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# The impact of sludge pre-treatments on mesophilic and thermophilic anaerobic digestion efficiency: Role of the organic load



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#### HIGHLIGHTS

- Semi-continuous mesophilic and thermophilic digestion, with sludge pre-treatments.
- Enhanced release of lipids by thermal hydrolysis and proteins by ultrasounds.

• Pre-treatments improved organics conversion to CH4 only at low-medium loading rate.

• At high organic load, TAD yield was 0.15 against 0.11 Nm<sup>3</sup> methane/kg COD<sub>fed</sub> of MAD.

• Generated charged polymers in TAD impaired filterability more rapidly than in MAD.

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#### ABSTRACT

Anaerobic digestion has been recognized as the most appropriate stabilization technology for approaching sludge reuse task, in part because of the methane production involved. The mesophilic anaerobic processing train can be upgraded by adding a pre-treatment step or by increasing the digestion temperature to thermophilic conditions. In this study, semi-continuous mesophilic (MAD) and thermophilic (TAD) anaerobic digesters were operated for 180 days at low (0.7-1.0 kg VS/m<sup>3</sup> d), medium (1.4-1.8 kg VS/m<sup>3</sup> d) and high (2.8–3.7 kg VS/m<sup>3</sup> d) organic loading rates (OLRs). The impact of ultrasound and thermal hydrolysis pre-treatments on the mesophilic and thermophilic digestion efficiency, respectively, was assessed by performing parallel digestion tests. The increase of soluble COD (Chemical Oxygen Demand) and colloidal surface charge after the sludge pre-treatments suggested biopolymers solubilization and changes in surface floc properties. Thermal hydrolysis enhanced the release of lipids and long chain fatty acids, while ultrasounds application resulted in proteins being the main component of the released matter. Operating the digesters at OLRs between 0.7 and 1.4 kg VS/m<sup>3</sup> d the methane conversion rate was not significantly affected by the temperature increase up to thermophilic conditions, whereas the integration of the pre-treatments accelerated the organic conversions, resulting in a noticeable methane gain (up to +51%). Conversely, at higher organic loads, the TAD yields were significantly higher with respect to the MAD ones, assuring the sustainable economic benefit of operating smaller anaerobic digesters to obtain higher methane productions. Nevertheless, the colloidal charge increase during thermophilic digestion impaired the sludge filterability much more rapidly than in mesophilic conditions.

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#### 1. Introduction

The "enhanced" digestion approach involves multi-step processes in which the first step generally includes a biomass pre-treatment to render it more amenable for further conversion steps. After pre-treatment, the biomass components are subjected to biological and/or chemical processing, which strives to create energy and/or material recovery. Anaerobic digestion (AD) is an

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efficient and sustainable technology to stabilize sludge by means of mass and pathogen reduction, which strives for energy in the form of methane. Anaerobic digesters are operated at mesophilic (35 °C) or thermophilic (55 °C) temperatures. In general, mesophilic anaerobic digestion (MAD) of sewage sludge is more widely used compared to thermophilic anaerobic digestion (TAD), primarily because of its lower energy requirements and higher process stability [1]. Nevertheless, thermophilic digestion is more efficient in terms of organic matter removal and methane production [2–5]. Moreover, at high temperatures, the destruction of pathogens, weed seeds and insect eggs increases, thus matching

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the hygienization criteria required for land use [6,7]. The design operating temperature establishes the minimum hydraulic retention time (HRT) required to achieve a given amount of volatile solids destruction, and it is possible to feed sufficient substrate by decreasing the HRT and increasing the organic sludge load. Sludge digestion at shorter HRTs have been reported by Appels et al. [8]; however; a major drawback associated with shortening the HRT is the poor destruction of volatile solids [9], which are generally associated with the hydrolysis (one of the four stages of the anaerobic digestion process: hydrolysis, acidogenesis, acetogenesis and methanogenesis) of sludge [10,11]. During hydrolysis, flocs are disintegrated, cell walls are ruptured and extracellular polymeric substances (EPS) are degraded, resulting in the release of readily available organic material for acidogenic microorganisms [8]. Nevertheless, for waste activated sludge (WAS), hydrolysis has been identified as the rate-limiting step in AD [8]. Renewed interest in anaerobic digestion arose from the possibility of performance improvement by adding an appropriate thermal, mechanical or chemical WAS pre-treatment leading to the breakage of flocs and particulate solubilization, which accelerates the hydrolysis step [10,12,13].

Regarding thermal pre-treatments, increments in soluble COD of approximately 25% and 60% have been reported after the thermal hydrolysis of secondary sludge at 130 and 170 °C, respectively [13,14]. However, Bougrier et al. [15] and Climent et al. [3] underlined the impact of the solubilization of particulate organic matter on biogas production enhancement during anaerobic digestion. The energetic expense can be balanced due to the increment in sludge biodegradability and the use of sludge residual heat for maintaining the digester temperature [13]. It has been hypothesized that thermal pre-treatment can enhance the biological activities of some thermophilic bacterial populations; for this reason, it can be considered a valid pre-treatment step before thermophilic digestion [16,17]. However, very few studies have addressed the combination of thermal pre-treatment and the thermophilic anaerobic digestion of sludge [1,4,16,18].

Additionally, mechanical sludge pre-treatment as ultrasounds can induce considerable changes to the physical, chemical and biological properties of sludge, which may permit the process to bypass the rate-limiting hydrolysis step, leading to either an increased biogas production with a reduced sludge quantity or shorter retention times [10–12,19,20] at mesophilic conditions.

Despite research efforts to investigate the effect of retention time and organic load on anaerobic mesophilic systems [12,21], the availability of satisfactory information concerning thermophilic anaerobic digestion and pre-treatments effects [1,3] in a continuous system [4,17,22] needs more investigation. Few literature articles [23-25] have focused on the effects of ultrasound pre-treatment on thermophilic digestion, and these studies highlighted the fact that the high efficiency of thermophilic anaerobic digestion was only slightly improved when combined with ultrasonic pre-treatment. Investigations conducted by Gavala et al. [1] on the AD of primary and secondary sludge reported higher efficiencies in solids removal and methane production in thermophilic processes at lower retention times than in mesophilic conditions. Thermophilic operations of a single stage reactor fed with activated sludge performed better in terms of solids removal (up to +12%) and biogas production (up to +25%) compared to the control mesophilic digester for all the SRTs tested [22].

Floc disintegration and solubilization by either bacterial hydrolysis or appropriate pre-treatments favor the release of soluble and colloidal charged fines that could potentially lead to worsening of dewaterability [2,7,13,26–28]. In fact, proteins and polysaccharides, which are the main components of EPS, have negatively charged functional groups, which affect sludge filterability through sequence specific interactions, hydrophobic interactions and hydrogen bonding [29]. Regarding the filterability of sonicated sludge, Chu et al. [30] reported that the capillary suction time (CST) for 0.33 W/mL of sonicated sludge increased 2.5 times after 1 h of sonication. In this case, the filterability of the sludge worsened significantly by the ultrasonic treatment due to the large amount of water attached to the increased surfaces on the small particles after sonication [9].

Regarding WAS dewaterability after thermal hydrolysis, several studies available in the literature have reported a deterioration of sludge filterability after applying temperatures lower than 150 °C; this deterioration was caused by the increase of colloidal and soluble particles. Conversely, for temperatures higher than 150 °C, the filterability results improved. This could be explained by the modification of the sludge structure and the released of linked water [31].

In this work, ultrasound and thermal pre-treatments were compared regarding the conversion of sludge into a soluble, more biodegradable form and the impact of the organic load on the successive anaerobic digestion process. Most research papers have only reported on the application of one type of sludge pre-treatment, making it difficult to compare the efficiency of different methods. Indeed, the operating conditions and sludge characteristics were not similar in many cases. Moreover, regarding digestion performances, the majority of the literature studies on the effect of sludge pre-treatment and thermophilic conditions on biodegradability and biogas production were conducted primarily in batch assays, and little work has been done on anaerobic digestion in semi-continuous digesters, particularly on the effects of enhanced solubilization and EPS release on the final sludge dewaterability [12,32,33].

The purpose of this study was to investigate the performances and treatment efficiency of sludge digestion in semi-continuous lab scale digesters at mesophilic and thermophilic conditions. This purpose was achieved using different organic loading rates and by treating real waste activated sludge from the same WWTP, with or without pre-treatment integration. The digestate quality after the different stabilization processes was evaluated in terms of dewaterability and the soluble load of organic compounds.

#### 2. Materials and methods

#### 2.1. Sludge origin

Waste activated sludge (WAS) was sampled from the municipal "Roma-Nord" wastewater treatment plant, which is one of the four wastewater treatment plants serving the city of Rome and has an organic load of approximately 700,000 p.e. The WWTP includes screening, primary clarification and secondary treatment of activated sludge with a high sludge age (20 days). The secondary sludge was collected from the recycling stream before it reached the thickener. The anaerobic inoculum was collected from the full-scale digester of the plant, which is fed with primary and secondary sludge.

The characteristics of the WAS are reported in Table 1. The sludge used as feed was characterized by a very low VS content due to its high sludge age.

Table 1	
Characteristics of the WAS samples collected from the Roma Nord W	WTP.

Parameter	Range
Total solids (g/L)	20.5-23.5
Volatile solids (g/L)	13.5-14.5
Soluble COD (mg/L)	50-100
Soluble N <sub>tot</sub> (mg/L)	20-45
Soluble P-PO <sub>4</sub> <sup>3–</sup> (mg/L)	50-55
pH	6.5-7.0

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