Effects of sludge properties in a combined process of mesophilic anaerobic-aerobic digestion

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Abstract. The combined medium-temperature anaerobic digestion and medium-temperature aerobic digestion (MAND/MAD) is a strategy that is suitable for the disposal of conventional activated sludge. The MAND/MAD sludge stabilization process was performed at three retention times (17, 24, and 38 days) of anaerobic digestion (AND-time) with further duration up to 22 days of the MAD process. The retention time of the MAD process had an important role in sludge stabilization. Its extension enhanced sludge stability in the aerobic digestion stage. During a MAD process, values of volatile/total solids ratio (VS/TS) presented the lowering course for the three investigated retention AND-times. The relatively long retention time of MAND leads to a higher level of soluble organic compounds (SCOD) and extracellular polymers (EPS) in the supernatant. Due to the shorter capillary uptake time at the end of digestion in the MAND/MAD system, the sludge with an AND-time of 38 days was extremely susceptible to dewatering compared to the sludge with an AND-time of 17 and 24 days.

1. Introduction
The traditional activated sludge process is the most commonly used biological treatment process for wastewater. [1]. The chemical and biological composition of the sewage sludge is dependent on the effluent composition. It is normally rich in organic matter and plant nutrients such as nitrogen, phosphorus and calcium, which improves the physical, chemical and biological properties of the soil, such as portability, aggregate stability, soil fertility, water flow and retaining power [2]. Nevertheless, the conventional activated sludge process generates a significant amount of sludge, which is hard to be stabilized and dehydrated. Biological treatment processes, involving the aerobic and anaerobic digestion stages, are typically utilized to stabilize sludge prior to its final disposition. Out of these processes, the combining of mesophilic anaerobic digestion and mesophilic aerobic digestion is accepted as a proper method due to its low cost and operational simplicity.[3].

Recently, the combined medium-temperature anaerobic digestion and medium-temperature aerobic digestion have been further developed to facilitate the applications and economize the aeration power consumption.[4]. One of the factors that affect the combination process is the retention time of anaerobic digestion. The previous investigators indicated that changing the residence time of anaerobic digestion can influence the efficiency of the MAND/MAD sludge stability process. Thus, it is significant to investigate the retention time of anaerobic digestion and to figure out the appropriate
operation mode to avoid disturbing the digesters by extending or shortening the time of anaerobic digestion.

The main object of the sludge stability is to deteriorate the organic matter included in the sludge and to inactivate the microorganisms and pathogens. [5]. Therefore, the conception of stabilization is often also associated with putrefaction and scent. In this regard, the ratio of volatiles/total solids can be utilized as a stability index. In principle, a ratio under 0.40 suggests that stability has been reached [6]. Previous researches have observed that many factors, such as residence time of anaerobic digestion, influence the stabilization of sludge. [7].

In addition, the efficiency of sludge dewatering is the critical to sludge reduction [8]. Nonetheless, some scholars have noted that sludge mixtures are complex in structure and contain hydrophilic substances such as microbial cells and extracellular polymers, making them difficult to dewater [9]. Further research on the sludge dewatering performance of medium temperature anaerobic digestion combined with medium temperature aerobic digestion is therefore necessary.

This work is intended to determine the suitable retention time for anaerobic digestion to attain stability in the MAND/MAD process. Batch experiments were conducted for three AND-times of 17, 24 and 38 days based on the results of earlier optimal operating parameters and the basic theory of anaerobic and aerobic digestion. The work presented also investigated the volatile solids removal and dewatering characteristics of conventional activated sludge and matched the data to the sludge characteristics before and after digestion treatment. The objectives of this work are to assess the capabilities of MAND/MAD and to recommend its large-scale implementation.

2. Materials and methods

2.1. Sludge sample

The sludge for the experiments was collected from the Jiujiang University wastewater treatment facility, where sequencing batch reactor (SBR) was used to treat domestic wastewater. The features are listed in Table 1. The total solid (TS), volatile solids (VS), NH4+-N, and pH value were measured based on standard ways. The capillary water absorption time was determined with a Triton 304 CST tester.

| Parameters | Results  |
|------------|---------|
| pH         | 7.7     |
| NH4+-N     | 54.7 mg/L |
| SCOD       | 822.6 mg/L |
| VS/TS      | 0.83    |
| CST        | 7289 s  |

2.2. Experimental settings

Five hundred ml of sludge solution was placed in an anaerobic reactor in a water bath at a constant temperature of 35 ± 2 °C for 17 days, 24 days, and 38 days, respectively. After anaerobic digestion, the sludge liquid was transferred to an anaerobic reactor with a water bath aeration at a thermostat of 35±2 °C. Samples were taken to determine TS, VS, pH, NH4+-N, CST and EPS after the prescribed aerobic aeration times (1, 3, 5, 8, 15 and 22 days).

2.3. EPS extraction and analysis

EPS was extracted by means of heat. The extraction method was as follows: 4 ml of the sludge was taken in a centrifuge tube, centrifuged at 4000 rpm for 5 minutes, and the supernatant was taken; then 0.05% NaCl solution was transferred to the centrifuge tube, placed in a water bath at 50°C for 5 minutes, and then centrifuged at 4000 rpm for 10 minutes, and the obtained supernatant was LB-EPS.

Prior to chemical determination, the sample supernatant was purified through a 0.45 μm filter. polysaccharide content in EPS was determined by anthranilic acid method based on glucose as the
standard; protein and humic content were measured by a modified Lowry method based on egg protein and humic acid as the standard [10].

3. Results and discussion

3.1. Changes of pH and NH$_4^+$-N concentration
Changes of pH and NH$_4^+$-N as a function of anaerobic digestion time (AD-time) in raw sludge and sludges digested aerobically for 17, 24, and 38 days are presented in Figure 1a and Figure 1b, respectively. In the batch operation experiments performed, the pH of the digester was not adjusted. Raw sludge pH dropped during the first 3 days of anaerobic digestion due to hydrolysis-acidification; then it gradually rose to more than 8.4, due to the release of proteins by cell lysis and the generation of large amounts of NH$_4^+$-N by means of deamination (Figure 1a). The values of pH observed indicated that the sludge acidity was within the accepted range from the agricultural use [11].

The pH changes during aerobic digestion of different AND-times (17, 24, and 38 days; Figure 1b) sludges increased continuously in the first 5 days, reaching the maxima of 8.5, 8.6, and 9.5, respectively. Previous researches indicated that pH is associated with the hydrolytic emission of reduced nitrogen (NH$_3$) from the sludge solution and its subsequent acid-base equilibrium [12]. Nevertheless, the pH value of the process of the combined mesophilic anaerobic digestion and mesophilic aerobic digestion system is relatively complicated and greatly influenced by numerous variables [13]. The pasteurization efficacy of the combined system is not only due to the temperature, but also because of the raised pH value during the handling procedure.

![Figure 1a](image1.png)

**Figure 1a.** Changes of raw sludge pH with the duration of the MAND process.

![Figure 1b](image2.png)

**Figure 1b.** Changes of 17, 24, and 38 days AND-time sludges pH with a duration of the MAD process.

The changes of NH$_4^+$-N concentration in the supernatant from anaerobically digested raw sludge and aerobically digested sludges of different AND-time are shown in Figures. 2a and 2b, respectively. During MAND of raw sludge (Figure 2a) NH$_4^+$-N concentration increased abruptly at the beginning of the process reaching Plato of 1800 mg/L after 30 days of AND time. The opposite trend was found for sludges preliminary MAND processed for 17, 24, and 38 days AND-time. In these sludges the concentration of NH$_4^+$-N decreased sharply to less than 500 mg/L, indicating NH$_4^+$-N loss during mesophilic aerobic digestion. It is in agreement with data of earlier researchers who pointed out that denitrification was inhibited during mesophilic anaerobic digestion and proceeded during mesophilic aerobic digestion with loss of nitrogen as gaseous ammonia [14]. Aeration reduced the content of NH$_4^+$-N in the supernatant due to the removal of the gaseous NH$_3$ according to equilibrium: $\text{NH}_3 + \text{H}_2\text{O} \rightleftharpoons \text{NH}_4^+ + \text{OH}^-$. This leads to an improvement of the pH value of the sludge supernatant in the combined MAND/MAD system [15].
3.2. Changes of SCOD

The changes of SCOD concentration in the raw sludge supernatant during the MAND process are illustrated in Figure 3a, while in the supernatants from aerobically digested sludges with 17, 24, and 38 days AND-time during the MAD process is shown in Figure 3b. During the raw sludge MAND process, SCOD concentration sharply increased to the highest values of 8000 mg/L at 10 days of anaerobic digestion. Based on prior research, a significant amount of organic intercellular material, consisting of a few thermostable enzymes, was unleashed into the supernatant as a result of endogenous decay and enhanced cell wall lysis, leading to a dramatic increase in SCOD concentrations [16]. With further AND-time the SCOD began to decrease, and gradually stabilized at a level around 3500 mg/L during the later stages of anaerobic digestion. The 17, 24, and 38 days AND-time sludges aerobic digestion resulted in a further rapid SCOD concentration decline in the post digestion supernatant during the MAD process (Figure 3b). This was attributed to the rapid proliferation of a large number of aerobic bacteria that consumed the organic matter in the supernatant, leading to a decrease in organic matter, as evidenced by the decrease in SCOD of the digested product. Our findings are in agreement with previously reported assessments of nutrient removing from supernatants resulting from anaerobic digestion of the organic fraction of municipal solid waste using biological means. The report's authors indicate that this type of effluent is distinguished by a high nutrient content, as organic and particulate nitrogen and phosphorus are hydrolyzed during anaerobic digestion, requiring adequate post-treatment physicochemical and biological processes [17].
3.3. Changes in EPS and CTS
EPS plays an essential role in the sludge dewatering system and controls the sludge properties such as surface charge, porosity, bound water content and flocculation strength [18]. The sludge has a high degree of dewatering due to the presence of compact flocs without single cells and solubilized EPS. The emission of an excess of EPS has a detrimental impact on sludge dewatering. The proteinaceous polymer in EPS may have contributed to the deterioration of dehydration after microwave processing. It was also found that the composition of EPS has a higher impact on sludge susceptibility to dewatering than their quantity [19]. The variation of normalized ESP composition from the raw anaerobically digested sludge as a function of AND-time is shown in Figures 4a, 4b, and 4c. During this process, the found concentration of proteins in the sludge scattered within a range from 365.5 - mg/L to nearly 800 mg/L, and the polysaccharide decreased steadily from 434.8 mg/L to 40.8 mg/L. Aerobic digestion of the 17, 24, and 38 days AND-time sludges for 22 days resulted in decreased protein content from 601.7, 445.5, and 777.1 mg/L to 73.0, 94.1, and 40.7 mg/L, as well as the polysaccharide content from 76.6, 49.2, and 48.0 mg/L to 5.0, 5.2 and 4.0 mg/L, respectively. This is due to the continuous hydrolytic reduction of organic matter in the sludge upon aerobic digestion.

![Figure 4a](image1.png)
**Figure 4a.** Changes of raw sludge LB-EPS concentration with a duration of AND-time.

![Figure 4b](image2.png)
**Figure 4b.** Changes of 17, 24, and 38 days AND-time sludges protein LB-EPS concentration with the duration of AD-time.

![Figure 4c](image3.png)
**Figure 4c.** Changes of 17, 24, and 38 days AND-time sludges polysaccharide LB-EPS concentration with a duration of AD-time.

Sludge dehydration is the critical to sludge dewatering [20]. Based on prior researches, sludge is hard to dewater as it has a complex configuration and contains hydrophilic substances including microbial cells and extracellular polymers. Normalized capillary wicking time offers a facile, rapid and inexpensive method to determine the susceptibility of sludge to dewatering. The changes of normalized CST time of the raw anaerobically digested sludge as a function of AND-time is shown in Figure 5a and 5b.
The CSTs of aerobically digested sludges of 17, 24, and 38 days AND-time gradually decreased with prolonged digestion time for 22 days to values low enough that all sludges become easy to dewater after digestion. Compared sludges of AND-time 17 and 24 days with the sludge of AND-time 38 days, the latter became much easier to dewater due to its lowest CST value at the end of digestion (around 500 s) which decreased 8 times in a course of aerobic digestion. Nevertheless, the CST of the 17-day AND-time sludge was comparatively elevated during the whole digestion process and remained high at the end of the digestion process (1200 s). It has been noted that sludge is complicated to dewater due to its composition and structure, and also due to the presence of hydrophilic substances such as microbial cells and extracellular polymers [21]. Also, a correlation was found between normalized CST and EPS parameters. In our work, sludge of 38 days AND-time showed the best dewaterability and also had the lowest EPSs concentration in the supernatant generated in the MAND/MAD system.

Figure 5a. Changes of raw sludge CST time with duration of AND-time.

Figure 5b. Changes of 17, 24, and 38 days AND-time sludges CST time with duration of AD-time.

3.4. Changes of VS/TS ratio

The main objective of sludge stabilization is to decompose the organic matter in the sludge and to inactivate microorganisms and pathogens. The idea of stability for this reason is often also associated with decayability and odor. the VS/TS ratio can be used as an indicator of stability. The variation of VS/TS ratio for anaerobically digested raw sludge is shown in Figure 6a. It illustrates a sharp VS/TS ratio decrease to 0.75 at 10th the day of anaerobic digestion. Its value after the 10th day further slowly decreased by 0.0025 per day, achieving a level of 0.68 at the 48th day of AND-time. The decreasing course of VS/TS ratio during aerobic digestion of different AND-time sludges is illustrated in Figure 6b. Its values are gradually lower for the studies with longer AND-time at any stage of aerobic digestion. The lowest values of the above ratio, which is 0.63, were found for 48 days aerobically digested sludge of 38 days AND-time. The outcomes suggest that the MAND/MAD system results in better sludge stabilization as the residence time of anaerobic digestion increases.

Figure 6a. AND-time Changes of raw sludge VS/TS ratio with the duration of AND-time.

Figure 6b. Changes of 17, 24, and 38 days AND-time sludges VS/TS ratio with the duration of AD-time.
4. Conclusions
The retention time of anaerobic digestion in the combined MAND/MAD system plays an essential role in sludge stabilization.

1) The longer is the anaerobic digestion retention time, the less soluble organic matter and EPS are in the supernatant.

2) When compared to sludge with AND-times of 17, 24, and 38 days, sludge with AND-times of 38 days was the most susceptible to dewatering due to the short capillary uptake CST time at the end of the MAND/MAD process.

3) The values of VS/TS ratio for sludge of 38 days AND-time were the lowest among the sludges of three different AND-time with the final value of 0.63 at the end of the MAND/MAD process.

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