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NaOH Activation of Raw Soils: Effect of NaOH Content on the Drying Kinetic and Its Modelling

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Abstract

NaOH activation of soils is an affordable and promising way to improve mechanical properties of earthen bricks. If for well-activated geopolymers, the hard polymeric network limits the influence of water on mechanical properties, for the weakly activated one, as non-calcined raw clayey soils, the influence of water on these properties would be more critical. This work aims to determine the effect of sodium hydroxide concentration on the drying kinetics of bricks made with raw clayey soils, and to model this kinetics. The results show that the drying kinetics is governed by the diffusion of water due to the absence of free water. The drying duration increases linearly with the increasing of NaOH content, while the volumetric shrinkage decreases, probably thanks to the reduction of the material porosity during the formation of the zeolitic structures. Besides, the drying duration is strongly and negatively correlated with the initial drying rate (−0.97) and bricks did not show visible cracks. Among the five parametric models tested, the Khazaei's model is the best in terms of all statistical criteria considered. For all models used, the coefficient of determination is ranged from 0.993 to 0.999, and the evolution of the models' parameters is in accordance with that of the drying kinetics observed.

Keywords

Earthen Brick, Stabilization, Alkaline Activation, Modeling, Geopolymer, Drying Kinetics

1. Introduction

Access to decent housing remains a significant challenge for many people, especially in developing countries [1]. In Sub-Saharan African villages, for example, the earthen dwellings remain the most numerous because they are low cost and easy to build. Indeed, for these constructions, the soil is usually taken from the immediate vicinity of the future house and used without stabilizer or with plant products (fibers, kernels) as reinforcement. Besides, the owners manufacture themselves or participate in the manufacture of the materials. In addition to be inexpensive, these houses are eco-friendly and have excellent thermal behavior [2]. However, they have the drawback to require frequent repairs due to the weak strength of the soils used. Indeed, unfortunately, only a few raw soils are suitable for construction without stabilization. For instance, for most standards, the suitable clay content to manufacture earthen bricks is ranged from 10 to 30% [3] [4]. For less and more clay contents, it is necessary to add a stabilizer as cement [5], lime [6], ash [7] [8] [9] or to use the improved technique as firing, geo-polymerization or soil activation [10] [11] [12] [13] [14]. The choice of the stabilizer depends on several factors, including its cost and availability, the nature of the soil, and the complexity of the technique [5] [6].

The geopolymer compressive strength depends notably on the nature of the aluminosilicate source, the type and concentration of the alkaline activator used, and the duration and the temperature of the curing [13] [14] [15]. It varies from 1 MPa for geopolymers made in solid-state to 110 MPa for fly ash activated with the mixture of sodium silicate and sodium hydroxide solution. Although the mixture of the sodium silicate and hydroxide sodium is better than the hydroxide sodium alone [13] [14] [15] [16], this last activator is the most used because it is the cheapest, and the most available [13]. Most studies on geopolymers are focused on their mechanical properties and the chemical reactions, while information on their drying kinetics is scarce. If for well-activated geopolymers, the hard polymeric network limits the influence of water on mechanical properties, for the weakly activated one, as non-calcined raw clayey soils [14] [16] [17], the influence of water on these properties would be more critical. Besides, it is well known that during their drying, clayey materials often shrink and crack due to the evaporation of water. The purpose of this work is to determine the effect of sodium hydroxide concentration on the drying kinetics of earth bricks, on the one hand, and to model this kinetics, on the other hand.

2. Materials and Methods

2.1. Soils and Manufacture of Bricks

Two raw clayey soils were used for the manufacture of bricks. The first one was obtained by crushing termite *Cubitermes spp*. mounds (CMS) collected in the savanna around NGO (2˚29'14"S; 15˚45'20"E). They are at most 50 cm high and 30 cm large and are usually used to build floors in traditional houses or as rural road pavement. The crushed soil was sieved and only grains smaller than 2 mm were retained to manufacture bricks. The characteristics of this soil have been published [18] and are recalled in **Table 1**.

The second soil used is the lateritic soil (LS) from Mouyami (4˚27'34"S; 14˚42'48"E). It has been sieved to retain only the grains smaller than 2 mm. Its

Table 1. Characteristics of the lateritic soil (LS) and the *Cubitermes* mound soil (CMS) used to manufacture earth bricks. W_L = Liquid limit; W_p = Plastic limit; ω_{omc} = Optimum moisture content; γ = apparent density; OM = organic matter.

Soil characteristics	LS	CMS
Clay (%)	31	25
Silt (%)	19	25
Sand (%)	50	50
W_L (%)	27.9	11.6
W_p (%)	9.9	2.1
PI (%)	18	9.5
ω_{omc} (%)	10	15.2
γ (t/m^3)	2.1	1.72
OM (%)	0.54	5.00

geotechnical characteristics have been determined as described in [18], that is, the particle size analysis was carried out in accordance with the NF 94 056 [19] and NF 94 057 [20] standards, and the Atterberg limits, the dry density, and the optimal moisture content were measured in accordance with the NF 94 051 [21] and NF P94-093 [22] standards, respectively. The organic matter content was determined by chemical route using the Walkley and Black method [23]. The results of these analyses are reported in **Table 1**.

For the stabilization of the bricks, five alkaline solutions were prepared (1.5%; 2.5%; 5%; 7.5% and 10% by weight) by dissolving sodium hydroxide pellets with a purity of 98% in distilled water. These NaOH solutions were used instead of the usual tap water for the manufacture of the bricks, at the content of the optimum moisture content determined through the Proctor test. The bricks have been molded by using a mechanical press with a pressure of 6 MPa. A whitish powder appeared on the surface of the stabilized bricks for 7.5% and 10% NaOH solutions. This powder is probably due to the reaction of sodium hydroxide with the air carbonic gas [13].

The volumetric shrinkage rate of bricks at the instant t, $\lambda(t)$, is equal to the difference between the initial volume $V(0)$ and the volume at the instant t, $V(t)$ divided by the initial volume.

2.2. Monitoring and Modeling of the Drying Kinetics

The monitoring of the drying kinetics of the bricks was carried out as described in [18], *i.e.*, the mass of the bricks was recorded until its stabilization. The brick is assumed dry when its mass variation during three consecutive days is less than 2%. For each NaOH solution and soil type, three tests were conducted to check the reproducibility of the results.

Five parametric models have been tested to simulate the drying kinetics of the bricks: the Page-Avrami model [24] [25], the diffusion model [18] [26], the

Khazaei's model [27] [28], the Peleg's model [29] [30] and the Yong's model [18] [31]. The meaning of their parameters has been recalled in [18] and their values are determined by minimizing the reduced chi-square using the origin Pro 8 software. The goodness of the fit is estimated through the value of its coefficient of determination R^2, the root mean square of the error (RMSE) and the Akaike's Information Criterion (AIC). The models' expressions are reported in **Table 2**.

The evaporable water content of the brick at the time t (the reduced mass) $M_r(t)$ is calculated according to the formula:

$$M_r(t) = \frac{M(0) - M(t)}{M(0) - M_f}$$

where $M(t)$ is the brick's mass at the time t and M_f the mass at the end of the drying.

3. Results and Discussion

3.1. Effect of the NaOH Content on the Drying Kinetics and Shrinkage of Bricks

The drying kinetics curves of earthen bricks made with *Cubitermes spp*. mound soil and lateritic soil activated with hydroxide sodium solutions are reported in **Figure 1(a)** and **Figure 1(b)**, respectively.

These curves did not show the constant drying rate phase (a straight line at the start of the curve), but a steady variation of the drying rate, that is, there is almost no free water in the bricks. In other words, these drying kinetics are governed by the diffusion of moisture from the interior to the surface of bricks [25]. This behavior could be linked to the fact that the moisture content used to make bricks is the optimum moisture content (OMC) of the soil determined by the Proctor test. Indeed, the OMC diminishes the friction between soil grains, allowing the soil to have the highest density during its compaction, without being liquid [22]. The drying duration and the initial drying rate deduced from these curves are represented versus the activator content in **Figure 2(a)** and **Figure 2(b)**, respectively. It appears that the difference between the clay contents of the two soils used has not a significant effect on the drying kinetics of the bricks. On the other hand, the activation of soils with NaOH increases the drying duration from about 23 days for non-stabilized bricks to about 28 days for the stabilized

Table 2. Expressions of the models with their parameters. $M_r(t)$ is the evaporable water content of the brick at the time t.

Model	$M_r(t)$	Parameters	References
Page-Avrami	$\exp(-kt^n)$	k, n	[24] [25]
Diffusion	$a\exp(-kt)+(1-a)\exp(-kbt)$	a, k, b	[18] [26]
Khazaei	$1-a(1-\exp(-t/T))-bt$	a, T, b	[27] [28]
Peleg	$1-t/(a+bt)$	a, b	[29] [30]
Yong	$ct^{-\mu}\exp(-t/T)$	c, μ, T	[18] [31]

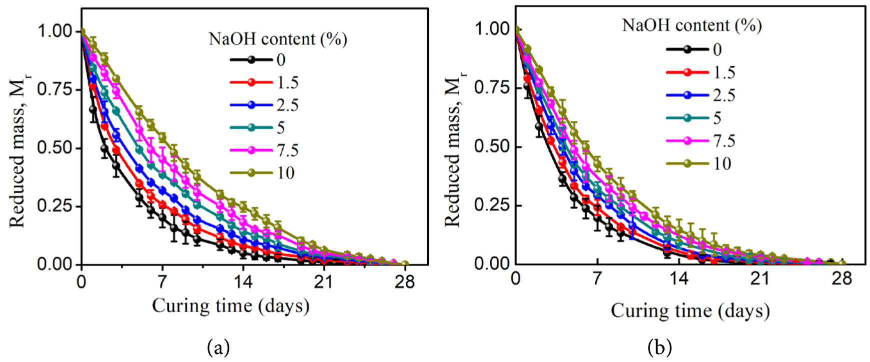


Figure 1. Drying kinetics curves of earth bricks made with: crushed *Cubitermes spp.* Mound soil (a), and lateritic soil (b). The NaOH content in solution was ranged from 0 to 10% by mass. (a) NaOH-activated CMS bricks; (b) NaOH-activated LS bricks.

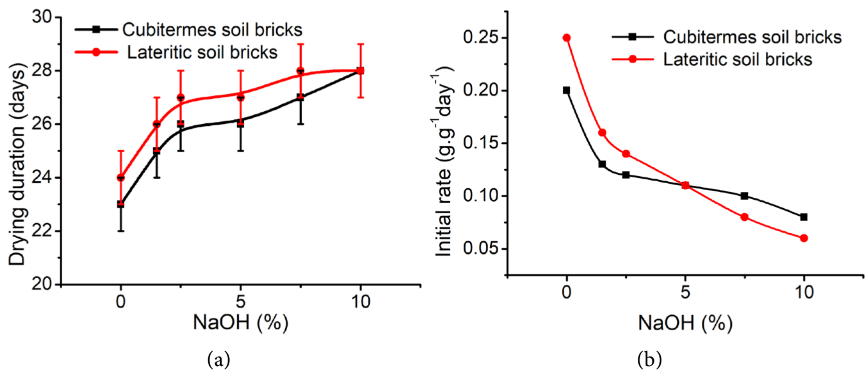


Figure 2. The increasing of NaOH content increases the drying duration (a), and decreases the initial drying rate (b). (a) Effect of NaOH content on the drying duration; (b) Effect of NaOH content on the initial drying.

bricks with the 10% NaOH solution. For the means values and by linear regression, the following relationship was found between the drying duration (D) and the NaOH content (α):

$$D = 24 + 0.8\alpha - 0.04\alpha^2; R^2 = 0.91$$

This lengthening of the drying duration by the NaOH activation could be explained by the tendency of NaOH to form compounds with water, and/or by the formation of the zeolitic structure between the initial soil grains [13] [16] [17]. Indeed, for an alkaline activated mortar made with a natural soil, Kim and al. observed that the formation of zeolitic structure is coupled with the reduction of the mortar porosity [16]. For their part, Diop and Grutzeck reported microstructure transformations during the alkaline activation of a clayey soil [17]. Besides, the drying duration has a strong and negative correlation with the initial drying rate (−0.97). Thus, NaOH activation of soil and stabilization of soil with cassava starch or amylopectin have opposite effects on the drying duration [18]. This variation of the effect of stabilizers on the brick behavior justifies the necessity to evaluate each process to optimize it.

As for the drying kinetics, there is no significant difference between the volumetric shrinkage rate of *Cubitermes spp.* soil bricks and that of lateritic soil bricks (**Figure 3(a)** and **Figure 3(b)**), despite the difference in clay content. The NaOH activation reduces significantly the shrinkage rate of bricks from about 5% for non-activated soils to 3% for soil activated with the 10% NaOH solution. This reduction of the shrinkage rate could be due to the formation of the zeolitic structure [13] [16] [17] that reduces the brick porosity as above mentioned. Besides, manufactured earth bricks did not show visible cracks. In fact, the increasing of the drying duration combined with the shrinkage reduction would reduce the appearance of cracks in bricks, and therefore would contribute to the increase of their strength. Indeed, slower lost of water, and weaker shrinkage generate less internal stresses, and thus the appearance of cracks in the bricks.

The shrinkage decrease observed here is lower than that induced by the addition of lime [6] and plant fibers [32] [33], while it is higher than that produced by the addition of cement [34] or polyethylene strap [35]. However, in average, the improvement of the earthen brick strength due to the addition of lime [6] [36], or plant fibers [32] [33] is lower than that reported for the NaOH activated soils [16] [17].

3.2. Evaluation of the Models

The reduced water contents predicted by the models are represented as functions of those measured in **Figure 4**. To evaluate the quality of these predictions,

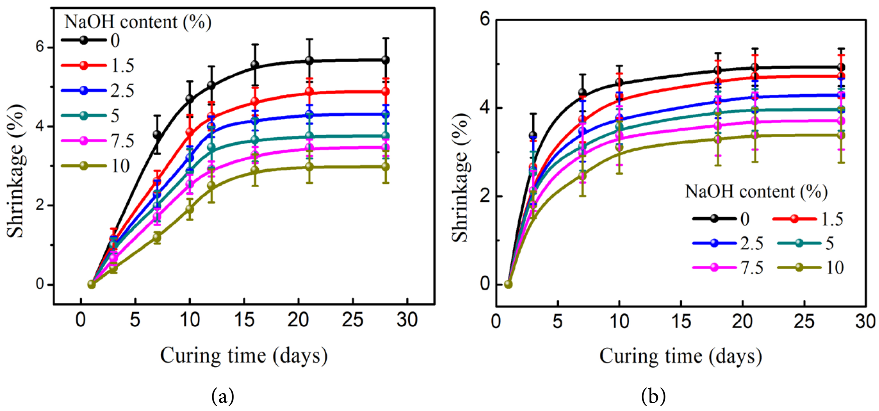
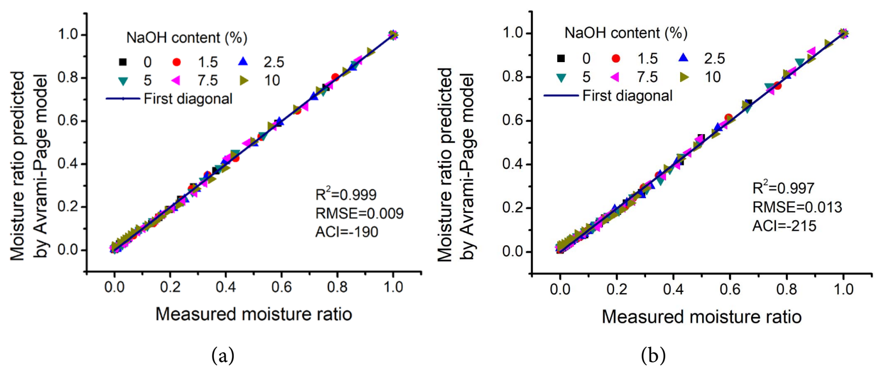


Figure 3. Effect of NaOH content on the volumetric shrinkage rate of CMS bricks (a) and LS bricks (b). For all bricks, the increasing of NaOH content reduces the shrinkage rate. (a) Shrinkage of CMS bricks; (b) Shrinkage of LS bricks.



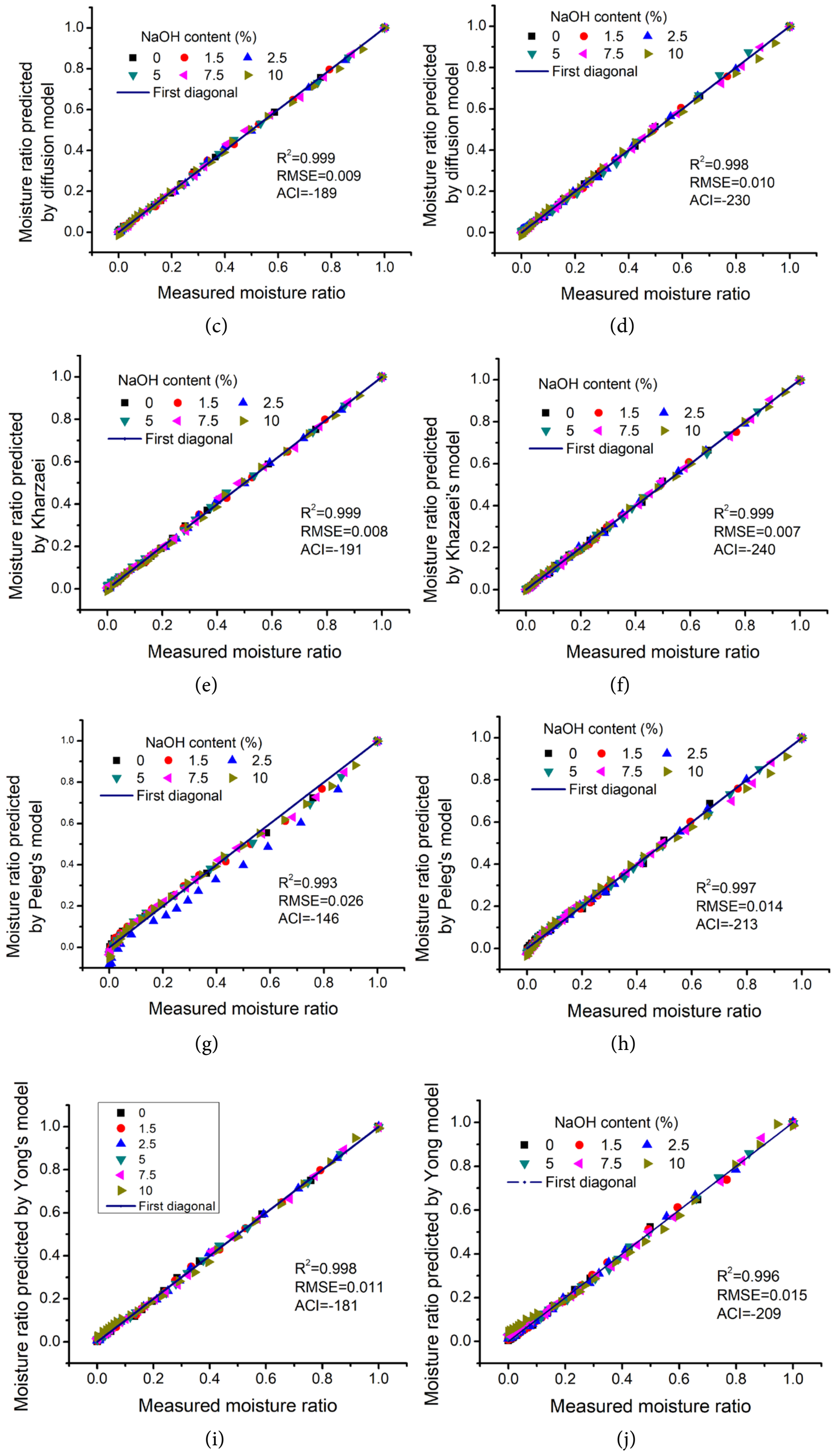


Figure 4. Comparison between the moisture contents predicted by the models and those measured. The quality of the predictions is evaluated through the coefficient of determination R^2, the root mean square of the error (RMSE) and Akaike's information criterion (AIC). (a) Avrami-Page model on CMS bricks; (b) Avrami-Page model on LS bricks; (c) Diffusion model on CMS bricks; (d) Diffusion model on LS bricks; (e) Khazaei's model on the CMS bricks; (f) Khazaei's model on the LS bricks; (g) Peleg's model on CMS the bricks; (h) Pelegs's model on the LS bricks; (i) Yong's model on the CMS bricks; (j) Yong's model on the LS bricks.

the mean values of the coefficient of determination (R^2), the root mean square error (RMSE), and the Aike's information criterion (AIC) for all models tested are also reported on these figures. For each model and any drying kinetics curve, the coefficient of determination is greater than 0.992, and the RMSE is less than 0.03. Therefore, all these models could be considered suitable to simulate this drying kinetics. However, the Khazaei model has better results than the others (**Table 3**), for all statistical criteria. As it has already proved to be the best for simulating the drying kinetics of earthen bricks stabilized with other binders [18], it seems very suitable for modeling the drying kinetics of compressed earth bricks.

The values of the parameter n (the exponent of the time) of the Page-Avrami model and those of the khazaei's model (**Table 4** and **Table 5**) are on average equal to 1. These values are consistent with the shape of the drying curves which shows a steady decrease of the drying rate. However, there is a slight increase in the value of n with the increase of NaOH content. All factors affecting negatively the drying rate could contribute to this increase, and in particular, the decrease of porosity by the formation of zeolitic structure, the appearance of the flour on the brick surface due to the NaOH-CO_2 reaction. Indeed, as we have already reported in the material and methods part, for the bricks activated with 7.5 and 10% NaOH solution, a whitish powder appeared on the surface of the bricks.

Table 3. The ranking of the goodness of fit of the different models according to the statistical criteria.

	CMS bricks			LS bricks		
Rank	R^2	X^2	**AIC**	R^2	X^2	**AIC**
1	Kharzaei	Kharzaei	Kharzaei	Kharzaei	Kharzaei	Kharzaei
2	Diffusion	Diffusion	Avrami	Diffusion	Diffusion	Diffusion
3	Avrami	Avrami	Diffusion	Avrami	Avrami	Avrami
4	Yong	Yong	Yong	Peleg	Peleg	Peleg
5	Peleg	Peleg	Peleg	Yong	Yong	Yong

Table 4. Values of the models' parameters applied to drying kinetics of *Cubitermes spp*. mound soil bricks activated with NaOH solutions.

	Page-Avrami		Diffusion			Yong			Khazaei				Peleg	
NaOH (%)	*k*	*n*	*a*	*b*	*k*	*c*	*T*	*u*	*a*	*b*	*n*	*T*	*a*	*b*
0	0.28	0.92	0.8	3.3	0.21	0.94	4.40	0.01	1.02	−0.001	0.90	4.2	2.7	0.87
1.5	0.22	0.97	1	4.5	0.21	0.98	4.90	0.01	1.03	−0.001	0.95	4.9	3.5	0.84
2.5	0.17	1.04	−13	1	0.14	1.03	5.40	−0.01	1.02	−0.001	1.03	5.8	4.4	0.80
5	0.15	1.06	0	22	0.01	1.04	5.89	−0.01	0.96	0.002	1.06	6	5	0.79
7.5	0.12	1.08	−5	1.1	0.10	1.04	6.50	−0.01	0.98	0.001	1.05	7	5.9	0.76
10	0.08	1.18	−29	1	0.07	1.09	7.15	−0.02	1.07	−0.001	1.10	9.1	7.8	0.67

Table 5. Values of the models' parameters applied to drying kinetics of lateritic soil bricks activated with NaOH solutions.

	Page-Avrami		Diffusion			Yong			Khazaei				Peleg	
NaOH (%)	k	n	a	b	k	c	T	u	a	b	n	T	a	b
0	0.39	0.76	0.7	8.3	0.19	0.78	5.30	0.04	1.1	−0.003	0.67	4.7	2.7	0.87
1.5	0.25	0.84	0.8	5.4	0.16	0.87	6.00	0.03	1.0	0.000	0.80	4.9	3.5	0.84
2.5	0.22	0.88	0.9	7	0.14	0.91	6.76	0.02	1.1	−0.002	0.80	6.9	4.4	0.80
5	0.14	1.00	−0.1	54	0.01	0.98	7.35	0.01	1.4	−0.007	0.87	10.9	5	0.79
7.5	0.09	1.14	−14	1.1	0.07	1.06	7.78	−0.01	1.1	−0.001	1.04	9.7	5.9	0.76
10	0.05	1.28	−29	1.1	0.05	1.12	8.42	−0.03	1.0	0.003	1.19	10.7	7.8	0.67

The values of the parameter k of the Page-Avrami model, those of $1/T$ of the Khazaei's model, and the inverse of the parameter a of the Peleg's model decrease when the NaOH content increases. This result is consistent with the fact that the first two parameters are directly related to the time after which 63.2% of water is evaporated, and the third with the initial drying speed.

The values of the parameter K of the diffusion model obtained here are larger than those obtained earlier [18] because the drying durations measured here are shorter than the previous ones.

4. Conclusions

The objective of this work was to study the effect of NaOH activation on the drying kinetics of earthen bricks and to test five parametric models on this kinetics.

The results obtained show that:

1) There is not a drying rate constant phase during the drying, and therefore the drying kinetics is governed mainly by the diffusion of water from the interior to the surface of the brick. The drying duration increases almost linearly with the NaOH content. It varies from about 23 days for non-activated bricks to 28 days for activated bricks with a 10% NaOH solution. This increase could be explained by the formation of the zeolite structure that reduces the soil porosity, the NaOH-H_2O affinity, and the NaOH-CO_2 reaction;

2) NaOH activation reduces the volumetric shrinkage rate of bricks for the soils studied. It varies from about 5% for non-activated bricks to about 3% for the activated bricks with 10% NaOH solution. The reduction of the porosity due to the formation of a zeolitic structure could explain this shrinkage decrease. Manufactured bricks did not show visible cracks;

3) The coefficients of determination of the five models range from 0.993 to 0.999. The Kazaei's model is the best of all. The parameter n of Avrami-Page and Khazaei models is about equal to 1, in accordance with the absence of free water in the bricks. This parameter increases slightly with the NaOH content. The evolution of the parameters k of the Page-Avrami and diffusion models, that of the

parameters T and a of the Kharzaei and Peleg models, respectively, are in agreement with the variation of the drying duration due to the NaOH content.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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