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Acoustic emission monitoring of fracture in dryed kaolin-clay processed with surfactant water solution

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Streszczenie

The method of fracture reduction in kaolin-clay products during intensive drying, which comprises application of surfactant water solutions for their forming, is the objective of this paper. Two oxyethylates surfactants, Rokacet R26 and Rokanol IT6, were used for the tests. Before forming the samples, the kaolin-clay in the form of dry powder was wetted with a prescribed amount of distilled water mixed with a different amount of surfactants to get a plastic mass. The samples in the form of cylinders were extruded and, after leveling the moisture distribution, subjected to convective drying in hot air. The acoustic emission (AE) method was used to monitor *on line* the development of crack formation in dried samples. It was stated that application of water solutions with the specific concentration of surfactants during material processing may significantly reduce material fractures due to intensive drying.
Keywords: Clay, Drying, Surfactant, Fracture, Acoustic emission

ZASTOSOWANIE METODY EMISJI AKUSTYCZNEJ DO MONITOROWANIE ZJAWISKA PĘKANIA PODCZAS SUSZENIA KAOLINU ZWILŻANEGO WODNYM ROZTWOREM SURFAKTANTU

Przedmiotem artykułu jest przedstawienie sposobu ograniczenia pękania wyrobów kaolinowych podczas ich intensywnego suszenia, a polegającego na zastosowaniu wodnych roztworów surfaktantów przy formowaniu tych wyrobów. W badaniach użyto dwa surfaktanty oksyetylenowe: Rokacet R26 i Rokanol IT6. Przed uformowaniem próbek proszek gliny kaolinowej został zwilżony odpowiednią ilością wody destylowanej zawierającej różne ilości surfaktantów, tak aby uzyskać stan plastyczny masy zarobowej. Po wyrównaniu rozkładu wilgoci w tej masie przez jej umieszczenie na 48 godz. w zamkniętym naczyniu, wytłaczano cylindryczne próbki, które następnie suszono konwekcyjnie w gorącym powietrzu. Metodę emisji akustycznej (AE) wykorzystano do monitorowania *on line* rozwoju pęknięć w suszonych próbkach. Stwierdzono, że zastosowanie roztworów wodnych o specyficznym stężeniu surfaktantów pozwala znacznie zredukować pękanie materiału wywołane intensywnym suszeniem.
Słowa kluczowe: surowiec ilasty, suszenie, środek powierzchniowo czynny, pękanie, emisja akustyczna

1. Introduction

Materials like kaolin-clay are used to create many ceramic products like bricks, roof tiles or sanitary products [1, 2]. To get molded plastic masses, the row clay-based materials are mixed witch appropriate amount of water. However, the capillary-porous structure of clay-like materials is prone to shrinkage and cracking during drying. This phenomenon is very unwanted and causes significant reduction of the products mechanical strength and sometimes renders their use impossible. The main reason of material cracking is the drying induced stress. The drying stresses occur when the moisture or temperature distributions become nonlinear in material, which happen mostly by intensive drying [3]. To improve moisture transport inside the dried body and make moisture distribution more uniform, the authors suggest wetting the row kaolin-clay with water containing surfactant agents. In this way the surface tension between water in pores and the pore walls is reduced, and the cracking possibility softened [4, 5, 6, 7]. According to our knowledge no earlier publications exist which describe the usage of surfactants for improving drying of clay-like materials.

Surfactants are compounds equipped in hydrophobic and hydrophilic fragments. They can be divided into three groups: anionic (a hydrophobic chain attached to an acidic group like carboxylate, sulphate or sulphonate), cationic (a hydrophobic chain attached to a group like quaternary ammonium) and nonionic (a hydrophobic chain attached to a polyalkoxylate chain). Surfactants are able to create stable colloidal aggregates called micelles, when concentration of surfactant solution exceeds the Critical Micelle Concentration (CMC). At a concentration of surfactant below CMC, they are always dissolved as monomers. There are works where surfactants with concentration over CMC are used to remove impurities or organic compounds from aqueous solutions [8, 9, 10]. Purkait *et al*. [11], for example, used surfactants in cloud point extraction to remove pigments from waste water.

The main aim of this work was to investigate the crack reduction effect in kaolin-clay material moisturized with oxyethylates surfactants during intensive convective drying. Two surfactants were used: first one was Rokacet R26 (oxyethylates ricin oil) and the second was Rokanol IT6 (oxyethylates alcohol). These surfactants added to the water moisturizing kaolin-clay are able to reduce significantly the material fracture during intensive drying. The samples after leveling the moisture distribution were subjected to convective drying in hot air at a constant temperature of 90 °C in a chamber dryer. All drying processes were monitored *on line* by acoustic emission (AE) method [12]. The acoustic emission (AE), as a non-destructive method, is very popular diagnostic technique in materials engineering. This method was applied to identify the drying period in which the cracks are developed, and to monitor the number of cracks and their size. During drying the samples were visually observed and photographed through a glass window of a chamber of the dryer. These photographs were used to assess the material quality after drying.

2. Material and method

Fig. 1 presents the scheme of the experimental equipment used for the tests. The drying processes were carried on in a laboratory chamber dryer Zalmed SML42/250/M. The sample mass, the temperature and the air humidity were measured, and the sample appearance photographed every half minute during drying. The scale pan with the holder was suspended to an electronic balance (Radwag WPS 2100/C), which registered mass changes with 0.01 g accuracy. The temperature and the relative humidity of air in the dryer chamber were measured with a Pt 100 temperature probe and a humidity sensor DO 9861T Delta OHM (Italy) located close to the drying sample. The digital detector measured the air temperature with 0.1 °C accuracy, and the air humidity was measured with 0.01% accuracy. All these variables were collected by a computer provided with a software for data acquisition. The AE sensor was attached to the bottom surface of the sample. The registered AE signals were transmitted into electric ones and strengthened by the amplifier.

The tested samples in the form of cylinders were made of KOC kaolin-clay provided by the Surmin Kaolin Company S.A. Nowogrodziec, Poland. The preparation of kaolin samples consisted of the following steps. In the first step dry kaolin has been grounded to powder and wetted with pure water or water solutions containing 0.1%, 0.01%, 0.001%, 0.0001% and 0.00001% of oxyethylate surfactans Rokacet R26 or the Rokanol IT6. These ingredients were mixed by hand to get a greasy paste. The kaolin-clay paste was stored in a tight container at room temperature for 24 hours to homogenize the material. The greasy paste was of initial moisture content approximately 40% (dry basis). Next, the soft kaolin-clay mass was used to mold cylindrical samples (d = 44 mm, h = 50 mm).

The acoustic emission (AE) method was applied to monitor *on line* the development of crack formation in the samples during drying. The acoustic emission arises in the stressed materials when the elastic energy accumulated inside the dried material is released. These energy is transported throughout the material and measured by very sensitive AE sensor. The AE energy sensor was installed under the sample and transmitted the AE descriptors. The following descriptors were measured in the tests: the AE energy per a time period (e.g. 30 s), the total AE energy, the number of AE signals per a time period, and the total number of AE signals. The number of signals reveals the number of cracks and the total AE energy shows the size of cracks, and thus give an information about the quality of dried samples. The manufactured samples contained various amounts of surfactants to analyze their effect on sample crack formation. The advantage of the AE method is that it enables monitoring *on line* the development of crack formations.

3. Result and discussion

All drying experiments were carried out at the constant air temperature of 90 °C, which ensured a high drying rate. Each test was repeated at least three times. The relative air humidity in the dryer chamber at 90 °C amounted about 2.5%, however, it changed slightly in several periods of drying.

Fig. 2 presents drying curves for different surfactant concentrations. As it is seen, the drying curves for kaolin-clay processed with water solution containing surfactant concentrations ranging from 0% to 1% do not differ to much from each other. So, such a small concentration of surfactant in water solution has an insignificant influence on the drying rate. However, we want to show that products made of kaolin-clay material and containing surfactants in the range from 0% to 1% have the tendency to considerable reduction of cracking during intensive drying at high air temperatures.

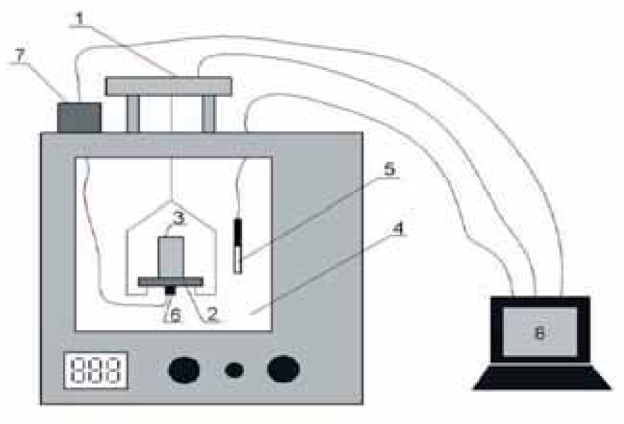

Fig. 1. Experimental set-up: 1 – balance, 2 – scale pan, 3 – sample, 4 – dryer chamber, 5 – temperature sensor, 6 – AE sensor, 7 – amplifier of AE signal, 8 – computer.

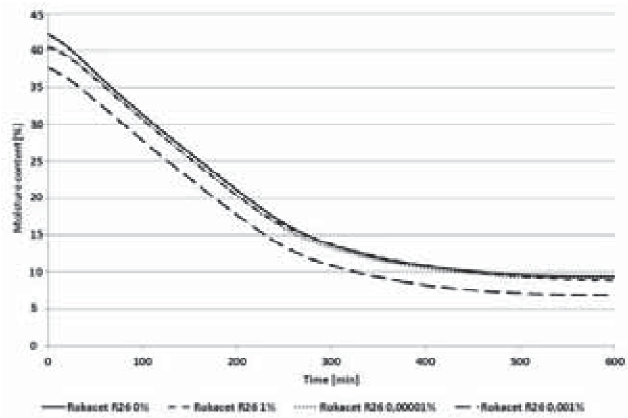

Fig. 2. Drying curves of clay samples at the air temperature of 90°C for different surfactant concentrations.

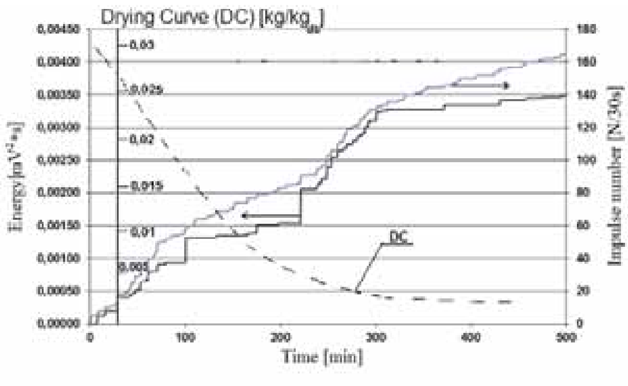

Fig. 3. Total AE energy and total number of AE impulses for kaolin--clay samples wetted by pure water.

Fig. 3 presents the descriptors of total AE energy and total number of AE impulses emitted during drying of kaolin-clay saturated with pure water.

Each rapid increase of the total AE energy visible on the AE curve denotes a crack which occurs in the kaolin-clay sample at a given moment. As seen in Fig. 3, the biggest cracks were formed at the end of the constant drying rate period (CDRP) and at the beginning of the falling drying rate period (FDRP) [13]. At this stage the sample surface become dry while the core of the sample is still wet, and material cracks usually occur at this stage (see Fig. 9a).

Figs. 4 and 5 present the total AE energy and the total number of AE impulses emitted during drying of kaolin-clay saturated with water surfactant solutions containing of 1% Rokanol R26 and 1% Rokacet IT6, respectively.

As it can be seen from Figs. 4 and 5, the addition of 1% surfactant to water solutions used for kaolin-clay saturation

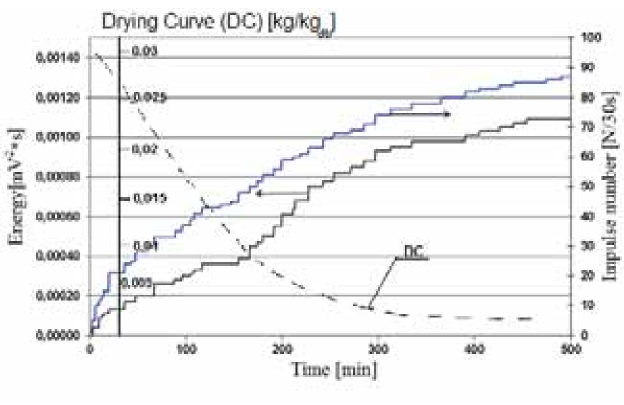

Fig. 4. Total AE energy and total number of AE impulses for kaolin--clay samples wetted with the solution containing 1% of Rokanol R26.

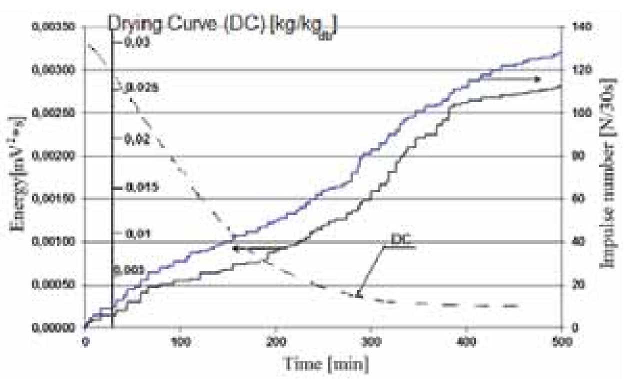

Fig. 5. Total AE energy and total number of AE impulses for kaolin--clay samples wetted with the solution containing 1% of Rokacet IT6.

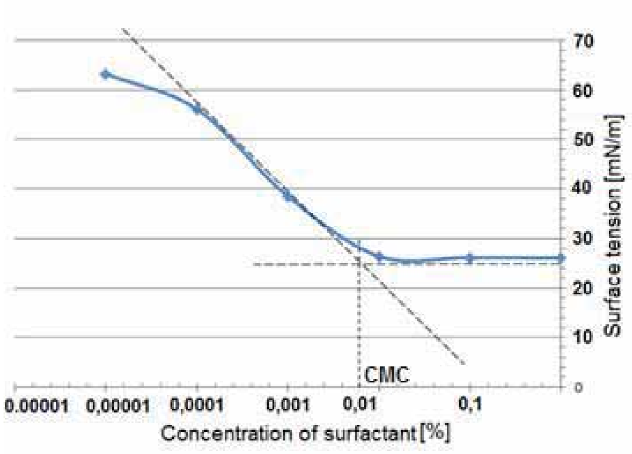

Fig. 6. Determination of the CMC for Rokacet IT6.

reduced the total AE energy and the total number of AE impulses in comparison to the kaolin-clay saturated with pure water (0% surfactant). However, the reduction of AE energy and AE impulses is not significant, and the quality of the samples obtained after drying is still not satisfactory.

The structure of surfactant solutions depends on the surfactant concentration. The limit value of the surfactant concentration, at which the molecules appear in the mono-molecular form is called *the critical micelle concentration* (CMC). The surfactant solution with concentration below the CMC is always dissolved as monomers, the excess of surfactants over CMC value may appear in the surfactant solution as associates called micelles. The CMC value was determined experimentally in the present studies with a specialized equipment.

Fig. 6 presents the graphical method of determination of the CMC for Rokacet IT6 surfactant

The value of CMC for a given surfactant solution was determined by drawing the corresponding tangent straight lines to the plot of surface tension versus concentration, as shown in Fig. 6. The point of intersection shows the CMC value.

The graphs shown in Figs. 7 and 8 present the AE energy and the total number of AE impulses for kaolin-clay samples with 0.01% addition of Rokanol R26 and Rokacet IT6, respectively.

As the graphs in Figs. 7 and 8 show, the total AE energy and the total number of AE signal are much smaller than that for the samples in previous tests. Fig. 9 shows that the quality

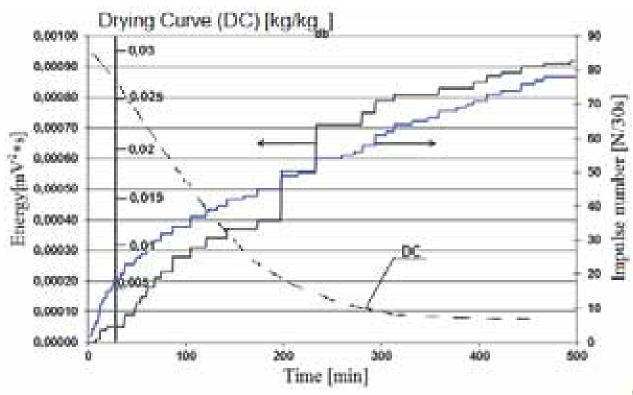

Fig. 7. Total AE energy and total number of AE impulses for kaolin--clay samples wetted with the solution containing 0.01% of Rokanol R26.

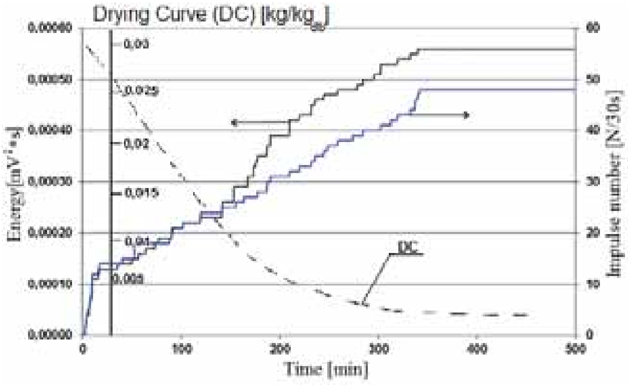

Fig. 8. Total AE energy and total number of AE impulses for kaolin--clay samples wetted with the solution containing 0.01% of Rokacet IT6.

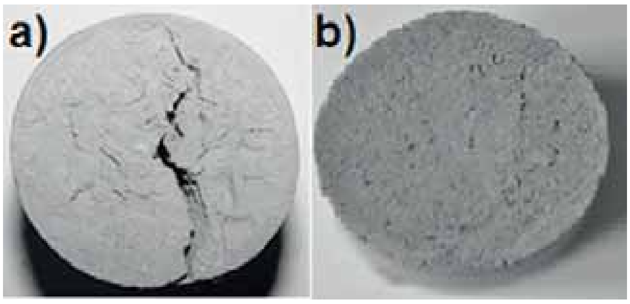

Fig. 9. Bottom surface of kaolin-clay samples after drying: a) without surfactant, b) with 0.01% of Rokacet IT6.

of sample with 0.01% Rokacet IT6 is much better than that with 0% (pure water) concentration.

Fig. 9 presents the visualization of kaolin-clay samples after drying in the case when the material before sample forming was wetted with pure water (Fig. 9a) and water solution with addition of 0.01% surfactant Rokacet IT6 (Fig. 9b). These photographs show evidently the reduction of sample cracks due to the application of the prescribed amount of surfactant to processing of kaolin-clay.

4. Conclusions

The results presented in this paper allow us to state that drying of product made of kaolin-clay materials containing a prescribed amount of surfactant addition considerable reduces the tendency of this material to cracking during intensive drying. The best quality of the dried samples was achieved for the kaolin-clay saturated with water containing rather low concentration of surfactants, that is, a concentration close to CMC.

The results of research with surfactant concentration close to CMC show that this parameter has very essential influence on moisture transport inside capillary-porous materials during drying. This phenomenon is related with the surface tension between moisture and the pore walls. The addition of surfactant to water used for wetting the kaolin-clay before forming products significantly decreases the surface tension of moisture, so it can be much easily evaporated from capillary-porous material during the drying process. The reduced surface tension between moisture and the skeleton of porous materials reduces the stresses inside the material, which are responsible for cracking of clay samples by drying. In this way it is possible to obtain much better quality of dried products made of clay at high drying rates.

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