace for perlite expansion has been constructed to overcome such drawbacks, with concurrent modeling studies (Angelopoulos et al., 2013<sup>[1]</sup>). Having already accomplished the production of various expanded perlite grades for many varying applications, the entire state space of product quality against key manipulated variables has also been explored, and ideal experimental condition ranges for process operation have also been identified (Angelopoulos et al., 2013<sup>[2]</sup>). Response Surface Methodologies (RSM) have a long track record of contribution to substantial improvements in many advanced chemical and material processing technologies. Their fundamental principle is the systematic exploration and statistical correlation of input (conditional) and output (response) variables with respect to interactions (if any) of the former and their combined effect (if any) on the latter – thus on end product quality. This paper focuses on using parametric sensitivity analysis results from the published furnace dynamic model as well as original experimental furnace results towards extracting a new three-dimensional RSM model which achieves an accurate correlation of both pivotal experimental variables (furnace temperature, air flowrate) with their combined effect on a known end-product quality metric (grain expansion factor): optimal condition ranges can thus be derived via bivariate quadratic polynomial fits for any plant and any raw perlite feed composition.*

### 1 INTRODUCTION

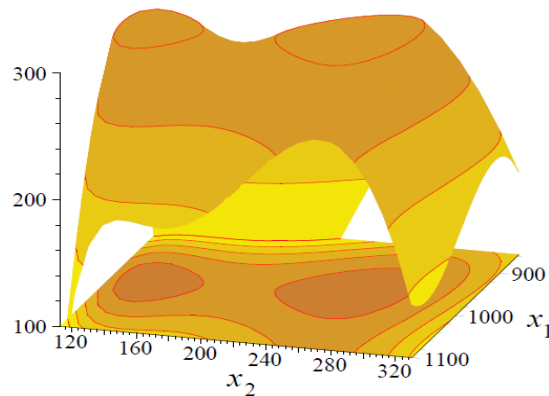
*First-principle process modeling* relies on constructing a mathematical model of key state variables and their static/algebraic or transient/dynamic interdependence as a function of system properties and external conditions. *Static or dynamic process simulation* thus occurs by means of computing operation points and/or dynamic state variable trajectories, thus validating models against available experimental data and designing new experiments. Verifying model credibility and applicability limits is therefore essential for industrial implementation purposes. *Systematic process optimization* transcends the scope of model validation and process routine operation and is concerned with identifying ideal production conditions via theoretically sound computational methodologies. Determining such optimal points may be *data-driven* if conducted via analyzing experimental measurements, or *model-based* when employing numerical algorithms operating on a (preferably validated) first-principle model. Hybrid variations of these two optimization method classes abound: empiricism enters either in model building (discounting first-principle understanding), in algorithmic structure (discounting mathematical rigor), or in both. Elaborating high-fidelity models may imply considerable effort before reliable optimization can be performed.

The *Response Surface Methodology (RSM)* encompasses statistical techniques for empirical model building which were originally developed to model experimental responses and then employed in numerical modeling<sup>[3]</sup>. The objective is to optimize a correlation metric of a technically critical state process response (output variable) which is a priori known or concurrently found to be affected by key experimental conditions (input variables)<sup>[4]</sup>. An input-output model requires coefficient matrix computation and possibly iterative structural modification<sup>[5]</sup>. Design of Experiments (DoE) coupled with RSM can achieve rapid process development under minimal cost. Selecting and implementing the most efficient optimization method for a given physicochemical process mainly depends on complexity, level of mechanistic understanding, experimental cost and pilot-plant data availability. In physical experiments, inaccuracy can be e.g. due to measurement errors while, in computer experiments, numerical noise is a result of incomplete convergence of iterative processes, round-off errors or the discrete representation of continuous physical phenomena; in all RSM methodologies, errors are assumed to be random.

Implementing RSM for process operation optimization can reduce the cost of expensive analysis methods (e.g. finite element method or CFD analysis) and their associated numerical noise. The problem can be approximated with smooth functions which improve the convergence of the optimization process as they reduce noise effects. Generally, the true structure of the relationship between the response (dependent) variable and the independent variables is unknown, hence the first step in RSM algorithms is to find a suitable mathematical approximation. The most common forms are smooth low-order polynomials (first- or second-order) requiring few parameters. For example, perlite expansion ( $y$ ) depends on volumetric air flow ( $x_1$ ) and furnace wall temperature ( $x_2$ ), and this experimentally known correlation can hereby be postulated to follow a bivariate (e.g. polynomial) function:

$$y = f(x_1, x_2) \quad (1)$$

The surface represented by the arbitrary bivariate function  $f(x_1, x_2)$  representing data is called a *response surface*. The response can be represented graphically, either in the three-dimensional space or as contour plots that help visualize the shape of the response surface. Contours are curves of constant response drawn in the  $x_1$ - $x_2$  plane keeping all other variables fixed; each contour corresponds to a particular height of the response surface (Fig. 1).



**Figure 1.** Three-dimensional surface and contour plots of a smooth and continuous bivariate model function.

## 2 PERLITE GRAIN EXPANSION TECHNOLOGY

Perlite is a natural occurring volcanic siliceous rock which consists mainly of amorphous silica (70-76 % wt.) but also contains smaller quantities of numerous other metal oxides ( $\text{Al}_2\text{O}_3$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{MgO}$ ). Perlite can be expanded from 4 to 20 times its original volume when heated at a temperature close to its softening point (700-1260 °C), due to the presence of 2-6% chemically bound water within its microstructure<sup>[1]</sup>. Conventional perlite expansion is usually accomplished by feeding ground, pre-sized perlite ore into a vertical furnace heated by a direct gas flame at its bottom end, thus directing forced air flow upwardly: ore particles are typically introduced into the hottest expansion chamber region, near the flame, at a temperature of 1450 °C<sup>[2]</sup>. During expansion, perlite acquires outstanding physical properties (e.g. low density, thermal and acoustic insulation) which render it suitable for numerous applications in the construction and manufacturing industries. The currently prevalent perlite expansion process is virtually the only globally reliable production method: although it has been in continuous use for more than 50 years, it remains a largely empirical industrial process, despite the rapid increase and proliferation of expanded perlite uses and applications over the past few decades. Its main technical problems include the increased heat losses due to the off-gas stream, leading to high energy consumption, but also the violent and poorly controlled heating of perlite which results in an expanded material with unfavourable mechanical properties, which adversely affect quality and limit the range of its applications.

To overcome these drawbacks, a new perlite expansion process has been designed and an NTUA pilot-scale production unit has been constructed, based on the concept of a vertical, electrically heated expansion furnace. The new vertical electrical furnace has been designed for maximizing experimental flexibility, in order to facilitate the adjustment of operating conditions versus raw material but also final product quality specifications. The new perlite expansion method allows the milder, gradual heating of perlite grains, as well as the variation of perlite grain residence time in the heating chamber. Experimental perlite expansion campaigns conducted on this pilot-scale production unit indicate a remarkable improvement as well as a definite operating cost reduction in expanded perlite properties compared with conventional processing. Hence, the multi-parametric mathematical investigation of the process system thermal behavior and the identification of the effect of all crucial operational parameters on perlite expansion dynamics is an endeavor of evident importance which must guide the eventual optimization of the entire pilot-scale experimental facility as well as every subsequent production-scale process.

### 3 THE MATHEMATICAL MODEL

The dynamic mathematical model previously developed <sup>[1][2]</sup> concentrates on the momentum and energy balance description of single-grain expansion in the vertical electrical furnace, at a microscopic and a macroscopic level. Microscopic mathematical analysis here refers to the investigation of internal particle temperature and steam bubble pressure evolution, as the interplay and rates of both these phenomena governs perlite grain expansion. Macroscopic mathematical analysis refers to particle motion (force balance) and air temperature distribution. These transport phenomena are analyzed via a nonlinear system of algebraic and ordinary differential equations.

#### 3.1 Perlite particle motion along the vertical heating chamber

The forces exerted on the falling particle are the gravitational force ( $F_G$ ) which accelerates the particle, the drag force ( $F_D$ ) and the buoyancy force ( $F_B$ ) which decelerate the perlite grain vertical fall by opposing its motion:

$$F_G = m_p g \quad (2) \quad F_D = \frac{1}{2} \rho_{air} C_D A_{proj} (U_{air} - U_p)^2 \quad (3) \quad F_B = \rho_{air} g V_p \quad (4)$$

The momentum balance on the falling perlite particle follows Newton's second law of mechanics: considering all forces exerted on a grain, the particle velocity evolution is computed by an ordinary differential equation <sup>[1][2]</sup>:

$$\frac{dU_p}{dt} = \frac{\rho_p - \rho_{air}}{\rho_p} g - \frac{3}{4} \frac{\rho_{air} C_D (U_{air} - U_p)^2}{D_p \rho_p} \quad (5)$$

The drag force coefficient ( $C_D$ ) is calculated via a correlation based on the particle Reynolds number ( $Re_p$ ) <sup>[1][2]</sup>:

$$C_D = \frac{K_1}{Re_p} + \frac{K_2}{Re_p^2} + K_3 \quad (6)$$

The particle Reynolds number ( $Re_p$ ) is calculated based on the relative velocity by the following equation <sup>[1][2]</sup>:

$$Re_p = \frac{\rho_{air} D_p |U_p - U_{air}|}{\mu_{air}} \quad (7)$$

The volumetric air flow rate due to the increase of air temperature and the decrease of air density along the grain trajectory in the heating chamber is taken into account toward air velocity calculation by the following equation:

$$U_{air} = \frac{\dot{Q}_{air,in} T_{air}}{A_{tube} T_{air,in}} \quad (8)$$

#### 3.2 Air heat balance in the vertical heating chamber

The problem of air temperature calculation in the heating chamber can be considered and solved as follows: air is injected into a vertical tube and flows at mean velocity, within heated furnace walls of constant temperature. Thermal energy is transferred to the fluid (air) by convection, thereby causing an increase in the air temperature. Air temperature calculation is based on an adiabatic energy balance, with the initial condition  $T_{air}(t=0) = T_{air,in}$ .

$$\frac{dT_{air}}{dt} = \left( \frac{\pi d h_{mean}}{\dot{m} C_{p,air,mean}} \right) (T_w - T_{air}) \quad (9)$$

The mean convective heat transfer coefficient ( $h_{mean}$ ) therein is calculated by the following algebraic equation:

$$h_{mean} = \frac{k_{air} Nu_{mean}}{d} \quad (10)$$

The mean Nusselt number along the heating chamber is calculated by Hausen's equation, which explicitly includes the effect of the thermal entry length, within which the heat transfer coefficient is not yet constant <sup>[1][2]</sup>:

$$Nu_{mean} = Nu_{\infty} + \left[ \frac{0.104 \left( \left( \frac{d}{L} \right) Re_t Pr_{air} \right)}{1 + 0.016 \left( \left( \frac{d}{L} \right) Re_t Pr_{air} \right)^{0.8}} \right] \left( \frac{\mu_{air}}{\mu_w} \right) \quad (11)$$

### 3.3 Perlite grain energy balance in the vertical heating chamber

The falling perlite grain is assumed to have a uniformly varying temperature, as internal gradients are negligible. The solid particle absorbs energy by radiation and convection: both contribute to heating and expansion and they are equally important heat transfer mechanisms in order to understand furnace as well as process efficiency. The grain temperature evolution due to radiative heating is calculated on the basis of the Stefan-Maxwell law, thus accounting for the thermal energy emitted by the furnace walls and absorbed by the moving perlite grain. Furthermore, the solid particle exchanges thermal energy with air by convective heating: this is an interesting phenomenon, as one can in fact numerically identify a sign reversal along the perlite grain trajectory: initially the cold particles are heated by the upward air current, but they are warmer than air as they approach the exit. Consequently, perlite grain temperature evolution is governed by the combination of radiative and convective heat transfer mechanisms and the dynamic heat balance is given by the following ordinary differential equation:

$$\frac{dT_p}{dt} = \varepsilon_w \cdot \frac{A_s s (T_w^4 - T_p^4)}{\rho_p V_p C_{p_p}} + \frac{A_s h_c (T_{air} - T_p)}{\rho_p V_p C_{p_p}} - \Delta H_{evap} \left( \frac{P_b}{R_g T_p} \right) \left( \frac{4\pi R_b^3}{3} \right) \quad (12)$$

Here,  $\varepsilon_w$  is the furnace wall emissivity (estimated at  $\varepsilon_w = 0.7$  by construction)<sup>[13]</sup>,  $A_s$  is the perlite particle surface area,  $s$  is the Stefan-Boltzmann constant and  $\Delta H_{evap}$  is the molar enthalpy of water evaporation (40.68 kJ·mol<sup>-1</sup>).

### 3.4 Steam bubble growth within the perlite grain

For the purpose of our modeling studies<sup>[1][2]</sup>, perlite expansion is approximated by a detailed single-grain model. The model particle consists of a spherical steam nucleus and a solid shell surrounding the steam bubble: steam is treated as an ideal gas, with no mass transfer across the steam-shell or the shell-environment interface permitted. During particle heating, the perlite grain temperature increase has a dual effect: firstly, steam enthalpy increases, thereby increasing the pressure exerted on the bubble-shell interface; concurrently, molten shell viscosity and cohesion decrease, thus facilitating bubble expansion and increasing steam bubble radius and perlite grain size.

The pressure factors acting on the bubble-shell interface are the surface tension ( $\sigma$ ), the steam pressure ( $P_b$ ), and the ambient pressure ( $P_a$ ): steam pressure acts towards expanding the bubble, while both surface tension and ambient pressure act against steam bubble growth. The steam bubble only contains the entire effective water amount, since the residual water amount is uniformly dispersed in the molten shell without affecting expansion. The initial steam bubble radius ( $R_{b,i}$ ) can be calculated by means of mass by the following algebraic equation:

$$R_{b,i} = \sqrt[3]{R_{p,i}^3 - \frac{3}{4\pi} \frac{m_m}{\rho_m}} \quad (13)$$

The molten shell mass ( $m_m$ ) is calculated using the grain water mass fraction ( $w_{H_2O}$ ) by the following equation:

$$m_m = (1 - w_{H_2O}) m_p \quad (14)$$

The steam in the core of the grain is treated as ideal gas: its instantaneous pressure is calculated by considering the bubble radius evolution in the bubble volume term and implementing the ideal gas law constitutive equation:

$$P_b(t) = \frac{3NR_g T_p}{4\pi R_b^3(t)} \quad (15)$$

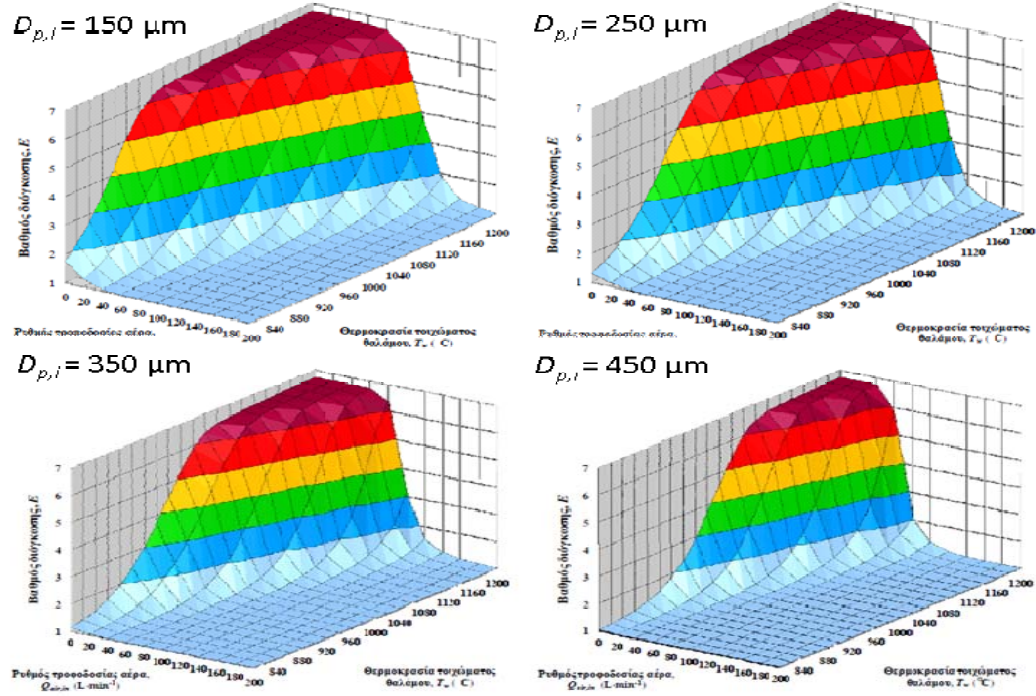
The Navier-Stokes equation for spherical creeping flow is combined with the melt radial velocity equation and the bubble surface stress balance, to yield an ordinary differential equation for bubble radius evolution<sup>[14][15][16]</sup>:

$$\frac{dR_b}{dt} = \frac{R_b}{4\mu_m} \left( P_b(t) - P_a - \frac{2\sigma}{R_b} \right) \quad (16)$$

The required initial condition is the miniscule steam bubble radius assumed at particle injection:  $R_b(t=0) = R_{b,i}$ .

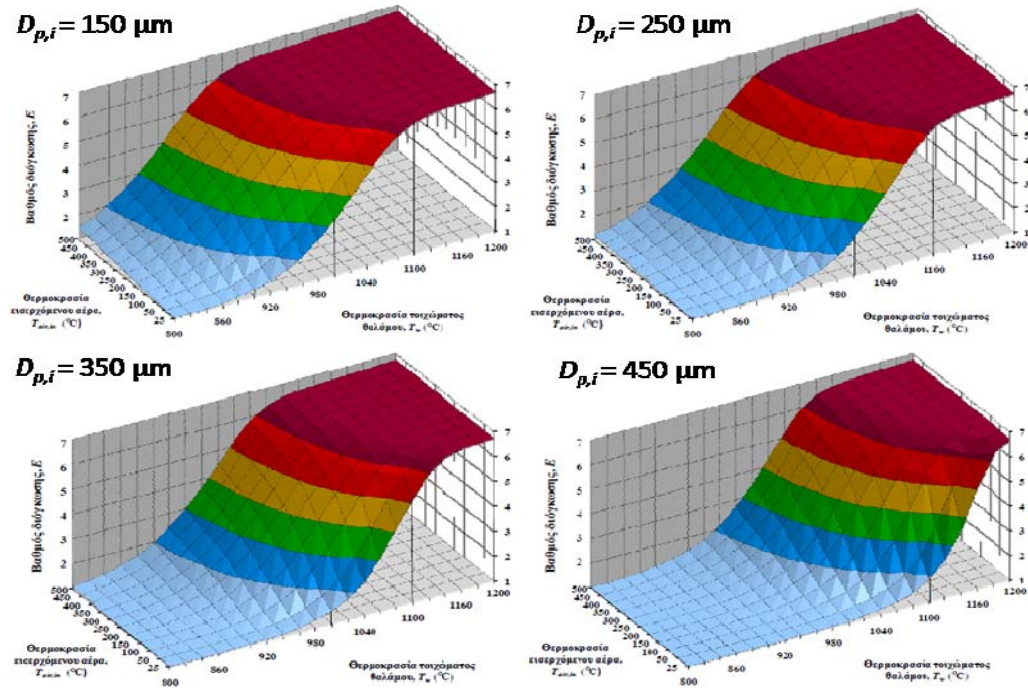
#### 4 DESCRIPTIVE STATISTICS

The expansion factor ( $E$ ) has been computed as a function of air flow ( $Q_{air}$ , left) and wall temperature ( $T_w$ , right).



**Figure 2.** Three-dimensional surfaces obtained from our perlite expansion model ( $E$  as a function of  $Q_{air}$ ,  $T_w$ ).

The response variable ( $E$ ) has also been studied as a function of air ( $T_{air}$ , left) and wall temperature ( $T_w$ , right).



**Figure 3.** Three-dimensional surfaces obtained from our perlite expansion model ( $E$  as a function of  $T_{air}$ ,  $T_w$ ).



## 5 PREDICTIVE STATISTICS

The application of the Response Surface Methodology (RSM) considers the bivariate quadratic function model:

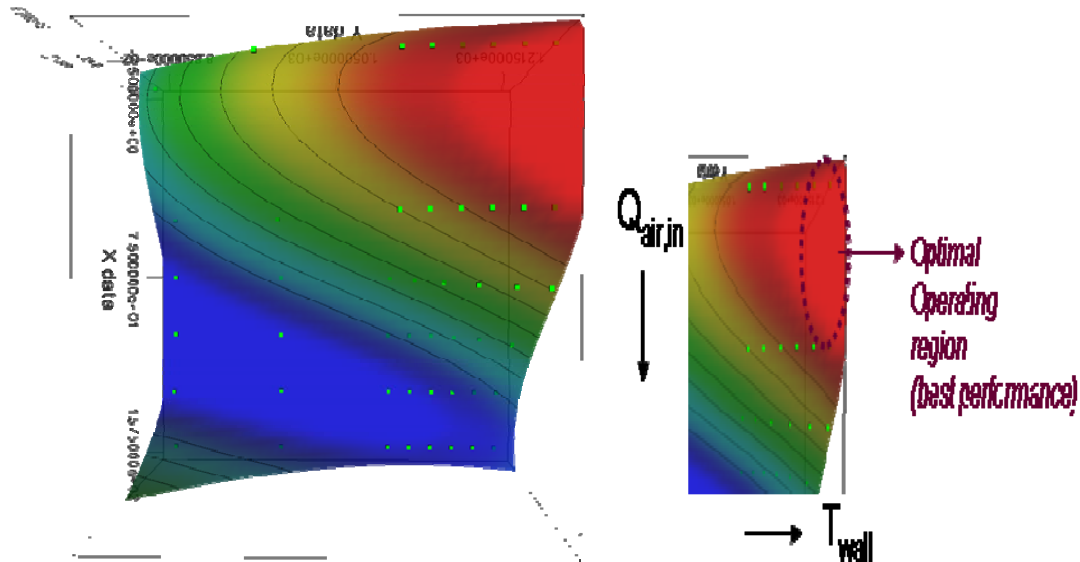
$$z = a + bx + cy + dx^2 + fy^2 + gxy \quad (17)$$

In this mathematical model,  $z$  denotes the response variable (final perlite particle expansion factor,  $E$ ) of interest,  $x$  denotes the first independent variable (volumetric air feed flow rate introduced at ambient temperature,  $Q_{air}$ ) and  $y$  denotes the second independent variable (wall temperature within the electrical furnace chamber,  $T_w$ ). Experimental (not model) results which have been obtained from NTUA vertical electrical furnace campaigns have been used to compute all model parameters ( $a, b, c, d, e, f$ ) via web-based software ([www.zunzun.com](http://www.zunzun.com)). RSM optimization results and all model parameters obtained are presented in Table 1 and illustrated in Figure 4.

**Table 1.** RSM optimization results for the bivariate quadratic function model (parameters and fitting statistics)

Model parameter values	Key statistical metrics
$a = 9.0350722499113196 \text{ E}+00$	Degrees of freedom (error): 34
$b = 1.09957916666666876 \text{ E}-01$	Degrees of freedom (regression): 5
$c = -3.1356046296627679 \text{ E}-02$	Chi-squared ( $\chi^2$ ): 13.5586008193
$d = 4.9515999999999644 \text{ E}-04$	R-squared ( $R^2$ ): 0.910003396114
$f = 2.9570124426975264 \text{ E}-05$	R-squared adjusted ( $R^2_{adj}$ ): 0.896768601425
$g = -2.2573273333333782 \text{ E}-04$	Model F-statistic: 68.7584067222
	Model F-statistic p-value: 1.11022302463 E-16
	Model log-likelihood: -35.1203741013
	Root Mean Squared Error (RMSE): 0.582207025449

RSM model results indicate that the most efficient perlite expansion (i.e., the highest expansion factor values) are obtained at the highest possible furnace wall temperatures (cf. Fig. 4 and respective  $y$  data – horizontal axis), but not at all at the highest possible air feed flow rates attainable (cf. Fig. 4 and respective  $x$  data – vertical axis). This fact has been known already from experimental campaigns and is exactly confirmed by our model results. The importance of RSM implementation and results, however, is that the construction of the response surface (Fig. 4) permits the rapid determination of optimal operating conditions in order to attain the desired expansion. Accordingly, for the feed quality assumed, the highest expansion factor achieved (maximum process efficiency) is attained using volumetric air flow rates between 50-75 L.min<sup>-1</sup> and furnace wall temperatures above 1100 °C.



**Figure 4.** Three-dimensional surface obtained by the Response Surface Method (RSM) for perlite expansion.

## 6 CONCLUSIONS

A Response Surface (RSM) mathematical model has been developed, statistically tested and validated (via comparison to model descriptive statistics) for perlite grain expansion within a novel vertical electrical furnace. The bivariate, second-order polynomial function selected to represent the known correlation of expansion factor (response variable) to volumetric air flow and furnace wall temperature (independent variables) requires the optimization-based calculation of only six parameters, a task which can be accomplished by available software. The dynamic model consists of ordinary differential equations for both air and particle heat and momentum balances, as well as nonlinear algebraic equations for air and perlite melt thermophysical properties, probing air temperature distribution as well as particle velocity, temperature and size along its trajectory in the chamber. The model computes particle state variables evolution as well as air temperature distribution in the furnace, allowing the detailed investigation of a new perlite expansion method towards its cost-effective optimization. The major conclusion of this optimization study is that the novel vertical electrical furnace design can successfully accomplish perlite expansion up to product quality standards, in an acceptable operating regime. Process optimization thus proceeds on the basis of both model and experimental results, but the RSM method is proven to be a credible computational tool which provides on-the-fly estimates of optimal operating conditions, on the assumption that perlite ore feed quality (particle size distribution, chemical composition, water content) does not change significantly between furnace runs; conversely, when feed quality variations are significant, mathematical modeling becomes crucial and the RSM methodology is used to obtain the new response surface.

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