Contents lists available at SciVerse ScienceDirect

Chemical Engineering Research and Design

journal homepage: www.elsevier.com/locate/cherd

Reduction of fractures in dried clay-like materials due to specific surfactants

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ABSTRACT

The possibility of fracture reduction as a result of application of specific surfactants during intensive drying of clay-like materials was examined in this paper. The dodecyl sulfate sodium salt (SDS) and the fluoric (FC 4430) surfactants were used for the tests. Different amounts of these surfactants were mixed with distilled water and used for wetting a dry clay material before forming clay samples. The clay samples in the form of cylinders 44 mm in diameter and 50 mm high were extruded and after leveling the moisture distribution subjected to convectively drying in hot air at 120 °C. 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 surfactants in a prescribed amount may significantly reduce the drying induced fractures in clay during its intensive drying and thus to obtain good quality products by high drying rates.

Keywords: Clay; Intensive drying; Surfactants; Crack reduction; Acoustic emission

1. Introduction

Clay-like row materials are widely used in buildings and ceramics industries for manufacturing of various products (bricks, roof and wall tiles) or for production of sanitary and tableware (Pampuch, 1988; Raabe and Bobryk, 1997). These raw clay-based materials before molding into required shape are mixed with an appropriate amount of water to get a plastic mass. The products after molding and leveling the moisture distribution are subjected to drying and next to firing. Unfortunately, the capillary-porous structure of clay-like materials is prone to shrinkage and cracking during drying, which is very disadvantageous as it weakens the mechanical strength of the products and significantly reduces the possibility of their use (Banaszak, 2007; Banaszak and Kowalski, 2002, 2005; Kowalski, 2003).

The reasons for material cracking are the stresses induced in the material during drying. The stresses arise if the moisture and/or temperature distributions in dried material become nonlinear (Augier et al., 2002; Kowalski et al., 1992, 2000; Kowalski and Rybicki, 2000; Scherer, 1986). Such an inconvenient moisture distribution is created, for example, by intense moisture removal from the surface and by hindered moisture transport inside of the material.

In order to improve moisture transport inside the dried body, and thus to assure more uniform distribution of moisture in the material and thus avoid its cracking, the authors propose wetting the raw clay with water containing surface active agents (surfactants). These agents have the ability to stimulate the surface tension between water and the pore walls and thus to improve moisture transport inside the material (Cottrell, 1970; Wert and Thomson, 1974).

To our knowledge no earlier publications exist which describe using surfactants for improving drying of clay-like materials. One can find, however, works where surfactants are used to improve manufacturing xerogels. Matos et al. (2006), for example, obtained a xerogel with high mesoporosity after synthesis with application of three different surfactants (non-ionic, cationic and anionic) with concentrations varying between 0.1% and 15% (wt./wt.). The addition of surfactants affected the porous structures of the carbon xerogels. Mosquera et al. (2008) proposed an innovative strategy to obtain crack-free gels by using a surfactant as a template for the silica pores. A neutral surfactant – n-octylamine – which

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Received 9 November 2011; Received in revised form 28 August 2012; Accepted 30 August 2012
0263-8762/$ – see front matter
http://dx.doi.org/10.1016/j.cherd.2012.08.020

weakly interacts by hydrogen bonding with the silica precursor was used. This allowed it to be removed by simple drying in ambient air. The syntheses promote the formation of a crack-free uniform mesoporous silica gel.

Surfactants are compounds composed of both lipophilic and hydrophilic fragments The number of each varies, but most commonly there is one of each kind. The more common surfactants are 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), o nonionic (a hydrophobic chain attached to a polyalkoxylate chain). Surfactants tend to be scarcely soluble in water as free molecules or ions, but they are able to form stable colloidal aggregates called micelles. The presence of ionic surfactant micelles depends on surfactant concentration being above the *Critical Micelle Concentration* (CMC). A concentration equal to, or below, CMC is always dissolved as monomers, the excess is present as micelles.

The surfactants with concentration over CMC are often used to remove impurities or organic compounds from aqueous solutions by means of two-step adsorption on surfactant micelles. The first step flocculates the micelles and creates sites with inverted polarity on the aggregate. The second step binds pollutant molecules (Creagh et al., 1994; Nystrom and Manttari, 1994; Paulenowa et al., 1998; Scamehorn et al., 1986, 1994). Purkait et al. (2004), for example, used surfactants in cloud point extraction to remove pigments from waste water.

The results of research with surfactant below CMC show that this parameter has very essential influence on the surface tension between moisture and the pore walls and thus on moisture transport inside capillary-porous materials during drying (Cottrell, 1970; Chen et al., 1998). Sacchi et al. (2001) described the results of extraction techniques available to obtain water and solutes from the argillaceous rocks. The paper focuses on the mechanisms involved in the extraction processes, the consequences on the isotropic and chemical composition of the extracted pore water.

The main goal of this work was to investigate the effect of the dodecyl sulfate sodium salt (SDS) and the fluoric (FC 4430) surfactants on the reduction of the drying induced stresses in dried materials. These surfactants applied in a prescribed amount are able to decrease the surface tension between moisture and the skeleton, and thus to reduce the material fracture during intensive drying. The acoustic emission (AE) method is applied to monitor *on line* the development of crack formation during drying.

The experiments were carried out on cylindrical samples which were molded of clay wetted with water of different surfactant concentration. The samples after leveling the moisture distribution were subjected to convective drying in hot air at temperature 120 °C in a dryer chamber. Apart from AE monitoring the samples were visually observed and photographed during drying through the glass window in the chamber wall. The samples after drying were subjected to compression tests to show the influence of different surfactant concentration on the material strength.

2. Materials and methods

2.1. Experimental

Fig. 1 presents the scheme of the experimental equipment used for the tests. The drying processes were carried on in the laboratory chamber dryer Zalmed SML42/250/M (4).

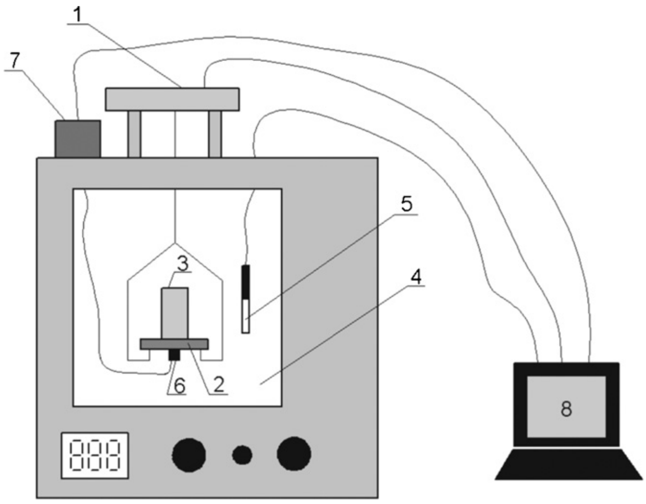

Fig. 1 – Experimental set-up: 1 – balance, 2 – scale pan, 3 – sample, 4 – dryer chamber, 5 – temperature sensor, 6 – AE sensor, 7 – amplifier of AE signal, 8 – computer.

Drying variables such as the air temperature and the relative humidity, and the reduction of sample mass were measured every half minute during drying. All these variables were transmitted to the computer (8) provided with the software for data acquisition. The air in the drying chamber was stationary. The temperature and the relative humidity of the air in the dryer chamber were measured with a Pt 100 temperature probe and humidity sensor DO 9861T Delta OHM (Italy) (5), which was located close to the drying sample (3). The digital indicator showed the air temperature with accuracy 0.1 °C, while the air humidity was measured with accuracy of 0.01%.

The acoustic emission AE sensor (6) was attached to the bottom surface of the sample, and the registered AE signals were transmitted into electric ones and strengthened by the amplifier (7) (Skaar et al., 1980; Kowalski and Pawłowski, 2011). The holder with the scale pan (2) was suspended to the electronic balance (Radwag WPS 2100/C) (1), which registered mass changes with accuracy 0.01 g.

2.2. Materials

The tested samples in the form of cylinders were made of clay delivered from clay pit in Kozłowice (Poland). Two kinds of surfactants were used in these studies. First one was the anionic sodium dodecyl sulfate (SDS). This organic compound usually occurs with a mixture of sodium alkyl sulfates, and its chemical formula is $C_{12}H_{25}SO_4Na$. The second one was the fluoric surfactant FC-4432 from 3M Deutschland GmbH (Neuss, Germany). It is also ionic surfactant containing 87–93% (mass.) of fluoroaliphatic polymeric esters and 0.5% mass. of 2-Methyl (nonafluorobutyl-sulphonyl-amino-ethyl) acrylate.

The preparation of clay samples consisted of the following steps. In the first step dry clay has been grounded to powder and sieved to eliminate particles exceeding 0.4 mm dimension. Such powder of amount 250 g was mixed with 120 ml of pure water or water solution containing 0.001%, 0.01%, 0.1%, 1% of surfactant. These ingredients were mixed by hand to get a thick paste.