Microwave/Ultrasound Based Sludge Treatment Methods

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The amount of waste activated sludge (WAS) is increasing annually, and since it presents potential environmental and health-related risks, an appropriate treatment and stabilization process is needed. It has been shown in numerous studies in the past few decades that amongst the advanced treatment methods of sludge, microwave and ultrasound-based processes offer promising and effective alternatives. The main advantage of these physical methods is that they are energy-efficient, easy to implement and can be combined with other types of treatment procedures without major difficulties.

Keywords: sludge ; sludge treatment ; microwave ; ultrasound

1. Introduction—General Aspects of Sludge

The waste residue which is generated during a variety of processes in a wastewater treatment plant is called sludge. Since it contains residual (mostly organic) pollutants, pathogenic microorganisms and other toxic compounds that originated from the treated wastewater, sludge is potentially harmful to both the environment and health. Moreover, it has been shown that during the conventional activated sludge process, the extent of waste activated sludge (WAS)—which is the by-product of the microbial organic pollutant removal subprocess—have been continuously increasing annually in the last few decades, and definitely will further in the near future [1]. Therefore, the proper treatment and stabilization of it are inevitable, however, the most significant problem is that these processes have high energy demands and costs. During a conventional activated sludge process, the expense for WAS treatment is costly, and the cost of its treatment can cover 50–60% of the total cost of a wastewater treatment plant [2][3].

The main objective of the activated sludge process is to generate a mixture of primary sludge (PS) and WAS which can be used for fertilization or can be stabilized and utilized afterwards via anaerobic digestion (AD). Anaerobic digestion or fermentation uses specific microorganism which converts organic substances to methane and carbon dioxide through different biochemical processes, which involves the hydrolysis of proteins, lipids and carbohydrates; fermentation of amino acids and sugars to acids (acidogenesis); oxidation of fatty acids and alcohols into acetic acid, CO_2 and hydrogen, and the conversion of acetic acid and hydrogen to methane [4].

2. Efficiency Indicators

It is evidently clear that to characterize a treatment or pre-treatment process in terms of its effectiveness, a specific indicator (or indicators) should be determined which allows the comparison of the different methods. In sludge (pre-) treatment methods, depending on the approach, several of these indicators can be defined, however, the scientific literature and research articles do not always use them consequentially, and thus the comparison of the different processes has its limitations.

In environmental sciences, chemical oxygen demand (COD) is an indicative measure of the amount of oxygen that is needed to oxidize the organic matter content of a given sample. Typically, COD is given in mgO_2/L. COD measurement and the determination of COD level is widely used in wastewater and sludge treatment, for the reason that the measurement is rather quick (especially when compared to the biochemical oxygen demand, BOD determination), and gives accurate information about the organic content of a wastewater or sludge sample [5]. When comparing different processes in terms of organic matter elimination, the reduction rate of COD can give a precise insight about how efficient a given method is, i.e., the more it can reduce the COD level, the more effective it is. “Pure” COD, however, is usually too general and sometimes does not provide enough information about the applied treatment method. Therefore it is favourable to split it into two different variants, the soluble chemical oxygen demand (SCOD) and the total chemical oxygen demand (TCOD). SCOD, as its name suggest, contains only those organic materials that can be found in the soluble phase, whilst TCOD contains all organic content (as well as in the soluble and in the solid phase). The ratio of these two (SCOD/TCOD) can accurately indicate what proportion of the total COD can be found in the soluble phase, and as its extent correlates with anaerobic digestibility [6], this ratio is commonly used in the scientific literature when comparing different methods or processes in sludge treatment.

Biochemical oxygen demand (BOD) determination serves a similar purpose as COD, however, both the method and its value differ from that of chemical oxygen demand. BOD also measures the amount of oxygen needed to break down organic compounds, although the determination process is based on microbial activity, i.e., how much oxygen do specific aerobic microorganisms need to oxidize the organic matter of the sample [7].

Disintegration degree (DD) is one of the most common indicators in sludge treatment. DD is calculated as the ratio of SCOD increase caused by the analysed disintegration method in relation to the SCOD increase caused by the chemical disintegration [8]. As its definition suggests, a given process can be evaluated in terms of its effectiveness by calculating DD; the more increment the process can cause in DD, the more it disintegrates the structure of the sludge, hence increasing the SCOD, and ultimately, the anaerobic digestibility. The estimation of WAS disintegration degree is generally based upon the values of SCOD, STOC and STN, as suggested by Ren et al. [9]. From an experimental point of view, there are some novel methods for the assessment of sludge DD, for example, differential centrifugal sedimentation [10].

Total solid (TS) and volatile solid (VS) percentages are also rather important in terms of sludge characterization, and for that commonly used as indicators as well.

During the treatment of wastewater in a wastewater treatment plant, an excessive amount of wastewater sludge is produced. The resulting sludge can be and is used for either fertilization purposes [11][12] or anaerobic digestion (AD), i.e., biogas production. The product of the AD, as discussed in the Introduction, is mainly methane and carbon dioxide. It can be stated that the more methane is being produced during AD, the more effective the process is (but of course there is a limitation in CH_4 extent). For these considerations, scientific literature often uses methane yield as an indicator, and when comparing different treatment or pre-treatment processes to each other in terms of effectivity, it is usually calculated how they can increase the overall methane yield in the following AD process. A slightly simpler method is to determine the exact biogas yield; however, it carries less information about the usable content of the biogas itself.

3. Principles of Chemical Treatments: Alkaline and H_2O_2

Alkaline compounds have been frequently used as a method in wastewater and sludge treatment to enhance several factors, such as anaerobic digestibility, disintegration or solubilisation. The addition of alkali increases the amount of hydroxyl ions in the material, and, therefore, its pH level. This means that the environment becomes hypotonic, which causes the turgor pressure in the microbial cells to increase to an extent the cell can no longer sustain, and, therefore, its cell wall breaks down [9]. By using an alkaline treatment, the biodegradability of the sludge can be significantly increased, as it induces the swelling of particulate organics, making them more accessible to enzymes [10]. Alkaline treatment can also enhance the solubilisation of COD; Kim et al. determined that by adding NaOH in a dosage of 5–21 g/L, approximately 40% of COD solubilisation of sludge can be achieved [13]. As shown in several studies, the addition of alkaline can affect the degree of disintegration and anaerobic biodegradability as well—in a recent 2021 study, Ayesha et al. have shown that standalone alkaline treatment (via 0.8% NaOH) of dewatered WAS resulted in an 11.3 disintegration degree and 32.6% higher methane yield, compared to the control samples [14]. Erkan et al. showed that during the electrochemical treatment of WAS, the initial pH of the sludge sample plays a role in terms of DD–higher pH values obtained via the addition of NaOH resulted in the increase of DD (from 3.8% to 7.33%) [15].

Naturally, the concentration or the dosage of the applied alkaline also plays a key role in terms of effectiveness. Sahinkaya and Sevimli showed that 0.05 N was the optimal concentration of NaOH [16], while Penaud et al. in 1999 concluded that 0.125 N was the most favourable, and resulted in a 40% increment in biodegradability [17].

However, standalone alkaline treatments have numerous disadvantages. The addition of various chemicals on one hand increases the overall cost requirements of the process [18], and on the other hand, can inhibit the anaerobic digestion during some conditions—it was reported that too high concentrations of K or Na-ions can inhibit the AD process [19].

Hydrogen peroxide (H_2O_2) is a highly reactive chemical compound, mainly used as an oxidizer; a low-cost strong oxidant, which produces clean oxidation product-water [20]. Numerous studies have shown that in wastewater and wastewater sludge treatment H_2O_2 can be implemented in various ways, mostly in Fenton’s/Fenton-like reactions or in a combination with physical treatments, such as microwave irradiation or ultrasonication [21][22].

4. Microwave Irradiation

In the electromagnetic spectrum, microwave (MW) irradiation occurs in a frequency range of 300 MHz to 300 GHz with a corresponding wavelength of 1 mm–1 m. MW irradiation is considered a promising alternative to conventional heating methods. In wastewater and wastewater sludge treatment, during microwave irradiation, the destruction of microorganisms and other molecules may occur in two ways: thermal and athermal (non-thermal) effects. Thermal effects are generated via ionic conduction (in shorter frequencies) and dipole rotation (in higher frequencies)—the former means the electrophoretic (conductive) migration of dissolved ions in the electromagnetic field [23], while the latter is generated through the rotation of dipole molecules (like water) due to the constant and repeated changes in the polarity of the field [24]. Athermal effects are induced by the change in dipole orientation of certain polar molecules, which increase the possibility of breaking down the hydrogen bonds of biopolymers (polysaccharides, proteins, DNA, RNA) [25][26]. In industrial use, a frequency of 915 MHz is the most favourable, since shorter frequencies have higher penetration depth [27], thus increasing the extent of thermal and athermal effects.

It was concluded that the SCOD/TCOD ratio increased from 2% to 22% of sludge after MW irradiation (630 kJ total irradiated energy) [28]. SCOD/TCOD ratios were also increased in waste activated sludge from 8% to 18% after microwave-based heating to 72.5 °C [29] and from 6% to 18% after MW heating to 96 °C [30]. Gil et al. reported that depending on the applied total energy and power ratings, a 43% to 66% of increment occurred in the solubility (COD/TVS ratio) of floated sewage sludge [31].

Standalone microwave irradiation can also increase the extent of biogas production from sludge. Waste activated sludge (WAS) heated to different temperatures through microwave treatment resulted in a higher rate and extent of biogas production [32]. Alqaralleh et al. showed that the microwave heating of thickened waste activated sludge up to 175 °C resulted in a 135% higher biogas yield compared to the control samples [33]. Applying a total of 14.000 kJ/kgTS microwave energy resulted in a +570% biogas yield, as reported by Ebenezer et al. in 2015 [34]. In another experiment, the effects of microwave irradiation on the removal of COD were investigated: Park et al. reported that the treatment of WAS by MW to 91 °C, 64% of COD decrement could be achieved [35]. Combination of microwave irradiation with ultrasonication can also be a promising method in wastewater and wastewater sludge treatment: Mesfin Yeneneh et al. applied ultrasonic irradiation (0.4 W/mL, 6 min) after MW treatment (2450 MHz, 3 min), which resulted in a higher cumulative biogas production compared to the control samples [36].

5. Ultrasound Treatment

Ultrasounds are longitudinal acoustic waves in the frequency range of 20 kHz and 10 MHz. Just like other acoustic waves, ultrasounds act differently depending on the material they are going through. To express to which extent the ultrasound can be absorbed in the irradiated material, the following expression can be used [37]:

$$A = A_0 e^{-\alpha x} \quad (1)$$

In the equation, A_0 represents the initial amplitude of the ultrasonic wave, x means the length of path and α is the attenuation coefficient.

The effects of ultrasonic treatments are mostly due to the cavitation process in the treated material. During this process, alternating high-pressure (compression) and low-pressure (rarefaction) cycles occur, and the rate is frequency-dependent. The so-called transient cavitation bubbles usually last for only a few cycles [38], their size can significantly increase, and when these bubbles reach a volume at which they can no longer absorb any more energy, they viciously collapse during a high-pressure cycle. During this collapse, extremely high pressure and temperature can be reached locally [39]. According to Ashokkumar, the theoretical temperature can be calculated via the following expression [40]:

$$T = T_0 \left(\frac{P_m (\gamma - 1)}{P_v} \right) \quad (2)$$

In the expression, T_0 is the ambient solution temperature, P_m demonstrates the sum of the hydrostatic and acoustic pressures, y is the specific heat ratio of the gas/vapour mixture and P_v is the pressure inside the cavitation bubble when it reaches its maximum volume.

These cavitation effects can undoubtedly cause severe structural, physical and chemical changes in the exposed material, such as wastewater or wastewater sludge. The use of sonication in wastewater treatment goes back to the late

1990s and early 2000s, and since then the various effects of the process have been heavily studied. It was shown that the hydromechanical shear force is the dominant effect when treating wastewater and sludge with ultrasonication [41], however other factors like locally high temperature and pressure or the formation of free radicals (H and OH; due to the extreme local temperatures) can also play a significant role in various mechanisms (e.g., sludge disintegration or solubilization).

Ultrasound treatment of sludge mainly results in the solubilization of organic particles and less in mineral particles, as shown by Bougrier et al. [42]. They reported that at a specific energy input of 15,000 kJ/kg TS, 29% of the organic particles were solubilized, whereas only 3% of mineral particles were solubilized. Solubilization of COD is mostly due to the disintegration of extracellular polymeric substances (EPS) [43]. These substances are high molecular weight polymers, which play a key role in floc size, stability and bioflocculation. When WAS is exposed to ultrasonication and the various effects caused by it, these EPS are shattered along with microorganism flocs and the key components of EPS (proteins, carbohydrates) and the intracellular substances of microbial cells (enzymes, DNA, carbohydrates) enter the soluble phase [44]. This will lead to an increased SCOD/TCOD ratio, which was shown to be beneficial in terms of biogas production [45]. In the study of Tian et al., it was also reported that the ultrasound irradiation of sludge resulted in a significant increase in loosely bound polysaccharide (PS) contents, and also in carbonyl, hydroxyl and amine functional group contents [46]. Several studies prove that ultrasonic pre-treatment of sludge can cause a significant increase in biogas production and volatile solid destruction [47][48]. Daukyns et al. stated that by disintegrating sludge with ultrasonic treatment, the methane content in the produced biogas was almost 72%, whereas in the case of non-disintegrated sludge, only 54% [49].

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