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# Study of the flocculation of anaerobically digested residue and filtration properties of bentonite based mineral conditioners

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ABSTRACT

The turbidimetric and capillary suction time (CST) methods were used for the assessment of some properties (e.g., filtration process, water uptake etc.) of diatomite–bentonite based conditioners for dewatering of effluents from digestate of a biogas plant. These physical conditioners were prepared based on natural porous highly dispersible systems such as bentonite and diatomaceous earth. A characterization of the materials was performed by scanning electron microscopy (SEM) and fluorescence of X-rays spectrometry (XRF). A cationic polyacrylamide polymer (ZETAG® 9014) was added prior to the dewatering process of the anaerobic digestion residue and an optimal dose of the mineral conditioner and the polymer was determined. It was suggested the formula, by means of which the amount of separated water was identified as water retention capacity (WRC). A favorable filterability region was determined with WRC between 1.5 and 4.4 (high level of WRC) and 1.5 and 2.9 (low level of WRC) in terms of the percentage of polymer in the dry cake.



## Introduction

Anaerobic digestion produces a methane-rich biogas, as well as a digested effluent also known as anaerobic digestion residue (ADR) or digestate, which contains significant amounts of various nutrients, including nitrogen, potassium and phosphorus among other plant nutrients. Currently, one of the most feasible options for the correct management of digestate is its direct application to agricultural fields. The relative cost of transportation of the ADR can be high due to the low concentration of both nutrients and dry matter. Conversely, stockpiling of digestate may occur as a result, meaning that nutrients contained therein may pose potential environmental risks to the surrounding water bodies if improperly managed. Consequently, a more effective separation from liquid to solid phase in the digestate would favor an increased sale ability of these products and their derivatives, with less environmental associated risks. Moreover, significant amounts of water can be recycled and returned to the inlet stream of the bio-gas process in order to process new batches of bio-degradable waste. On the other hand, wastewater treatment processes (WWTPs) produce large amounts of sludge commonly containing over 90% of water with similar transportation and handling problems as for the ADR.

Colloidal systems which are present in undewatered sludge and the ADR form a stable suspension in water and enhances difficulties (commonly encountered) in the mechanical dewatering process such as vacuum and pressurized filtration. The addition of chemical conditioners such as coagulants and flocculants is frequently necessary to help the sludge or ADR particles to agglomerate into larger aggregates which precede the solid–water separation. However, due to the highly compressible nature of the sludge solids, the sludge dewatering rate is often hindered by the blinding of the filtration media and the filter cake [1,11–13]. Novak et al. [14] suggested that the clogging or blinding of pores in the filter cake is primarily responsible for the deterioration of the filtration rather than the blinding of the filtering media. Furthermore, when high molecular weight organic polymers are used for flocculation, sludge dewatering rate can be increased to a

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certain extent by decreasing the sludge specific resistance. In this stage and under a certain pressure, no further water can be removed [15]. Such long compression times equate to extremely slow rates of dewatering.

There are two widely accepted flocculation mechanisms: particle–particle bridging and surface charge neutralization [16–18]. With the particle bridging mechanism, the polyelectrolyte chains adsorb on the solid surface forming loops, trains and tail configurations. When two particles come together, the loops and tails of one particle attach themselves to bare patches on the approaching particle to form bridges [19]. In general, the effectiveness of bridging flocculation is directly related to the molecular weight (MW) or chain length of the polyelectrolyte [20].

Physical conditioners, commonly known as skeleton builders or filter aids improve the mechanical strength and permeability of the sludge cake during compression. These materials can form a permeable and more rigid lattice structure which can remain porous under high pressure during mechanical dewatering [2]. A wide range of carbon-based materials have been used as physical conditioners, including char [3], coal fines [3,4] and bio-waste such as wood chips and wheat dregs [5] and bagasse [6]. Minerals including fly ash [2,6,7], cement kiln dust [6] and gypsum [8,9] have also been used for such approach. A physical conditioner can be used individually to enhance sludge dewatering such as in the investigation carried out by Jing et al. [10].

The requirements of the physical mineral conditioners for filtration of sludge are that they should be inert and should avoid filter blinding [1]. However and as investigated from [2], the surface of these systems could have a charge. Therefore, it means that during the dewatering by means of the addition of mineral conditioners, there is not a “pure” filtration achieved, due to the interactive surfaces of such systems involved in the flocculation process. The degree of such interaction is dependent on:

- Surface area of filter-aids powder;
- Surface charges;
- Porosity (pore volume, mean diameter of pore);
- Types of functional groups on the surface.

The analysis of literature points out that the selection of such materials has a random character and there is not logical strategy in the selection of these agents. On one hand, relatively inert materials are used (e.g., coal) as done previously by [3,4], and on the other hand, there are physical conditioners which have surfaces with very active groups (cement, lime, char etc.) [6,9]. Thirdly, there are materials inert but without any porosity etc.

The main material of the mineral conditioner used in this study is a bentonite (from the Republic of Armenia). Typically, the measured specific surface area by use of nitrogen gas gives results in the range of 20–100 $m^2/g$ of clay regardless of the preparation technique and montmorillonite content. Comparison with the total theoretical surface area of montmorillonite of around 750 $m^2/g$ indicates that montmorillonite layers in close contact dominate the dry material, and that the interlayers are inaccessible for the nitrogen gas [22]. The second important component in the mineral conditioner is the silica containing material (diatomite earth). The use of the silica-containing component provides the structure for making porosity and rigidity of the mineral conditioner. These properties provide good filterability of the aforementioned materials in order to avoid blinding of the cake during the filtration as investigated by Novak et al. [14]. In addition, the bentonite and diatomite system contains Ca which influences the mechanism of clogging [23] and the flocculation of colloidal suspensions [24,25].

Commonly, the physical conditioner addition is followed by coagulation or flocculation with a chemical conditioner. When physical conditioners are used in conjunction with chemical conditioners, sludge dewaterability can achieve its optimum. Without chemical conditioners, which are used to manage sludge colloids, physical conditioners alone do not usually function as filter aids to the same extent or at all.

It has also been found that the application of physical conditioners can reduce the use of chemical conditioners and, thus, the cost of the treatment process, while still achieving the same level of dewatering performance [11].

The goal of this study was to optimize the dose of the polyelectrolyte (ZETAG® 9014) in the dewatering of digestate by vacuum filtration. For that purpose, natural high dispersible porosity materials as mineral conditioners were prepared as a bentonite and diatomite combined system. The latter was added to the digestate prior to the addition of ZETAG® 9014 for improving the dewatering process [10,11]. Moreover, a favorable filterability region in terms of the concentration of ZETAG® 9014 in the dry cake and WRC in a high and low level, was determined in order to avoid the filter cloth clogging [12,13].

## Materials and methods

### *ADR substrate*

The ADR substrate that was used in all the experiments was sampled from the rejected stream after the decanter from the bio-gas plant of Lindum AS (Norway). The physicochemical parameters of the ADR substrate are shown in Table 1 and were analyzed in accordance to the standard methods for the examination of water and wastewater [21].

### *Mineral conditioner DB-12Ca*

The code of the mineral conditioner “12” refers to the ratio of diatomite to bentonite mixed. This filter material was treated with a slurry of CaO in order to increase its Ca content up to 5% and the mixture was washed several times with deionized water for removal of the excess of CaO. The material was afterwards dried at 105 °C in an air oven until constant weight.

### *Cationic polymer (ZETAG® 9014)*

A suspension of the cationic polyacrylamide polymer (ZETAG® 9014) produced by BAFS SE (Germany), was used as the chemical conditioner. The dried matter content of the polymer was 50% (w/v) and was determined by a gravimetric method by drying it at 105 °C until constant weight in an air oven. This step was carried out to express the results in the next section as $mg_{polymer}/g\ TS_{ADR}$. The polymer was added to the ADR as a suspension in the dewatering experiments.

**Table 1**
Physicochemical parameters of the ADR.

| Parameter | Value or concentration |
|---|---|
| pH[a] | 8.3 ± 0.2 |
| TS | 0.8 ± 0.1% |
| SS | 0.4 ± 0.07% |
| VS | 99.90 ± 0.01% |
| Conductivity | 7.65 ± 0.02 mS/cm |
| Turbidity | 4700 ± 50 FTU |
| $P_{total}$ | 83 ± 5 mg/L |
| orto-P | 2.5 ± 0.2 mg/L |
| K | 350 ± 20 mg/L |
| $NH_4$ | 50 ± 3 mg/L |
| COD | 2500 ± 70 mg/L |

[a] Measured at 25 °C.