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Method for avoiding cracks during drying of masonry units made of illite raw material

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Abstract. Drying is one of the most important steps in the production process of masonry units. In order to prevent the formation of cracks during drying information's about the moisture migration rate variability as well as the material strength variability through drying are necessary. The main goal of this paper was to find a solution how to prevent the crack formation at the beginning (during the first hour) of the drying for the drying sensitive illite raw material. The first step was to record a series of isothermal Deff – MR curves at different drying air temperatures and constant drying air velocity and humidity. As it was already reported all moisture transport mechanisms during isothermal drying are visible on those curves. Characteristic spots registered on these curves were then transposed on the experimentally registered figure material strength us moisture content. It was found that the material strength for the cracked masonry units at the beginning of drying was around 0.09 MPa and that the time of cracking was near the characteristic spot B. Registered material strength and the crack time position (spot B) has additionally confirmed that the drying sensitivity of the raw material are obviously related with the present clay mineral constituents structure and the initial moisture content of the green heavy clay units. Using the Deff values registered for each experiment in the spot B we were able to calculate the maximal moisture transport rate and consequently the proper drying air parameters which are safe and which will not initialize the formation of the cracks at the beginning of the drying.

1. Introduction

The stresses, which are inevitably generating during drying as a result of the large difference between the surface evaporation rate and the moisture flow rate towards the surface of the green masonry units or the lack of the clay bonding minerals, are one of the main reasons for the plastic - viscoplastic deformations (warping or twisting) as well as the crack genesis. In other words, any kind of non-uniform shrinkage, which appears during drying, is caused by the non-uniform distributions of the temperature and/or moisture content and is related with the generation of the semi-stresses inside the green masonry unit [1].

In order to overcome the potential difficulties during drying of heavy clay units two measures can be applied. The first one is to reassess the row material (clay mineral content, particle size distribution, plasticity and drying sensitivity) and to adapt its composition due to the either an excess or deficiency of the plastic bonding (clay) minerals. The second one is to change the drying regime and to carefully regulate the drying conditions in accordance with the recommendations which arose from the recently reported author's theory of moisture transport during drying and the nature of the raw material [2-4].

The second measure is applied in this study. The main goal of this paper was to find a solution how to prevent the crack formation at the beginning (during the first several hours) of the drying for the

drying sensitive illite raw material. A part of the preliminary research is presented in this paper. It is based on the idea that the strength of the green heavy clay unit, and its correlation with the non-uniform shrinkage related deformations and cracks, is dependent on the moisture content and drying temperature. It is important to express, that in accordance with the current stand points of several common drying theories and its corresponding mathematical models, non-uniform maximal shrinkage gradient is expected to be registered at the moment which corresponds to the end of the surface shrinkage [5-7]. According to the previously mentioned theory [2], this moment can be easily identified on the curves Deff – MR as the characteristic spot E. However, the highest drying susceptibility for the crack formation is in some cases experimentally registered much earlier, (in the vicinity of the characteristic spot B) especially if the green masonry units were formed from the illite / smectite raw material. Previously mention facts are obviously contradictory and can be explained if the green masonry unit strength is assumed to be dependent on the moisture content.

2. Materials and Methods

The raw material, used in this study, was obtained from the Serbian brick producer. Its characterization has included: classical silicate analysis, XRD, particle size determination, and dilatometry test. The raw material was then crashed down in the pan mill until the whole content has passed through the sieve of 5 mm. After that the crashed material was simultaneously moisturized and further milled, using the laboratory dual rotor crusher which gap was first set to 3 and then to 1 mm. Laboratory tiles (120 x 50 x 60 mm) and cubes (33 x 33 mm) were formed, from the previously homogenized clay in the extruder "Hendle" type 4, under a vacuum of 0.8 bar. Formed tiles were packed into plastic bags which were afterwards sealed. This was done to prevent the moisture content fluctuations within the stored samples.

The process of drying characterization was carried out on these tiles in a specially constructed laboratory dryer. In each experiment the drying air velocity was set to 1 m/s. The tile mass and the corresponding linear shrinkage were continually monitored and recorded during drying. The accuracies of these measurements were 0.01g and 0.2mm. Drying air parameters were regulated inside the dryer with accuracies of $\pm0.2°C$, $\pm0.2\%$ and $\pm0.1\%$ for temperature, humidity and velocity, respectively.

The first task was to determine the critical drying rate. The drying air parameters, which were kept constant during drying, were carefully chosen. In each experiment three samples were dried at the same time. Since most dryers, operates with the hot air which has approximately $25gH_20/kg$, this value was used in the calculation process. The initial cooling limit temperature was 20^0C. The starting psychometric temperature difference, which represents the difference between the dry bulb temperature and the cooling limit temperature, of 6^0C was set in the first experiment. If cracks were not detected on dried tiles after the first experiment was over, the psychometric temperature difference was doubled in the second one. If cracks were detected in the second experiment the psychometric temperature difference was decreased to 3^0C. This procedure was repeated several times and the maximum allowable drying rate was easily determined.

According to the author's theory Deff – MR curves can be used for identification of all active mechanisms of moisture transport and their transitions during isothermal drying. The calculation procedure was specified in article [2]. The second task was to record a series of isothermal drying curves and to calculate the corresponding Deff – MR curves. Three sets of isothermal experiments were recorded. Drying air humidity was kept constant at 80, 70 and 60% respectively in each corresponding isothermal set. The drying air temperature in the first experiment of each series was set to 20^0C. The drying air temperature was raised for 5^0C in each following experiment.

The third task was to detect the influence of moisture content on material (compressive) strength. Formed cube samples were used for this characterization. The moisture content in the formed cube was first detected. Three cubes were dried isothermally (25^0C, 70%, 1 m/s) in the dryer. These experiments, has allowed us to calculate the drying time (min) of reaching any desired moisture content within the initial (formed) and the equilibrium one. Twelve different moisture contents were

specified. Five cubes were dried up to the each previously defined moisture content. After that each set of cubes was packed and stored for 24h into the glass container. Glass containers were kept in the air-conditioned room at 25^0C. The compressive test was carried out on these samples. The press loading rate was maintained at 2 N/s.
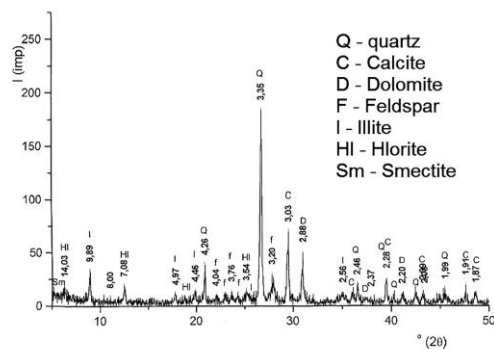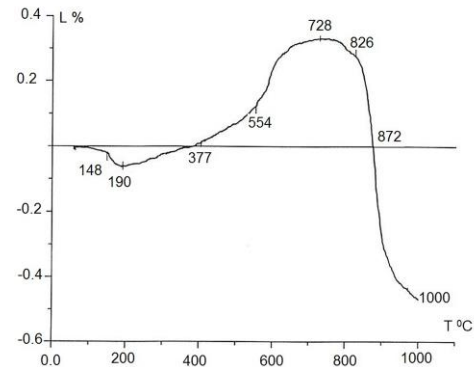
3. Results and discussion

XRD and chemical results (figure 1 and table 1) are pointing out that the analyzed raw material has predominantly the illite character. Beside illite, small quantities of hlorite and smectite minerals were also detected. Quarc, plagioclase, carbonate, iron hydroxide and organic material were also registered. According to the sieve test the analyzed raw material is classified as alevrite clay with very low sand content.

Table 1. Results of chemical and sieve analysis.

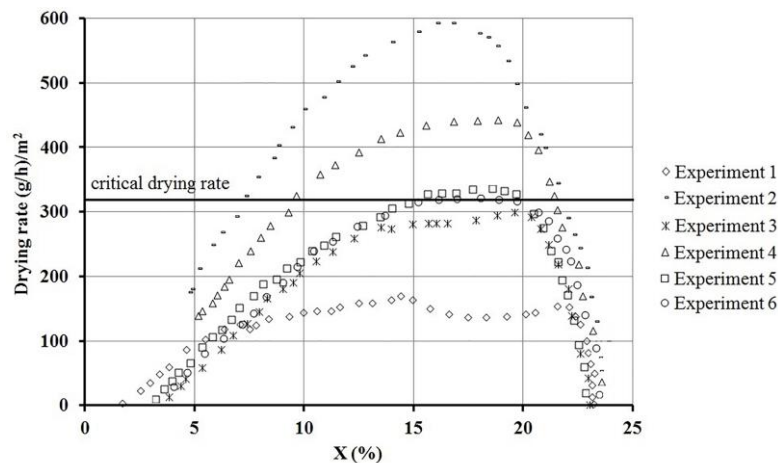| *Chemical analysis*	Raw material	*Sieve analysis*	Raw material
Composition	%	Raw material fractions	%
Loss ignition on 1000^0C	7.31	Send (>20μm)	1.25
SiO_2	46.30	Alevrit (2-20μm)	54.60
Al_2O_3	15.04	Clay (<20 μm)	44.15
Fe_2O_3	4.68		
CaO	11.67	Fractions in mm	%
MgO	3.93	0.125	0.00
SO_3	0.03	0.063	0.95
S^{2-}	0.01	0.050	0.30
Na_2O	0.70	0.020	12.85
K_2O	1.65	0.010	19.35
MnO	0.10	0.005	22.40
TiO_2	0.05	0.001	44.15
CO_2	8.78		
Organic material	0.19	Mean particles	mm
Summary:	100.44		0.011

Table 2. Technological properties of the raw material during forming and drying process.

Forming process				Drying process	
The amount of water for forming in (%)	23.25		Bigot's curve	Shrinkage at critical point (%)	4.88
Plasticity according to Feferkorn	Plasticity index	28.95		Water loss at critical point (%)	9.05
	Plasticity criteria	Good plasticity	Drying sensitivity according to Piltz	Time when first crack is appearing (min)	4.1
				Drying criteria	Sensitive to drying (4-6)

Figure 1. XRD curve.
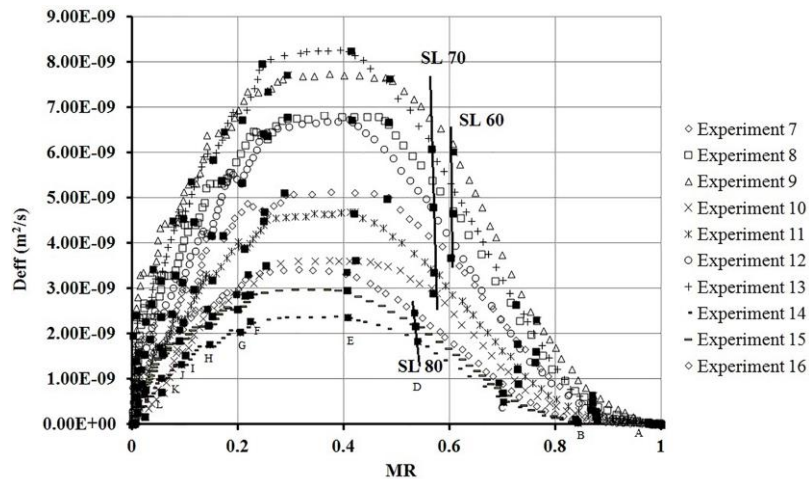

Figure 2. Dilatometry curve.

Results presented in table 2, have indicated that the formed mass has good plasticity. Detected plasticity index is similar as the recommended value for brick production which was reported in the study [8]. Although Bigot's curve and Piltz method provide different values of the drying sensitivity, it is more acceptable to classify the analyzed ceramic body as highly sensitive. These findings are also in accordance with the mineralogical composition of clay, due to the fact that in the analyzed ceramic mass the illite and smectite minerals are dominant. Based on the dilatometry and chemical test results (figure 2 and table 1) the firing temperature of 950^0C was recommended.

It was found that only 6 experiments were sufficient for determination of the critical drying rate. This was additional confirmation that with only a few psychometric temperature difference iterations, previously mention limiting drying rate can be easily calculated.
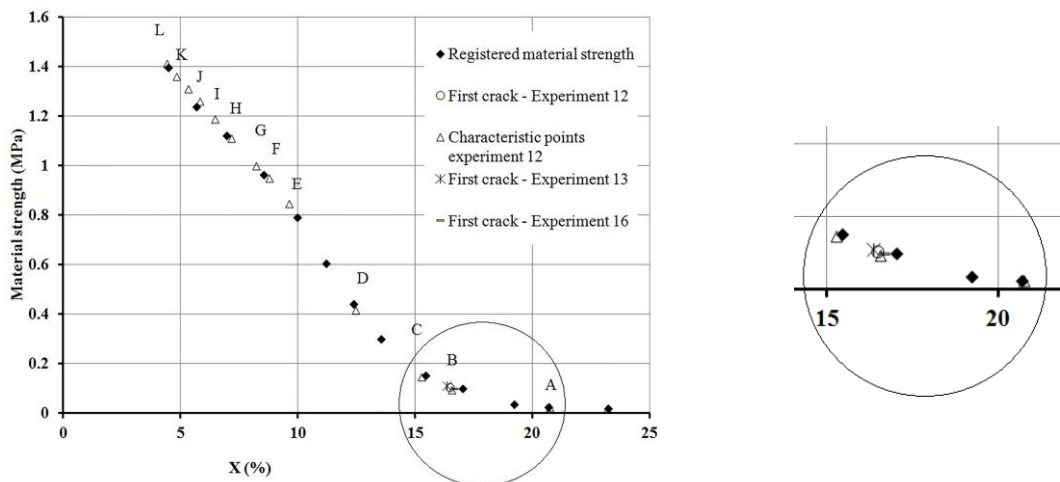

Figure 3. Drying rate curves.

From figure 3 it can be seen that the critical drying rate in the first drying phase for the tested raw material was 318 $(g/h)/m^2$. Curves Deff – MR, which were constructed using the calculation method outlined in reference [2], are presented in figure 4. Moisture transport mechanisms that are active within each drying segment, identified on the previously mention figure (as black spots) are summarized in the reference [3]. It is important to note that only characteristic points which are related to the experiment 7 were additionally labeled as an example, using the same designation code (numbers A - L) as it was recommended in the reference [2]. This was done to prevent the overlapping of Deff - MR curves with its indentified corresponding designation numbers. The same characteristic point D, belonging to different experiments of the same series (I, II or III), were connected (SL-80,

SL-70 and SL-60) as an example on the figure 4. It is important to say that SL lines obtained for different characteristic points belonging to the same experiment series are parallel to each other. This "parallel" pattern is very important, especially for experiments in which samples had cracked. Cracks were not detected on tiles which belonged to the first isothermal experimental series. This was expected due to the fact that the critical drying rate was not exceeded in those experiments. Cracks were detected on tiles in experiment 12, 13 and 16. Deff – MR curves were constructed for these three experiments using the SL pattern in accordance with the procedure which is valid for cracked samples and was firstly reported in the reference [3].

Figure 4. Deff – MR curves.

The test results of all performed compressive tests were presented in figure 5. The material strength of the analyzed raw material evidently depends on moisture content. The shape of the curve is suggesting that the material strength of illite clay has the highest value in the dried state and with the increase of moisture content it will decrease rapidly. It is very interesting to note that if the moisture content is higher than 15% the material strength is below 0.2MPa. These findings are comparable with the results reported for illite clay in the Banaszak study [9].

Figure 5. Registered material strength – moisture content curve.

In other words, the material strength of the green clay element formed of the illite raw material is small during drying until the moisture content of 15% is reached. This is closely related with the clay nature (its mineral composition) and is a good indicator that this type of clay is sensitive to drying. This result corresponds well with the previously reported values of the Bigot and Piltz drying sensitivity.

The illite clay minerals are layer silicates mostly characterized by structural motive formed by two kinds of units. The first kind is formed by SiO_4 tetrahedrons constituted by silica atom in the centre of the four oxygen ions at vertexes. The tetrahedral groups are then bonded ones other to form a hexagonal ring, which can repeat them self indefinitely. Tetrahedrons are coplanar with a face, so that free vertexes are all directed to the other part. By this way two layers of oxygen atoms are formed. A layer of silica atom is embedded between them. The second kind of structural unit consists of aluminum, iron, potassium atom layer, to which atoms of oxygen and OH^- are tetrahedrally coordinated in two parallel layers. The first one is above and the other is below the one formed by aluminum, iron and potassium. The combination of the previously mentioned structural motives type 2:2:1 (three layers one octahedric, included between two tetrahedric with spacing of 10 Å) spaced by potassium cations and water molecules represents the illite clay structure [10].

The distance between two tetrahedric layers or two successive sheets is causing a very weak bond between single structural units (only the Van der Waals strengths). That allows the interposition of water or other kind molecules (mostly organic one) into the illite structure. This is usually followed with a consequent crystalline lattice extension. The material strength up to the optimal moisture content is in the case of illite clay mainly influenced by the previously mentioned forces. If more water is added to the raw material than the optimum one, continuous fall of the cohesion forces between grains will cause the rapid decrease of the material strength value since too much water will cause liquefaction of the ceramic mass.

During drying from the optimal moisture content (formed one) up to the approximately 15% the distance between structural layers will still be relatively unchanged and the corresponding material strength will have small values due to the fact that the surface Van der Waals bonds are very weak. That is the reason why the crack susceptibility is maximal in this early drying phase. As drying continuous the distance between grains will be decreased and the surface bonds will become stronger. This effect will continually increase the material strength.

Characteristic spots registered on the curves Deff-MR for samples that has cracked in experiment 12 were transposed on the experimentally registered figure material strength us moisture content as an example. The moisture content, which corresponds to the moment in which the first crack was registered in experiment 12, 13 and 16, were also transposed on the previously mentioned curve. It was found that the material strength for the cracked masonry units was around 0.096 MPa and that the time of cracking was near the characteristic spot B.

According to the common drying theories, the stresses which are generating during drying are caused by the discrepancy between the surface evaporation rate and the moisture flow rate towards the surface of the green masonry units or the absence of the clay bonding minerals. The peak of shrinkage differences occurs namely when the shrinkage at the surface has reached its final measure. That is the reason why it is expected that the non-uniform maximal shrinkage gradient is observed at the moment which corresponds to the end of the surface shrinkage [5-7] (characteristic spot E). However, the highest drying susceptibility for the crack formation exists for illite /smectite clays precisely at the start of drying. In our case the first crack was experimentally registered in the vicinity of the characteristic spot B. Previously mention facts are obviously contradictory and can be explained if the green masonry unit strength is assumed to be dependent on the moisture content. As we have already explained, the material strength of the green heavy clay elements will be relatively small due to the presence of surface Van der Waals bonds during drying until the moisture content of approximately 16.3 % is reached (see figure 5.) The drying rate calculated for experiments 12, 13 and 16 was higher than 318 $(g/h)/m^2$. Due to the fact that the moisture rate toward the surface is higher than the critical

one the stresses which are generating in this drying segment will over cross the limiting material strength value 0.096 MPa and the cracks will be formed.

It is important to say that the nature of the raw material (illite/smectite) and the relatively small value of the material strength until the moisture content of 16.3 is reached, is dictating the proper drying air parameters. In order to dry this kind of clay elements the drying air humidity has to be higher as much as possible until the previously mentioned moisture content is reached. According to the experiments the maximal drying temperature in this drying segment should be 30^0C. When the moisture content of 16.3% has been reached simultaneously the drying air humidity should be decreased and the drying air temperature should be increased.

4. Conclusions

One of the major problems which arise during drying of the green elements formed of the illite/smectite raw material is to set up the proper drying air parameters and to prevent the crack formation, especially in the first several hours of drying. The generation of the semi-stresses inside the green clay elements during drying is caused by the non-uniform distributions of the temperature and/or moisture content. Even though it is expected that the non-uniform maximal shrinkage gradient is observed at the moment which corresponds to the end of the surface shrinkage the highest drying susceptibility for the crack formation exists for illite /smectite clays precisely at the start of drying. It has been experimentally confirmed that the moisture and temperature dependences are related with the otherwise highly material-dependent strength parameters. Reported results allowed us to form the general statement how the tendency for crack formation can be prevented in the early phase of green elements drying. In this drying phase, the drying air humidity has to be higher as much as possible, while the maximal drying temperature should be 30^0C.

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