Novel method suitable for decreasing the roofing tile failures generated during rapid drying

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Abstract. Only few papers in which principles for decreasing the failures generated during semi and rapid drying of porous shrinking materials, for example roofing tiles, are reported. One of the main differences between the traditional and rapid drying regimes is related with the fact that during the heating step products are heated to the much higher temperatures during rapid drying. If the humidity inside the industrial drying tunnels or chambers is not controlled all the time in the prescribed range of 90 - 95 %, especially during the heating step of the rapid drying regime, crack failures will be developed. In order to inhibit the cracks generation during intensive drying the non-ionic surfactant Igepal CO-630 was added in the raw material during forming process. Various amounts of surfactant around the critical micelle concentration (CMC) were used. Five isothermal experiments were recorded, on laboratory extruded roofing tiles, for each surfactant concentration. Calculated effective diffusivity and material strength - moisture ratio (Deff-MR & MS-MR) curves were used to monitor the development of the cracks during drying and to determine the most suitable concentration of the surfactant. The explanation, how the interaction of the surfactants with clay helps the water molecules to easily move up to the surface was also reported. The application of surfactant in a recommended amount has significantly reduced the drying induced fractures in roofing tiles during its intensive drying and consequently the industrial scarp-rate was accordingly lowered.

1. Introduction

The unwanted shrinkage of the green roofing tiles during intensive drying can cause significant reduction of the products material strength and in some cases can lead to the formation of cracks. This phenomenon is directly related with the drying induced stresses. These stresses are always generating within the products when the amount of bonding clay minerals in the raw material is insufficient or moisture - temperature distributions becomes nonlinear [1,2]. Even though different methods and modeling techniques as well as specialized software’s for setting up the safe and optimal levels of drying air parameters inside the various types of industrial roofing tile dryers were recently reported by authors, its efficiency is still directly correlated with the measuring and regulation equipment installed in the dryer, especially if the rapid drying regimes are applied [3-5]. The positive example of industrial semi-rapid drying regime, in which stable control of air humidity from 82 - 88% was achieved within the first drying phase, was recently reported [6]. The impossibility to practically control and keep the humidity inside the industrial drying tunnels or chambers during rapid drying, in the theoretically prescribed range of 90 - 95 %, all the time will inevitably lead to the cracks genesis.

The main goal of this paper was to find a solution how to decrease the roofing tile failures generated during the intensive heating phase of the rapid drying regime.
Several measures are available. The first one is to adapt the row material composition by adding the clay or sand fraction in order to change overall plasticity or drying sensitivity. The second one is to upgrade or install the new measuring and regulating equipment, capable to fulfill all theoretical demands related to the rapid drying as specified in [4,6], inside the dryers. The third measure is to improve the moisture transport from the inside of the drying products and to make moisture distribution more uniform. This can be achieved using different surfactants.

Kowalski and his researcher group investigate the effect of several surfactants (Rokacet R26, Rokanol IT6, SDS and FC 4430) on the reduction of drying induced stresses inside the kaolin-clay products during drying. The acoustic emission method was used to monitor on line the development of crack formation in dried samples. The best quality of the dried products was achieved for clay saturated with water containing low concentration of surfactants below but close to the critical micelle concentration (CMC). The main conclusion was that the addition of surfactant to water, used for wetting the clay prior to the forming process, will significantly decreases the surface moisture tension allowing much easier moisture movement from the inside of the capillary-porous skeleton [7,8]. Gulzena has used non-ionic surfactant Triton TX-100. In this research clay bricks were made from the raw material in which illite clays were dominant. Digital camera was used to record the drying process. Obtained pictures were used for measuring the crack development. The smallest number of cracks was registered when the surfactant concentration was near the CMC [9].

Biswald's has compared non-ionic surfactants Triton TX-100 and Igepal CO-630. He has reported that the Hamaker constant and work of adhesion decrease gradually with increasing surfactant concentration and ultimately reach plateau regions above the CMC. It was also stated that the Igepal CO-630 has a lower CMC value (0.08 mmol/l) and better wetting properties at low concentrations than Triton TX-100 (0.15 mmol/l) as well as comparable properties near the CMC. The main conclusion was the statement that Igepal CO-630 was proved to be a more beneficial wetting agent than Triton TX-100 [10].

This was one of the reasons why previously mentioned non-ionic surfactants was selected and used in this study. The second one was the fact that the influence of non-ionic surfactant concentrations, just above the CM on the reduction of the drying induced stress was not investigated in detail even though in some references can be found that if unsaturated soil is treated with surfactant solution suction consistency is reduced with the increasing of surfactant concentration [11].

2. Materials and Methods

The raw material was taken from the production line of the Wienerberger's roofing tile factory situated in Kanjiza. Classical silicate and particle size analysis, qualitative and semi-qualitative X-ray analysis which corresponds to the used raw material, was reported earlier in the reference [12]. The row material was first grinded in the pan mill. After that the simultaneous moisturization and milling in the dual rotor crushers were applied. During the moisturization step surfactant Igepal CO-630 CAS number 9002-93-1 was added as defined in table 1. Five sets of roofing tiles (120 x 50 x 60 mm) and cubes (33 x 33 mm) were then extruded on the laboratory press under a constant pressure of 80 kPa.

| Table 1. Surfactants concentration. |
|-------------------------------------|
| SC set | I | II | III | IV | V |
| Concentration (%) | 0 | 0.1 | 0.05 | 0.005 | 0.0001 |

Formed samples were closed in plastic bags and kept at room temperature. Five isothermal experiments were recorded on roofing tiles for each SC. The corresponding drying air parameters are defined in table 2. These parameters were controlled with accuracies of ±0.2 °C, ±0.2 % and ±0.1%. The sample mass and linear shrinkage were registered too. The accuracies of these measurements were 0.01 g and 0.2 mm. The Arabic numbers were used for the identification of the isothermal experiments while Roman numerals were used for identification of the surfactant concentration as presented in table 1. Obtained data were used for the construction of Deff-MR curves.
Table 2. Drying air parameters.

| Experiment | 1   | 2   | 3   | 4   | 5   |
|------------|-----|-----|-----|-----|-----|
| Temperature (°C) | 30  | 35  | 40  | 45  | 50  |
| Humidity (%)    | 90  | 90  | 90  | 90  | 90  |
| Velocity (m/s)  | 3   | 3   | 3   | 3   | 3   |

Three cubes picked from the same SC set were dried isothermally at (25°C, 70%, 1 m/s). MR - t curves were plotted. Total drying time was divided on ten identical intervals. Five cubes taken from the same SC set were then dried up to each defined moisture content. Dried cubes were closed and kept for 24h at 25°C. The compressive test was determined on these samples in accordance with the SRPS EN 771-1. Deff – MR curve was then formed. Described procedure was repeated for each SC set.

3. Results and discussion

Results presented in the reference [12] (tables 2 and 3) have classified the raw material as silt loam. Illite, montmorillonite, chlorite, kaolinite, large amounts of calcite and dolomite, quartz, micas and feldspar were identified in the raw material. Illite and muscovite were marked as dominant clay minerals.

The structure of surfactant solution depends on the surfactant concentration. In many applications the CMC is defining the limiting concentration for meaningful surfactant use. This value is important since the surface tension does not reduce further when concentration of surfactant is above CMC. When the CMC is reached the molecules are arranged in a monomolecular form. The micelle formation is shown in figure 1. The CMC value for the Igepal CO-630 was taken from the reference [10] as 0.08mmol/l (49.36mg/l or 0.005%). The particle size distributions registered in SC II-V sets were slightly different than the one registered in SC-I set. This was expected result. Similar trend was also reported in reference [9].

Cracks were detected in experiment 5. All possible mechanisms of moisture transport and their transition from one to another during the constant and the falling drying period were identified on Deff – MR curves using the procedure outlined in references [3-4]. The construction of SL lines on Deff-MR curves for the samples which have cracked during drying was reported in reference [4]. Resulting five curves corresponding to experiment 1 and 5 were given as an example. The overcrowded figure could obstruct the reader. That was the reason why only the Deff – MR curves, detected in experiments 2-I, 3-I and 4-I, were reported on figure 2. By doing that the characteristic marks, and the corresponding SL lines were also more visible allowing the reader to become more focused on receiving the comprehensive and complete information about the history of moisture transport during drying.
In our previous papers it was stated that in the case of raw materials in which illite-smectite clays are dominant the most critical drying segment is at the beginning of the drying in the vicinity of characteristic point B [13]. The moisture transport registered in experiments in 1-II, 1-III, 1-IV and 1-V up to the mark C is slightly lower than the one registered in 1-I. The overall stresses in this segment, in the products containing surfactants, are slightly neutralized as a consequence of the more uniform temperature distribution within the products. In other words the drying susceptibility for the crack formation is reduced in this segment. This is very important since this drying segment was in our previous study identified as critical [13].

The overall moisture transport registered in experiments 1-II, 1-III, 1-IV and 1-V after the mark C is higher than the one registered in the products formed without surfactant addition. This is a direct confirmation that the presence of surfactant has improved the internal moisture transport. If individual Deff – MR curves for example from the experimental 1 are compared, it can be seen that the maximal increase in overall moisture transport during drying was registered in experiment IV. This means that surfactant concentration slightly above or close to the CMC concentration provides the maximum adsorption to the clay surface. It is important to mention that the drying behavior pattern reported in experiment 1 was registered and confirmed in all other experiments.

Calculated MS curves are presented in figure 3. The shape of curves reported for experiments I-V was the same as one registered in references [13, 15 and 16]. Regardless to the presence or absence of the surfactant the material strength is small during drying until the MR of 0.84 is reached. This MR value corresponds to the moist content of 14.88%. This is another confirmation that this type of raw material is sensitive to drying especially at the beginning. The addition of surfactant to the raw material is evidently affecting the material strength. This effect is directly correlated with the surfactant concentration. The highest material strengths were registered for the surfactant concentration below or near the CM. These findings are in agreement with the one obtained from the figure 2.

The adsorption of no-ionic surfactants such as Igepal CO-630 is attributed to hydrogen and hydrophobic bonding. If hydrogen bonding is responsible to adsorption the bond that is formed between the surfactant functional groups and clay mineral surfaces must be stronger than the bond that is formed between the mineral and interfacial water molecules [14]. An adsorption on solids that possess a fully or partially hydrophobic surface takes place mainly due to hydrophobic bonding. For kaolinite the two surfaces have different structure. The siloxane surface interacts very weakly with interfacial water molecules, while the hydroxyl surface interacts strongly with water through hydrogen
bonding. The illite siloxane surface has introduced a permanent change as a result of isomorphous substitution. Its surface displays a degree of charge imbalance as a result of the ion substitutions in the octahedral and tetrahedral sheets. That is the reason why illite hydrogen reaction with the surfactants is stronger than in the case of kaolinite. Since substitutions are more extensive in montmorillonite it is assumed that the absorption power is larger than for illite or kaolinite minerals. Direct relationship between the adsorption of the nonionic surfactant and the amount of clay minerals in the adsorbents was reported in reference [16].

Igepal 630's hydrophilic monomers are interacting with clay minerals mostly through hydrogen bonding. This will lead to the orientation of the surfactant in such way that its hydrophobic tail is directed towards the interior of the pores. The presence of surfactant tails within the pores will reduce the interactions between the water molecules and the porous skeleton during drying process allowing them to pass much easier from the inside of the green products up to the surface. This mechanism is responsible for the reduction of internal moisture stresses during the drying process.

Recommended surfactant concentration of 0.005% was added and industrial roofing tiles were produced. The industrial drying regime reported in reference [6] was applied. The bending strength after drying was measured inside the factory in accordance with its factory production plan. These results are confidential and the authors did not have the factory approval to publish it. We can only say that the bending strength results of the treated samples are higher and that the drying scarp rate was lower for 3 percent.

4. Conclusions

In order to decrease the roofing tiles failures generated during intensive drying the non-ionic surfactant Igepal CO-630 was added in the raw material during forming process. Various amounts of surfactant were used. The mechanism responsible for the reduction of internal moisture stresses during the drying process was reported. The surfactant tails will reduce the interactions between the water molecules and the porous skeleton during drying process. This will increase the internal moisture transport from the inside of the green products up to its surface. Results have confirmed that the optimal concentration of the surfactant is near or below the CM. When surfactant was applied in a recommended amount it has significantly reduced the drying induced fractures in roofing tiles during its intensive drying and consequently the industrial scarp-rate was accordingly lowered for 3 percent.

5. References

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