DETERMINAREA COEFICIENTULUI EFECTIV DE DIFUZIE
DETERMINATION OF THE EFFECTIVE DIFFUSION COEFFICIENT

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The equation for drying kinetics is obtained, based on the analytical solution of the differential equation with a boundary condition in the form of the flux. This equation was initially developed by G. Efremov. In this paper a modification of the Efremov drying equation will be presented. Shrinkage correction will be included in that equation for the first time.

Two programs were designed to compute the effective diffusion coefficient. First program did not include shrinkage effect during drying into the computation algorithm while the second one has included it. Two models for predicting the drying behavior were obtained as the result of cited programs calculation. First model did not include shrinkage (model 1) and second one (model 2) has included it. The predicted values obtained from model 2 fit with experimental data. Results presented in this study showed that the values of effective diffusion coefficient determined by designed computer programs have similar values with values available in literature related to the same coefficient determined for different clays.

A fost obținută o soluție analitică a ecuației diferențiale care caracterizează cinetica procesului de uscare, rezolvată împreună cu condiții la limită de tip Neumann. Această ecuație a fost inițial propusă de G. Efremov. În lucrare se prezintă o modificare a ecuației Efremov. Pentru prima dată, în ecuație este inclusă și contracția materialului.

Două aplicații software au fost dezvoltate în scopul calculului coeficientului efectiv de difuzie. Prima dintre acestea nu include contracția în timp ce a doua o include. Corespunzător, au fost obținute două modele pentru predicția comportării la uscare a materialului umed. Primul model (modelul 1) nu include această corecție în timp ce al doilea (modelul 2), da. S-a constatat că rezultatele aplcării modelului 2 estimează corect valorile experimentale. Rezultatele prezentate în acest studiu au arătat că valorile coeficientului efectiv de difuzie determinat cu ajutorul aplicațiilor de calcul sunt similare celor disponibile în literatura de specialitate pentru diferite argile.

Keywords: *effective diffusion coefficient, analytical solution, software*

1. Introduction

Researchers from all around the world are interested to describe the drying process. Their effort is concentrated on establishing a number of theoretical, semi-theoretical or empirical drying models which more or less agree with experimentally determined data. Simultaneous processes of mass and energy transfer, which are usually non stationary as well as different nature and properties of drying material (hygroscope, capillarity, pores size distribution, shrinkage effect etc.), make the determination of drying process even more complicated. That is the reason why a unique drying theory which could explain this process and can be valid for different kind of materials is not available in literature.

The diffusion process regarded, as a transport of matter via random molecular motion, is inherent and typical for drying. Transfer of moisture within solid body at certain temperature is driven by the core – to – surface moisture content difference. It is necessary to say that pure diffusion is not exclusively the only mass transport process but is usually the most frequent one. The rate of mass transfer by pure diffusion is proportional to the moisture concentration gradient. Diffusion coefficient is representing a factor of proportionality.

If we want to present reliably the mass transfer process described by Fick's second law we need to know the diffusion coefficient. In Crank's monograph [1] "The mathematics of diffusion" several different ways for determination and computation of Fick's equation were presented.

Beside pure diffusion, drying process is characterized by existence of several other, secondary types of interior mass transfer such as: surface diffusion, Knudsen's diffusion, capillary flow, evaporation and condensation, thermo-diffusion etc. which in some small amount influence the value of complete mass transfer [2]. In drying process mass transfer is mainly done by pure diffusion. It is a common practice to describe complete mass transfer with same equations as pure diffusion and to take the correction, for all secondary types of mass transfer into account simply by replacing the pure diffusion coefficient with effective diffusion.

There are numerous papers in which

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predicted values for drying kinetics obtained by using different drying models for different drying materials, which include or ignore shrinkage of drying material, are compared with experimentally determined drying kinetic parameters [3-6].

For material such as food or construction material, clay or wood which possess shrinkage effect during drying, that can not be diminished and which is connected with process of losing moisture, previously mentioned papers and many others showed the necessity for introduction of the shrinkage correction in the process of drying modeling.

In most of drying models, shrinkage is not considered in drying equations and usually we start from the assumption that it does not exist. Such models without any physical and mathematical consideration are applied on materials that show shrinkage and shrinkage deviations are fixed by including correction factors in drying equation.

Small number of papers, which describe drying process of ceramic materials and especially clay, is available in literature. Some data can be found in papers of Guerman I. Efremov [7] (bricks), Saber Chemkhi [8], F. Zagrouba [9,10] (clays), Darko Skansi [11,12] (heavy clay tiles), and others.

2. Description of work method

Raw material sample taken at the locality "Ćirilkovac" was analyzed it this paper. After initial characterization of cited raw material sample which included chemical, mineralogical, XRD, TGA and granulometric examination, it was subjected to further classical preparation. Raw material sample was treated using classical processing procedure; first, it was dried at 60°C and then milled down in lab perforated rolls mill. After that, clay was moisturized and milled in lab differential mill first at gap of 3mm and then of 1mm. Laboratory samples size 120x50x14 mm were formed in laboratory extruder "Hendle" type 4, under the vacuum of 0.8 bar. These samples were used in further experimental work.

Linear shrinkage and weight changes of created masonry products during drying process, in the specially constructed laboratory dryer, were recorded.

Laboratory recirculation dryer provides:

- regulation of drying air temperature within 0-125 C°, with accuracy 0.2 ± C°
- regulation of drying air relative humidity within 20-100%, with accuracy of 0.2%
- speed regulation of drying air within 0-3.5 m/s, with accuracy 1%.
- monitoring and recording the drying samples weight within 0-2000g, with 0.01 g accuracy
- monitoring and recording the linear shrinkage within 0-23 mm with accuracy of 0.2 mm
- continuous time monitoring during drying.

On prepared heavy clay tiles (samples), in laboratory recirculation dryer, under experimental conditions which are presented in Table 1, drying kinetic curves were recorded [13].

Table 1

Experimental conditions / *Condiții experimentale*

Experi-ment	Air velocity *Viteza aerului* W (m/s)	Air temperature *Temperatura aerului* T (^{0}C)	Air humidity *Umiditatea aerului* V (%)
1	3	40	60
2	3	40	40
3	3	55	60
4	3	70	60
5	3	70	40

In literature, the diffusion coefficient can be obtained from drying curve by slope method [14,15], or by comparing experimentally determined curves with curves obtained from Fick's equations predicted analytically [2,3,16] or numerically [6,17].

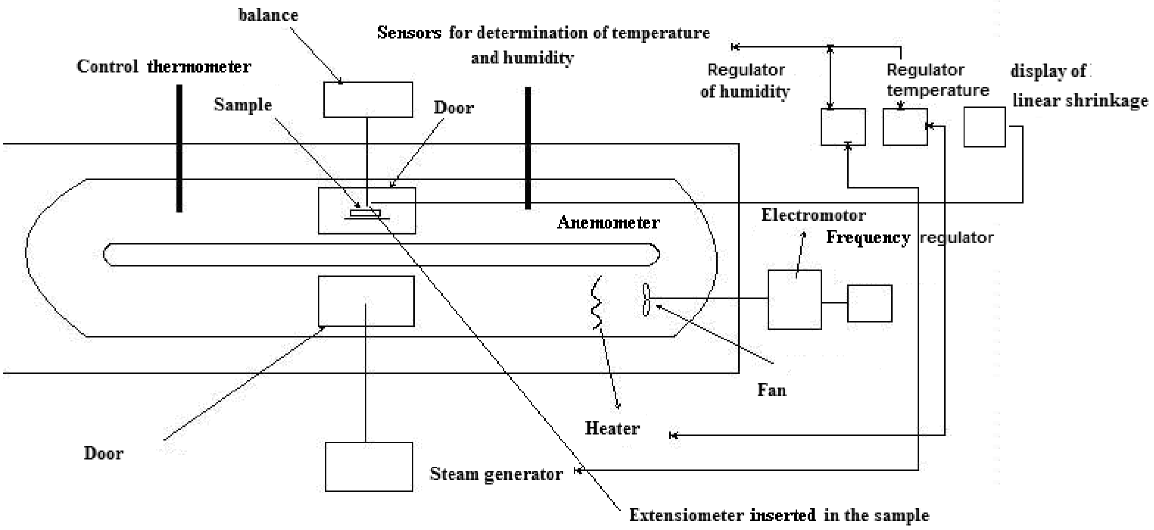


Fig. 1 - Schematic view of laboratory recirculation dryer / *Schema uscătorului de laborator, cu recirculare.*

If parameters of drying medium are kept constant during convective drying of solid bodies, moisture transfer could be treated on macro level as quasi diffusion with appropriate effective diffusion coefficient D_{eff}. The general expression (below) for mass conductivity (Fick's second law) can be presented as a partial differential equation of diffusivity.

$$\frac{\partial X}{\partial t} = div(D_{eff} \cdot gradX)\,; \rightarrow \ \frac{\partial X}{\partial t} = D_{eff}\frac{\partial^2 X}{\partial x^2} \qquad (1)$$

The exact solution for drying kinetics can be obtained by applying Laplace transform method in time t for equation of isotropic diffusion with boundary conditions in a form of mass flux J. This flux is proportional to the difference between an equilibrium concentration in the pores of the material X_{eq} and the current concentration X on the material surface.

$$J = -D_{eff} \cdot \frac{\partial X}{\partial x}\Big|_{x=0} = k \cdot \left(X_{eq} - X\right) \qquad (2)$$

Kinetic desorption coefficient k (m/s) in equation (2) can be calculated as a ratio x (characteristic value) and time $\left(k = {x}/{t}\right)$.

By applying Laplace transform method to equation (2) Efremov in his PhD thesis [18] presented the solution given by equation (3).

$$\frac{X - X_{eq}}{X_0 - X_{eq}} = erf\left(\frac{x}{2\sqrt{D_{eff}t}}\right) + \qquad (3)$$

$$+\exp\left(\frac{k}{D_{eff}}\,x + \frac{k^2}{D_{eff}}\cdot t\right)\cdot erfc\left(k\sqrt{\frac{t}{D_{eff}}} + \frac{x}{2\sqrt{D_{eff}t}}\right)$$

The mass flux on the material surface (x=0) can be calculated through the use of the concentration ratio which is given in equation (4)

$$\frac{X - X_{eq}}{X_0 - X_{eq}} = \exp\left(\frac{k^2}{D_{eff}}\cdot t\right)\cdot erfc\left(k\sqrt{\frac{t}{D_{eff}}}\right) \qquad (4)$$

Equation (4) satisfies conditions of drying process: (t=0): $X=X_0$ and $\frac{X - X_{eq}}{X_0 - X_{eq}}=1$, while for long periods of time ($t \rightarrow \infty$): $X=X_{eq}$ and $\frac{X - X_{eq}}{X_0 - X_{eq}}=0$.

In order to avoid two unknown parameters (k and D) Efremov has introduced the parameter δ called characteristic time defined by equation (5).

$$\delta = {D_{eff}}\Big/{\pi \cdot k^2} \qquad (5)$$

$$\frac{X - X_{eq}}{X_0 - X_{eq}} = \exp\left(\frac{t}{\pi\delta}\right)\cdot erfc\left(\sqrt{\frac{t}{\pi\delta}}\right) \qquad (6)$$

As equation (3) was obtained for the process of molecular diffusion, the convective mass transfer can be entered into calculation by introducing the power function of the argument in equations (6), thus the drying kinetic equation (7) is obtained.

$$\frac{X - X_{eq}}{X_0 - X_{eq}} = \exp\left(\frac{1}{\pi}\left(\frac{t}{\delta}\right)^n\right)\cdot erfc\left(\sqrt{\frac{1}{\pi}\left(\frac{t}{\delta}\right)^n}\right) \qquad (7)$$

Simple approximation formula for function erf(A) is defined by equation (8) and can be found in Sergei Winitzki [19,20] papers. The relative precision of this approximation is higher than $4 \cdot 10^{-3,}$ uniformly for all real A.

$$erf(A) = \left[1 - \exp\left(-A^2\frac{1.27 + 0.14A^2}{1 + 0.14A^2}\right)\right]^{1/2} \qquad (8)$$

After some mathematical manipulation, knowing that erfc(A) = 1 – erf(A), the final drying kinetic equation (9) is obtained.

$$\mathrm{MR} = \frac{\mathrm{X} - \mathrm{X_{eq}}}{\mathrm{X_0} - \mathrm{X_{eq}}} = \exp\left(\frac{1}{\pi}\left(\frac{\mathrm{t}}{\delta}\right)^{\mathrm{n}}\right)\cdot \qquad (9)$$

$$\cdot\left\{1 - \left[1 - \exp\left(-\frac{1}{\pi}\left(\frac{t}{\delta}\right)^n \cdot \frac{1.27 + 0.14\frac{1}{\pi}\left(\frac{t}{\delta}\right)^n}{1 + 0.14\frac{1}{\pi}\left(\frac{t}{\delta}\right)^n}\right)\right]^{1/2}\right\}$$

Analytical solution of Fick's equation was determined for several geometries taking into account the following assumptions: moisture transport is done by diffusion, product shrinkage is neglected and value of diffusion coefficient and temperature are constant. For the case of thin plate geometry, Crank has recommended the solution in a form of Equation (10).

$$\mathrm{MR} = \frac{8}{\pi^2}\sum_{n=N+1}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2}{4}\pi^2\frac{D_{eff}t}{x^2}\right) +$$

(10)

$$+\frac{8}{\pi^2}\sum_{n=1}^{N}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2}{4}\pi^2\frac{D_{eff}t}{x^2}\right) \qquad (10)$$

MR =

$$\frac{8}{\pi^2}\sum_{n=1}^{N}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2}{4}\pi^2\frac{D_{eff}t}{x^2}\right) \quad (10a)$$

Transformation of equation (10) into (10a) can be obtained by introducing of coefficient ε, which enables calculation of number of members N which will remain further in equation (10a). Value of ε = 0.05 is accepted for further calculation in this paper. When t=0, MR=1 equation (10) is transformed into equation (10b)

$$\frac{8}{\pi^2}\left(\sum_{n=N+1}^{\infty}\frac{1}{(2n+1)^2}+\sum_{n=1}^{N}\frac{1}{(2n+1)^2}\right)=1 \quad (10b)$$

$$\frac{8}{\pi^2}\sum_{n=1}^{N}\frac{1}{(2n+1)^2}=(1-\varepsilon)=0.95 \qquad (10c)$$

From the equation (10c) it is possible to calculate the number N which is used in equation (10a).

MR is reduced moisture content. In this study X_0, X, X_{eq}, D_{eff} and x represents initial, current and equilibrium moisture content on the dry base (kg moisture / kg dry material), effective diffusion coefficient (m^2/s) and half thickness of sample thin plate (m) respectively.

3. Results

The aim of this paper is to calculate the average effective diffusion coefficient. MR_{eks} values are obtained from experimentally determined values for X_0, X, $X_{eq,}$ and consequently MR_{eks}= f(t) is obtained too.

In order to calculate average effective diffusion coefficient we first need to compute characteristic time δ from the equation (9). Determination of parameter δ can be done using the concept of optimized comparison method, which is developed specially for this purpose. Introducing the parameter δ and k into equation (5) we obtain the following equation (11).

$$D_{eff} = \delta \cdot \pi \, {x^2}/{t^2} \qquad (11)$$

The value x can be fixed in the case of drying with neglected shrinkage effect or can be a function of time in the case of drying with active shrinkage effect.

Average value D_{eff} is then computed for the whole time interval t. Value ε is set as 0.05 and N value used in equation (10) is calculated. Function MR_{an} - t is determined by using average value D_{eff} and equation (10). Evaluation of calculated average D_{eff} value is done by comparing functions MR_{eks}– t and MR_{an}–t.

In the following section we will present the concept of optimized comparison method and the software algorithm for determination of average effective diffusion coefficient. The algorithm presented below is the same for any software program. On the base of this algorithm two programs for computing the average effective diffusion coefficient were written using Borland C compiler on standard Pentium IV computer. First program neglects shrinkage and second calculates with shrinkage correction.

In the program which neglects shrinkage, the algorithm contains the following steps:

1. Read the values from database: the time (in min), MR_{eks}.
2. Enter number ε (Usually ε =0.05).
3. Enter the initial value of δ (δ =$1\cdot10^{-20}$)
4. Enter the characteristic dimension x (samples half thickness in mm)
5. Enter the value n form equation (9) (n=2)
6. For each value from the database using equation (9) MR_{an} will be determined.
7. For each value from database χ^2 will be determined using the following formula $\chi^2 = \sum_{1}^{i}(MR_{eks\ i} - MR_{an\ i})^2$.
8. In next cycle step starting value δ is doubled and a new value MR_{an} will be determined, and initial used for determination of new χ^2.
9. If $\chi^2_{first-step} < \chi^2_{second-step}$ the cycle step continues. The iterative process stops in the moment when $\chi^2_{first-step} > \chi^2_{second-step}$.
   ***Remark:** $\chi^2_{first-step}, \chi^2_{second-step}$ regards to last and next to last value of the cycle in which χ^2 is determined.*
10. Last three values for δ and χ^2 will be saved. The interval from $\delta_{\text{third from the last}}$ till δ_{last} will be divided into 100 parts. Step s is defined as 0.01 part of the previously mentioned interval. Now we enter again into cycling step with initial value of δ as δ = $\delta_{\text{third from the last}}$ + s. Cycle will continue until $\chi^2_{first-step} < \chi^2_{second-step}$ and < $1\cdot10^{-10}$. In other words the cycle stops when $\chi^2_{first-step}$ - $\chi^2_{second-step}$ =$1\cdot10^{-10}$. In that moment the optimal value δ is determined.
11. From the equation (11) we compute D_{eff} as a function of time. Than we determine an average value of D_{eff} in given time interval.
12. Value N is determined.

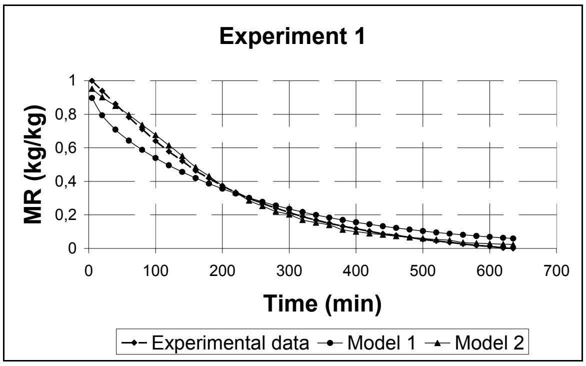
13. Values of MR_{an} will be determined by using the equation (10a)
14. Result will be saved as database: time (in min), MR_{eks}, MR_{an}, and value of average D_{eff}.
15. On the base of this database a graphical view can be displayed

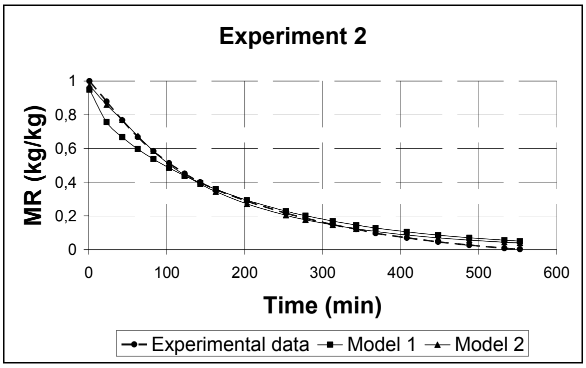
In the case of the program which includes shrinkage, the previously presented algorithm contains a few modifications: in steps 1, 12 and 13.

1. Read the values from database: the time (in min), MR_{eks}, and characteristic x.
11. In equation (11) x is a function of time; x is provided from database where values of x were determined by experimental measuring of thin plate sample shrinkage vs. time.
13. In equation (10a) x is a function of time and vales are provided from the same experimental database as quoted in previous point 11.

4. Interpretations and discussions

Determined values for average value of D_{eff}, obtained through the use of previously described programs are presented in Table 2, while kinetic data are shown graphically in Figure 2.



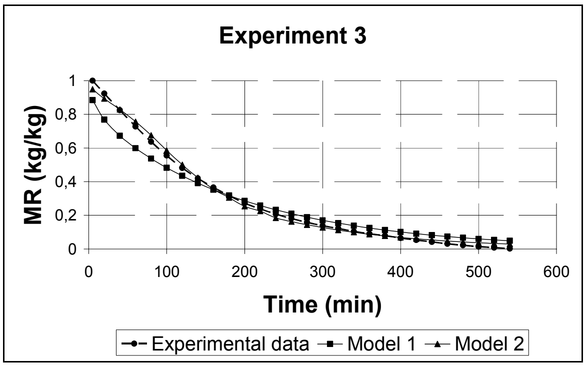


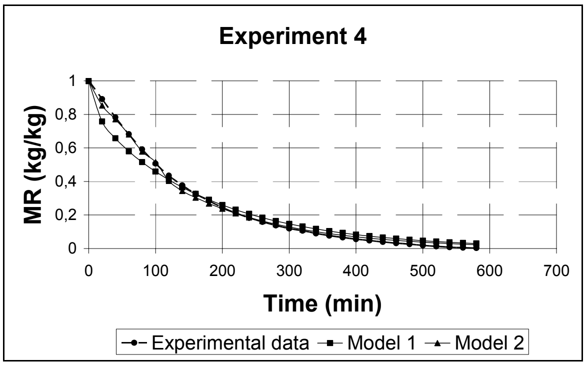
It is obvious that the value of mean effective coefficient (D_{eff} in m/s) determined using the model which included sample shrinkage correction is higher than the same coefficient value computed using the model which neglected the sample shrinkage.

Table 2

Determined mean value of effective diffusion coefficient
Valorile medii, calculate, ale coeficientului efectiv de difuzie

Experiment	$Deff \cdot 10^9$ u m^2/s	
	without correction *fără corecție*	with correction *cu corecție*
1	0.678	0.341
2	0.100	0.415
3	0.077	0.431
4	0.150	0.472
5	0.126	0.583





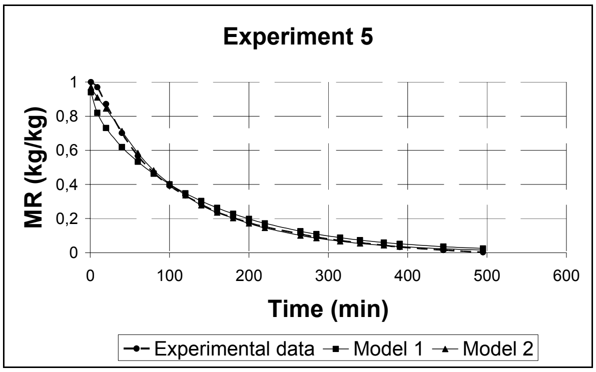


Fig. 2 - Drying kinetics data for all experiments / *Curbe de uscare pentru toate experimentele.*
Note: Model 1 = model without shrinkage, Model 2 = model with shrinkage

For long drying time, equation (10) can be transformed into equation (12). In Lalić's master work [21] effective diffusion coefficient was determined from equation (12), through the use of samples made of the same clay under the same experimental conditions as one used in this paper. Determined values of effective diffusion coefficient are presented in Table 3.

$$MR= \frac{8}{\pi^2}\exp\left(-\frac{(2n+1)^2}{4}\pi^2\frac{D_{eff}t}{l^2}\right) \qquad (12)$$

Determined values of data for D_{eff} presented in table 3 were higher from the data presented in Table 2. This is the expected result which is in agreement with the D_{eff} determination.

Table 3

Plots of experimental and computed values for D_{eff}
Valori experimentale ale coeficientului efectiv de difuzie

Experiment	Deff · 10^9 u m^2/s
1	1.24
2	1.93
3	2.00
4	2.32
5	2.76

That is another proof that the determination model which included shrinkage effect during drying has given more precise D_{eff} values. Only a few scientific papers [8,11] in which effective diffusion coefficients for masonry clay products were determined are available. In these papers D_{eff} values are in range of 10^{-7} up to 10^{-12} m^2/s. This relatively large range for D_{eff} values is connected with the different nature of heavy clay and different methods used for its determination. D_{eff} values presented in Tables 2 and 3 are beneath previously mentioned range.

Kinetic diagram analysis show that the kinetic curves representing the model which neglects shrinkage effect do not completely follow the configuration of experimentally determined kinetic curves. Deviations of that model from experimental drying curves are bigger at the beginning of the drying process and after some time deviations disappear. Disappearing moment matches the moment from which sample dries further without shrinkage. Drying kinetic curves of the model which includes shrinkage follow the configuration of experimentally determined curves and their matching can be more than 95% (experiment 4). If minor deviations exist it is at the beginning of the drying process and are most probably caused by time interval which has to pass until stationary experimental conditions are fulfilled and products are heated up to the temperature in the dryer. The intersection point of experimental drying curves and modeled drying curves is characterized as the critical point. Critical point is a characteristic kinetic parameter which is important because it determines the moment in time after which the products do not shrink anymore.

5. Conclusion

Two programs which analyze the problem of one dimensional diffusion and effective diffusion coefficient during drying of heavy clay tile samples were designed. Kinetic diagram analysis show that the kinetic curves representing the model which neglects shrinkage effect do not totally follow the configuration of experimentally determined kinetic curves while in the case of the model which includes shrinkage representing curve follows experimental one. Introduction of shrinkage correction into equations (10a) and (11) is entirely justified. Determined values of effective diffusion coefficient are beneath the value which could be found in literature. Effective diffusion coefficient values determined by using the model which includes shrinkage are higher then the values determined by using the model which neglects shrinkage. The intersection point of experimental drying curves and modeled drying curves is characterized as critical point.

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