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Defining a procedure for predicting the duration of the approximately isothermal segments within the proposed drying regime as a function of the drying air parameters

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Abstract. One of the main disadvantages of the recently reported method, for setting up the drying regime based on the theory of moisture migration during drying, lies in a fact that it is based on a large number of isothermal experiments. In addition each isothermal experiment requires the use of different drying air parameters. The main goal of this paper was to find a way how to reduce the number of isothermal experiments without affecting the quality of the previously proposed calculation method. The first task was to define the lower and upper inputs as well as the output of the "black box" which will be used in the Box-Wilkinson's orthogonal multi- factorial experimental design. Three inputs (drying air temperature, humidity and velocity) were used within the experimental design. The output parameter of the model represents the time interval between any two chosen characteristic points presented on the Deff – t. The second task was to calculate the output parameter for each planed experiments. The final output of the model is the equation which can predict the time interval between any two chosen characteristic points as a function of the drying air parameters. This equation is valid for any value of the drying air parameters which are within the defined area designated with lower and upper limiting values.

1. Introduction

Even though drying of porous material has been investigated for decades, it is still a current and actual topic of researchers in many scientific areas, e.g. chemical engineering, civil engineering and soil science. Physics and engineering have provided basics principles, with which this aspect of science can be additionally examined and discussed. A comprehensive understanding of the way in which water is transported from within the porous medium up to its surface, during drying can lead to many technical innovation and energy savings. In order to properly solve heat and mass transfer problems both the transport in air and in the porous material has to be modeled. This can be achieved at different complexity levels in both media. Calculation techniques (models) which are commonly used can be classified into four major groups: diffusion [1-3], receding front [4,5], macroscopic continuum models for coupled multiphase heat and mass transport in porous materials [6,7] and pore network models [8]. The conjugate modeling degree in each drying model can be determined by the way in which the heat and mass transport in air are accounted for in the calculation procedure.

The procedure for setting up the non isothermal drying regime, that is consistent with the theory of moisture migration during drying, was recently reported [9]. In order to properly apply this procedure it is necessary to firstly determine the change of effective moisture diffusivity vs. moisture content or drying time (Deff – MR or Deff - t curve) for each isothermal experiment, since these plots represents a good indicator for evaluation and presentation of the overall mass transport property of moisture during isothermal drying. In other words all possible mechanisms of moisture transport and their transition from one to another during isothermal drying, within a clay roofing tile, are visible on previously mentioned plots. Detailed information regarding the procedure for identification and quantification of moisture transport and their transition during drying can be found in reference [10]. Optimal drying regime is consisted of five isothermal segments. Durations of previously mentioned drying segments were detected from the relevant Deff – MR curves.

One of the main disadvantages of the reported method, for setting up the drying regime based on the theory of moisture migration during drying, lies in a fact that it is based on a large number of isothermal experiments. In addition each isothermal experiment requires the use of different drying air parameters. The main objective of this study was to find a way how to reduce the number of isothermal experiments without affecting the quality of the previously proposed calculation method. In order to complete this task and to find a mathematical equation, which can predict the time interval between any two chosen characteristic points (duration of each characteristic drying segments) as a function of the drying air parameters, within the defined area designated with lower and upper limiting values of input parameters, the Box-Wilkinson’s orthogonal multi-factorial experimental design was used.

2. Materials and Methods

The raw material, used in this study, was obtained from the roofing tile producer „Potisije Kanjiža“. Its detail characterization was reported in the study [11]. The raw material was first dried at 60^0C and then crashed down in a laboratory perforated rolls mill. After that simultaneously it was moisturized and milled in a laboratory differential mill, first with a gap of 3 mm and then of 1 mm. Laboratory roofing tile samples 120 × 50 × 14 mm were formed, from the previously prepared clay, in a laboratory extruder "Hendle" type 4, under a vacuum of 0.8 bar. Drying experiments were performed on previously formed roofing tile samples in laboratory recirculation dryer. The mass of the samples and their linear shrinkage were continually monitored and recorded during drying. The accuracies of these measurements were 0.01 g and 0.2 mm. Drying air parameters were regulated inside the dryer with accuracies of ±0.2 °C, ±0.2 % and ±0.1 % for temperature, humidity and velocity, respectively.

The response function in the Box-Wilkinson’s orthogonal multi-factorial experimental design is presented in a form of the equation (1).

$$y = f(x_1, x_2, \ldots x_k) \tag{1}$$

This equation corresponds to a surface in a multidimensional space, called the response surface. The space in which the response surface exists is called the factorial space. In the general case, when k factors are covered, equation (1) describes the response surface in k +1 measurement space. This function is usually defined as a polynomial expression (2).

$$y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i<j}^{k} b_{ij} x_i x_j + \sum b_{ii} x_i^2 + \cdots \tag{2}$$

The methodology, valid for isothermal experiments, presented in the [10] was used to calculate the functional dependence of the effective diffusivity with moisture content (Deff – MR), to divide obtained curves in segments and to identify all possible mechanisms of moisture transport. Obtained data were analyzed and used to predict the response y (duration of the proposed non isothermal drying segments).

Parameters x_1, x_2..., x_i represents the independent variables or factors (drying air velocity, temperature and humidity). The parameters b_0, b_i, b_{ij} represent the regression coefficients. When the regression coefficients are determined and the dependence defined by equation (2) is established the resulting equation is called a mathematical model. The adequacy of experiment reproducibility is checked using the Kohren criteria [12], while the adequacy of the model is checked using the Fisher criterion F [12]. The calculation of regression coefficients for the equation model and their analysis was realized using the Minitab 15 software package.

Experimental conditions presented at table 1 were used in the present study. Each experiment was repeated 2 times. Drying air parameters which were maintained in each proposed non-isothermal drying regimes are presented in table 2.

Table 1. Experimental conditions - isothermal drying regimes.

Experiment	Air velocity, *W* / m/s	Air temperature, *T* / °C	Air humidity, *V* / %
1 & 9	1	25	60
2 & 10	3	25	60
3 & 11	1	25	90
4 & 12	3	25	90
5 & 13	1	45	60
6 & 14	3	45	60
7 & 15	1	45	90
8 & 16	3	45	90

Table 2. Experimental conditions - proposed drying regimes.

Experiment	Segment														
	I (0-C)			II (C-D)			III (D-E)			IV (E-F)			V (90)		
	%	^{0}C	m/s	%	^{0}C	m/s	%	^{0}C	m/s	%	^{0}C	m/s	%	^{0}C	m/s
17	80	30	1	80	40	1.5	70	40	2.0	70	45	2.5	40	70	3
18	80	40	1.5	70	40	2.0	60	40	2.5	60	45	3	40	70	3

3. Results and discussion

Drying segments along with mechanisms that can take place in them according to the reference [10] are summarized at table 3. All possible mechanisms of moisture transport and their transition from one to another during the constant and the falling drying period up to the "lower critical" point F, for isothermal experiments, were identified on the corresponding Deff – MR curves and are summarized in table 4.

The procedure for setting up the non isothermal drying regime, that is consistent with the theory of moisture migration during drying (see table 2) was based on the principle of controlling the mass transport during the drying process and has demanded to divide the drying process into 5 segments. In each of these segments approximately isothermal drying conditions were maintained.

The main functions of the first drying segment was to restrain the moisture transport (evaporation), through the boundary layer between material surface and the bulk air, and to heat the ceramic body to the temperature of the drying air. During the second drying segment external (surface evaporation) and internal transport (of liquid water from the ceramic body up to the surface) should be increased and simultaneously harmonized in such way that the drying surface remains fully covered by a water film. The main function of the third segment is to provide the conditions that will lead to the fact that partially wet surfaces provide a constant rate of drying. Within the fourth drying segment, the liquid transport originating from the pores which are near or just below the "dry" patches on the surface, and are still in the funicular state, has to be simultaneously harmonized with the liquid flow originating from the surface "wet" patches.

Duration of the first drying segment was equal to the time interval detected in the corresponding isothermal experiment, from the beginning up to the characteristic points C. Duration of the second drying segment was equal to the time interval detected in the corresponding isothermal experiment, between the characteristic points C and D. Duration of the third drying segment was equal to the time interval detected in the corresponding isothermal experiment, between the characteristic points D and E. Duration of the fourth drying segment was equal to the time interval detected in the corresponding isothermal experiment, between the characteristic points E and F. Duration of the fifth segment was limited to 90 minutes.

Table 3. Possible drying mechanisms according to reference [10].

Drying segment	Transport of liquid water	Transport of vapor
A B	Capillary pumping flow (CPF) through the biggest capillaries	/
B C	CPF through macro capillaries, HF	/
C D	CPF through mezzo capillaries, HF	/
D E	CPF (from capillaries in funicular state),HF and liquid diffusion in the pores	hydrodynamic flow (HF)
E F	Creeping along the capillary when the liquid is in the funicular state or by the successive evaporation – condensation mechanism between liquid bridges.	HF (difference in total pressure)
F G	the successive evaporation – condensation mechanism between liquid bridges of pendular water	HF (difference in total pressure)
G H	the successive evaporation – condensation mechanism between liquid bridges of pendular water	Stefan Flux (difference in partial pressure), HF(difference in total pressure)
H I	The evaporation – condensation mechanism	HF (difference in total pressure)
I J	/	Molecular diffusion
J K	/	Transition diffusion
K L	/	Knudsen diffusion
0A - Initial period / AE - Constant period / FL - Falling period / D - "upper critical" point; "funicular state" – continuous threads of moisture are present in the pores. Surface is completely wet; Drying front start to recede into body / DE – Partially wet surface / F - "lower critical" point; "pendular state" – continuous threads of moisture are not present in the pores; "last" wet patches has disappeared from the surface.		

Table 4. Characteristic data for isothermal experiments from point A up to point F.

Exp.	t (min) / MR					
	A	B	C	D	E	F
1	40 / 0.989	110 / 0.888	171 / 0.781	273 / 0.551	327 / 0.474	491 / 0.282
2	32 / 0.990	90 / 0.891	140 / 0.782	223 / 0.553	268 / 0.475	402 / 0.285
3	70 / 0.986	160 / 0.872	272 / 0.771	403 / 0.541	508 / 0.415	741 / 0.218
4	64 / 0.987	145 / 0.880	246 / 0.774	365 / 0.545	460 / 0.417	672 / 0.222
5	21 / 0.986	75 / 0.883	116 / 0.779	186 / 0.546	223 / 0.471	335 / 0.276
6	16 / 0.985	53 / 0.880	82 / 0.778	131 / 0.544	158 / 0.470	237 / 0.273
7	55 / 0.983	125 / 0.878	212 / 0.773	315 / 0.544	397 / 0.416	579 / 0.220
8	48 / 0.982	105 / 0.870	178 / 0.770	265 / 0.540	330 / 0.414	486 / 0.216
9	41 / 0.988	111 / 0.889	171 / 0.782	272 / 0.552	327 / 0.473	490 / 0.281
10	32 / 0.991	89 / 0.892	141 / 0.781	222 / 0.555	267 / 0.474	401 / 0.284
11	71 / 0.988	159 / 0.871	272 / 0.771	402 / 0.542	509 / 0.417	740 / 0.217
12	64 / 0.987	144 / 0.882	245 / 0.773	366 / 0.546	461 / 0.418	671 / 0.220
13	22 / 0.985	75 / 0.883	115 / 0.778	187 / 0.548	224 / 0.472	335 / 0.276
14	17 / 0.984	53 / 0.880	83 / 0.779	130 / 0.542	159 / 0.471	236 / 0.272
15	55 / 0.983	126 / 0.879	213 / 0.774	313 / 0.543	396 / 0.415	578 / 0.219
16	47 / 0.981	106 / 0.871	178 / 0.770	266 / 0.539	331 / 0.412	487 / 0.215

Input parameters in the Box-Wilkinson's orthogonal multi-factorial experimental design were temperature, humidity and velocity (see table 1). The output parameters of the model are calculated from data presented in table 4. These parameters were:
-time interval to reach characteristic point C;
-time interval between characteristic points C and D;
-time interval between characteristic points D and E and

-time interval between characteristic points E and F.

The final model equation uses the regression coefficients in natural form and is valid for any values of the drying air parameters that are in the range defined by the experiment matrix (table 1). The estimated regression coefficients are presented in table 5. The final model equation for calculation the time interval to reach characteristic point C is labelled as equation (3) and is presented as an example. The data necessary for model valuation are presented in table 6. It can be seen that the evaluated models coefficients are very accurate and precise.

Table 5. Estimated regression coefficients for the proposed non isothermal drying segments.

independent variables - factors	Estimated regression coefficient			
	t (0-C)	t (C-D)	t (D-E)	t (E-F)
Constant	46.188	104.938	-28.563	111.375
Temperature	-2.138	-1.788	-0.688	-2.275
Humidity	3.152	0.567	1.733	1.875
Velocity	-29.063	-14.563	-11.813	-26.750
Temperature*Humidity	0.011	0.015	0.003	0.015
Temperature*Velocity	0.213	-0.088	0.363	-0.050
Humidity*Velocity	0.048	0.025	0.046	0.054
Temperature*Humidity*Velocity	-0.005	0.000	-0.006	-0.001

$$t(0\text{-}C)=46.188-2.138*A+3.152*B-29.0623*C+0.011*AB+0.213*AC+0.048*BC-0.005*ABC \tag{3}$$

Table 6. Statistical parameters for model valuation.

Statistical parameters for model validation	t (0-C)	t (C-D)	t (D-E)	t (E-F)
R-Sq / %	100	99.86	99.95	99.98
R-Sq(pred) / %	99.98	99.43	99.82	99.92
R-Sq(adj) / %	99.99	99.73	99.92	99.96
Note: *R-Sq – is representing the probability of correlation coefficient accuracy.* *R-Sq (pred) – is representing the model prediction accuracy.* *R-Sq (adj) – is representing the adjusted model prediction accuracy. This model accuracy is including the correction which is related on the number of model parameters.*				

Normal effects plot is used to compare the relative magnitude and the statistical significance of both the main and interaction effects. Minitab draws a line to indicate where the points would be expected to fall if all effects were zero. The points that do not fall near the line usually signal significant effects. Such effects are larger and generally further from the fitted line than unimportant effects. A Pareto chart of the effects is used to compare the relative magnitude and the statistical significance of both the main and interaction effects. Minitab plots the effects in decreasing order of the absolute value of the effects. The reference line on the chart indicates which effects are significant. The normal plot and Pareto charts of the standardized effect are presented in Fig. 1

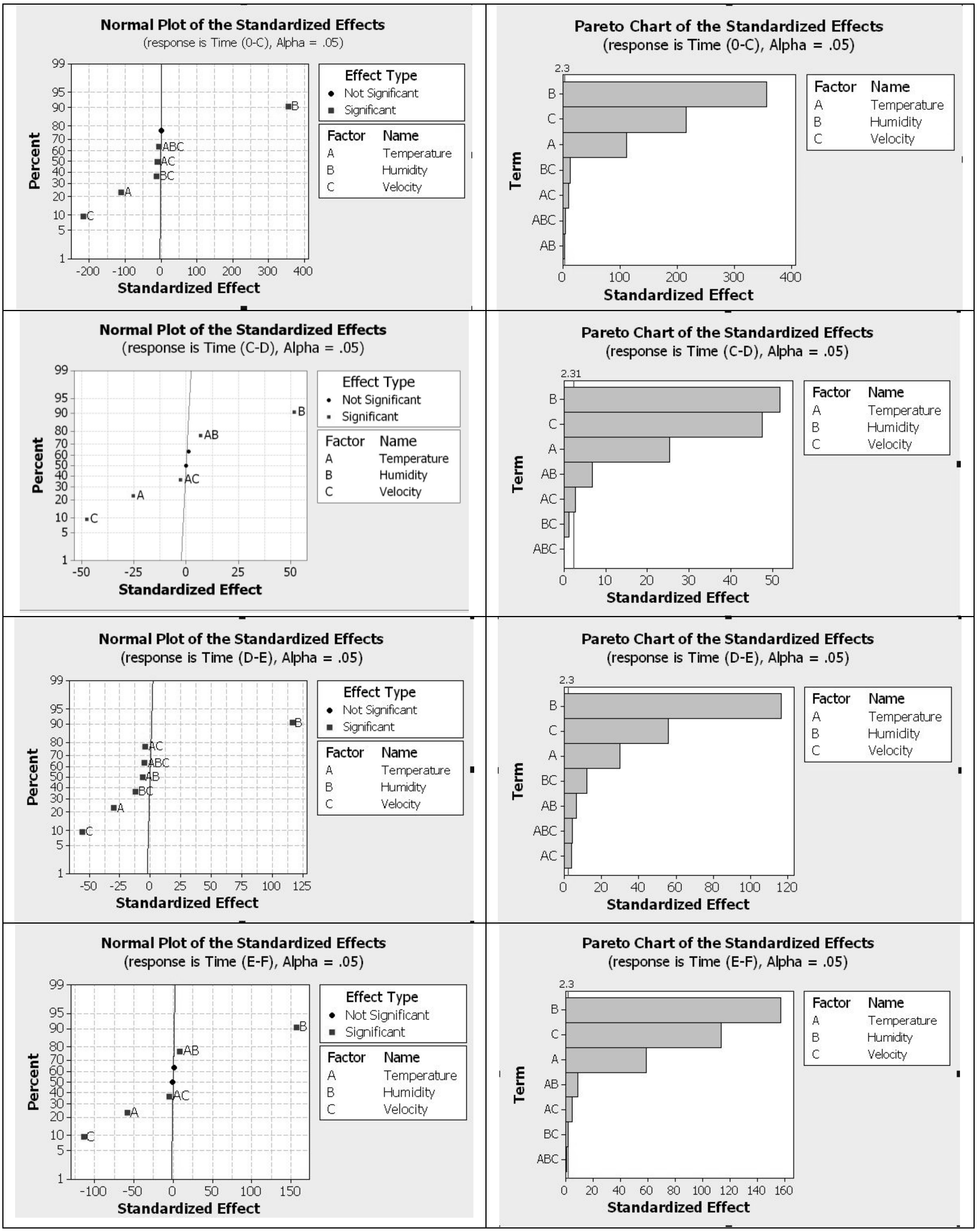

Figure 1. Normal plot and Pareto chart of the standardized effect.

It can be seen that all main effects A, B and C (drying air temperature, humidity and velocity) are statistically significant. Interaction effects (AB, AC, BC and ABC) had lower impact on the response model value (duration of each proposed drying segments) than the main one. The greatest impact on the response model value has the main effect B. It is followed by the main effects C and A. This is expected result since higher values of drying air humidity will restrain the external moisture transport (evaporation), through the boundary layer between material surface and the bulk air, while the higher drying air temperature and velocity will simultaneously increase internal as well as external moisture transport. Calculated results for each of the proposed non isothermal drying segments are presented in table 7. Proposed drying regimes were tested. Clay roofing tiles were dried without cracks.

Table 7. Duration of the proposed drying segments

Exp.	t (0-C)	t (C-D)	t (D-E)	t (E-F)	V
17	231	103	52	117	90
18	202	82	33	79	90

4. Conclusion

One of the disadvantages of the previously reported method, for setting up the non isothermal drying regime that is consistent with the theory of moisture migration, lies in a fact that it is based on a large number of isothermal experiments. In order to reduce the number of isothermal experiments without affecting the quality of the previously proposed calculation method, the Box-Wilkinson’s orthogonal multi-factorial experimental design was used. Input parameters of the Box-Wilkinson model were drying air temperature, humidity and velocity. The output parameters of the Box-Wilkinson model (time interval between characteristic points OC, CD, DE and EF) were detected in accordance with the theory of moisture migration during for each experiment defined in the designed experimental matrix. Four mathematical equations for prediction the time interval between characteristic points (duration of characteristic drying segments) as a function of the drying air parameters were developed. These simple equations are valid for any values of the drying air parameters that are in the range defined by the experiment matrix.

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