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SONICATION AND ELECTROOXIDATION FOR MUNICIPAL WASTEWATER PRIMARY SEWAGE SLUDGE STABILIZATION

During wastewater treatment (WT), sewage sludge (SS) is generated, containing organic matter and pathogenic microorganisms that pose health risks and must therefore be stabilised. Several authors have reported the stabilisation of secondary SS (sludge from the WT bioreactor) for its use as a soil fertiliser. However, to date, primary SS (sludge from the primary settler or chemical precipitation tank in WT) remains underutilised. In this study, we applied sonication and electrooxidation to primary SS to reduce concentrations of total coliforms, faecal coliforms (FC), and chemical oxygen demand (COD). We show that sonication reduces FC concentrations in wastewater SS twice as effectively as electrooxidation, and that this process is influenced by the interaction between treatment time and applied pH. Additionally, the interaction of pH and current intensity affects the reduction of FC and COD in primary SS treated by electrooxidation. Our results demonstrate that sonication at pH 5 for 10 min reduces FC concentration in primary sludge by 31% compared to untreated sludge. Complementary treatments are necessary to further reduce the concentrations of pathogenic microorganisms.

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1. INTRODUCTION

Sewage sludge consists of semisolid waste (75% of water) that has a high content of organic matter (70% as dry matter) and nitrogen (3–5% as dry matter) [1], and can also contain pathogens such as bacteria, virus, fungi, protozoa, and helminths that represent risks for the health of humans, animals, and plants [2], and many organic components that produce odours [3]. Sometimes, sewage sludge can contain inorganic and organic hazardous contaminants such as heavy metals, polycyclic aromatic hydrocarbons [2], polychlorinated biphenyls, pesticides, surfactants, hormones, pharmaceuticals, nanoparticles [4], microplastics, dioxins, and others [5].

The improper use and disposal of sewage sludge cause severe environmental impacts and risks to human health. The final disposal of sewage sludge is one of the most expensive aspects in wastewater treatment plants (WWTPs), accounting for about 50% of the plant's operating costs. Specifically, wastewater treatment by activated sludge generates approximately 0.25 kg of sludge on a dry basis (primary and secondary) per cubic meter of treated wastewater [1]. Therefore, the search for and application of economically feasible treatments represents one of the most important missions for environmental authorities [6].

Relative to the point of the wastewater treatment plant where the sludge is produced, it is generally classified as primary sludge (this comes from the primary settler or the chemical precipitation tank); secondary sludge, which is also known as activated sludge (and which comes from the biological reactor); mixed sludge (as the name implies, it is a mixture of primary and secondary sludge), and tertiary sludge (generated in the tertiary treatment of wastewater) [7]. Separately or together, these sludges are taken to the treatment system, which generally begins with the thickening of the secondary sludge. Sludge is stabilised to reduce pathogens, eliminate odours, and reduce or eliminate the potential for decomposition that develops odours and attracts vectors [8]. Stabilisation processes can produce different qualities of sludge or biosolids, depending on the type of stabilisation they provide.

Sludge can be treated by biological, physical, and chemical methods. Electrooxidation is a chemical process that is aimed at mineralising organic compounds. In this process, a direct current is applied in an electrochemical cell, leading to an electrochemical conversion and generating free radicals, which are responsible for the degradation of organic pollutants, and, in particular, hydroxyl radicals (OH^-) are responsible for the attack on organic compounds [9]. On the other hand, sonication is a physical process that is due to the phenomenon of acoustic cavitation, which is the formation and collapse of microbubbles occurring in milliseconds and producing extreme temperature and pressure gradients [10]. Exposure of microbial cells to the energy of sonication breaks the membrane and cell wall, freeing the cellular components in the solution, which favours digestibility [11]. Sonication has the advantage of being simple, economical, and environmentally friendly, while not requiring heat treatment [12].

This study aimed to select conditions for electrooxidation and sonication processes in order to decrease total and fecal coliforms and COD concentrations in primary sewage sludge from municipal wastewater treatment.

2. MATERIALS AND METHODS

Sludge sample. Sampling of the sludge was completely random, and the samples were taken from the primary settling tank of a treatment plant in Ecatepec (geographic coordinates 19°35′57″N 99°02′57″O), State of Mexico, Mexico, which is fed by municipal wastewater from the Grand Canal of Ecatepec.

Physicochemical parameters. The chemical oxygen demand (COD) was determined by using the closed reflux method (5220B), total organic carbon by wet oxidation method (5310D), ammonia nitrogen (4500C), conductivity (2510B), pH (4500H), settleable solids (2540F), total solids (2540B), volatile solids (2540E), total suspended solids (2540D), and total dissolved solids (2540C) following standardised methods [13].

Metal concentrations in the aqueous solution of the sludge were measured using inductively coupled plasma atomic emission spectroscopy (ICP-AES).

Microbiological parameters. For the determination of total and fecal coliforms, the most probable number (MPN) technique modified from 9221B and 9221E, respectively, was used [13]. Decimal dilutions were made in 0.85% saline solution until the final dilution of 10^{-7}. For the quantification of total coliforms (presumptive test), 1 cm^3 of each dilution was inoculated in 3 tubes with 9 cm^3 of sodium lauryl sulfate broth, and incubated for 48 h at 35±1 °C. The gas present in the Durham tubes produced during the incubation indicated a positive test result. For fecal coliforms (confirmatory test), the positive tubes from the presumptive test were selected, and 1 cm^3 was added to a tube with 9 cm^3 of 2% brilliant green bile broth and incubated for 48 h at 44.5±1 °C, considering the test to be positive if the Durham tube had gas present. The results for both total and fecal coliforms were expressed in terms of MPN/cm^3 of the number of tubes with positive results. All samples were analysed in triplicate.

Helminth eggs and concentration of *Salmonella* spp. were determined by the standardised methods 9260B [13] using tetrathionate broth as enrichment medium and brilliant green agar as isolation medium.

Electrooxidation treatment. Electrooxidation was carried out using an electrochemical reactor RU4 (Betts Environmental, USA) with a capacity of 5 dm^3 operated in a batch in the laboratory at 50% capacity (2.5 dm^3). The reactor consisted of three electrodes, two carbon anodes (dimensions 4.5 × 21 × 1 cm) and a rotating steel cathode (14 cm in diameter and 15 cm high). The current was supplied by a B&K Precision model

1901B power supply (Yorba Linda, California, USA) with a maximum capacity of 30 A. The current intensity, pH, and time were varied according to the experimental conditions established in a 2^3 factorial design (Table 1), resulting in 8 treatments with 3 replicates each, along with residual sludge as the control (C1).

Table 1

Design of the experiment

Treatment	Electrooxidation			Sonication		
	Time [s]	Current intensity [A]	pH	Time [min]	Temperature [°C]	pH
1	10	0.2	5	10	6	5
2	50	0.2	5	50	6	5
3	10	3	5	10	45	5
4	50	3	5	50	45	5
5	10	0.2	8.5	10	6	8.5
6	50	0.2	8.5	50	6	8.5
7	10	3	8.5	10	45	8.5
8	50	3	8.5	50	45	8.5

Sonication treatment. Sonication was applied to 100 cm^3 of sludge in a glass flask, which was placed inside a Branson model 2210 sonicator with a frequency of 40 kHz and 80 W of power using distilled water as a diffusion medium. The operating time, pH, and water temperature were adjusted according to a 2^3 factorial design (Table 1), resulting in 8 treatments with 3 repetitions each, as well as residual sludge as a control (C1).

Statistical analysis. From the results of the 2^3 factorial design for the electrooxidation and sonication treatments, a data matrix was created where the total and fecal coliforms, as well as the COD evaluated in the treatments, were taken as response variables, and the variables corresponding to each of the treatments were taken as input variables (Table 1). The interpretation of the results was carried out utilizing an analysis of variance (ANOVA) with a 95% confidence interval using the Minitab 17® software. Response surface graphs were developed with the help of the Design Expert® version 11 statistical software.

3. RESULTS AND DISCUSSION

3.1. CHARACTERISATION OF SLUDGE

The limitation regarding the application of sludge to soil lies in the concentrations of metals and pathogens [15], although in the analysed sludge, the metals have very low concentrations (Table 2) compared to what was reported by Martín et al. [1] and Sava et al. [16]. As in the present study, low concentrations of metals were also reported in residual sludge in the study by Pliego-Bravo et al. [17] (see Table 2), compared to other authors and the maximum permissible limits of Mexican regulations. In contrast, the coliform count, both total and fecal (Table 3), exceeds the maximum permissible limits for biosolids following Mexican regulations [14], therefore, considering this parameter, sludge is not viable as a soil improver. The presence of *Salmonella* spp. was not detected, nor were helminth eggs detected. The rest of the parameters that were determined in this study are not mentioned in NOM-004-SEMARNAT-2002 [14], although primary sludge contains micro and macro nutrients such as carbon, nitrogen, and phosphorus that are important for plant growth [18].

Table 2

Metal content in primary sludge
(Ecatepec Wastewater Treatment Plant, Mexico State, Mexico)

Element	Concentration [mg/kg]	Maximum Mexican limit for biosolid quality [14] [mg/kg dry basis]		Concentration [mg/kg dry basis]	
		Excellent	Good	Martín et al. [1]	Sava et al. [16]
Ag	0.005±0.002	N.A.	N.A.	N.A.	N.A.
Al	N.D.	N.A.	N.A.	N.A.	N.A.
As	N.D.	41	75	N.A.	0.638±0.758
Ba	0.117±0.012	N.A.	N.A.	N.A.	N.A.
Be	N.D.	N.A.	N.A.	N.A.	N.A.
Ca	78.376±13.892	N.A.	N.A.	N.A.	N.A.
Cd	0.001±0.0002	39	85	6.5±2.1	1.017±0.618
Co	0.003±0.0004	N.A.	N.A.	N.A.	5.32±2.83
Cr	0.092±0.008	1200	3000	89.5±2.1	73.0±27.2
Cu	0.174±0.020	1500	4300	261.5±55.9	406±63
Fe	5.806±0.675	N.A.	N.A.	5450±400	N.A.
K	2.738±0.346	N.A.	N.A.	10 400±200	N.A.
Mg	4.092±0.357	N.A.	N.A.	18 300±800	N.A.
Mn	0.116±0.012	N.A.	N.A.	N.A.	N.A.
Mo	0.007±0.001	N.A.	N.A.	N.A.	N.A.
Na	7.871±1.027	N.A.	N.A.	2000±600	N.A.
Ni	0.040±0.003	420	420	39±5.7	34.0±7.3
Pb	0.023±0.005	300	840	39.5±10.6	39.9±5.9
Sb	0.006±0.002	N.A.	N.A.	N.A.	N.A.
Se	N.D.	N.A.	N.A.	N.A.	N.A.
Sn	N.D.	N.A.	N.A.	N.A.	N.A.
Sr	N.D.	N.A.	N.A.	N.A.	N.A.
Ti	0.230±0.012	N.A.	N.A.	N.A.	N.A.
Tl	N.D.	N.A.	N.A.	N.A.	N.A.
V	0.036±0.004	N.A.	N.A.	N.A.	N.A.
Zn	0.466±0.048	2800	7500	378±2.8	1453±297
Hg	N.D.	17	57	N.A.	0.50±0.12

N.D. – not detectable, N.A. – not applicable.

Table 3

Sewage sludge characteristics
(Ecatepec Wastewater Treatment Plant, Mexico State, Mexico)

Parameter	Primary sewage sludge	Maximum limit Mexican regulations [14]
Total coliforms, MPN/cm^3	$3.63\times10^9\pm1.15\times10^9$	N.A.
Fecal coliforms, MPN/cm^3	$2.24\times10^8\pm1.81\times10^8$	< 1000
Salmonella spp.	N.D.	< 3
Helminth eggs	N.D.	< 1
pH	6.77±0.06	N.A.
Apparent density, g/cm^3	1.030±0.013	N.A.
Conductivity, dS/m	3.78±0.01	N.A.
Organic carbon, mg/dm^3	704±27.71	N.A.
Settleable solids, cm^3/dm^3	991.67±2.89	N.A.
Total solids, mg/dm^3	72 843.33±1818.52	N.A.
Volatile solids, mg/dm^3	36 918.33±2106.43	N.A.
Suspended solids, mg/dm^3	34 085±1 643.06	N.A.
Dissolved solids, mg/dm^3	1840±200.19	N.A.
COD, mg O_2/dm^3	12 598.33±1298.95	N.A.
Soluble phosphorus, mg P-PO_4^{3-}/dm^3	8.795±2.31	N.A.
Ammoniacal nitrogen, mg N-NH_4^+/dm^3	13.908±2.07	N.A.
Nitrates, mg NO_3^-/dm^3	N.D.	N.A.

N.D. – not detectable, N.A. – not applicable.

The primary sludge that was studied has different characteristics in relation to other types of sludge, such as the mixture of primary and secondary sludge, as reported in the study by Tian et al. [19], which notes higher values of COD (19 500–25 000 mg/dm^3) and lower values for total solids (16 200–17 200 mg/dm^3). It is noteworthy that in the primary sludge characterised in this study, there is a high concentration of total solids present. Of these, approximately 50% correspond to volatile solids, which are indicators of organic matter [20] and, therefore, degradable material in the sludge. The values of each of the parameters in the sludge vary according to the nature of the wastewater from which they come, as demonstrated by comparing the values of the present study (Table 3) with those reported in the study by Martín et al. [1], which noted higher concentrations of COD (25 706 mg/dm^3) and ammoniacal nitrogen (723 mg/dm^3), but lower concentrations of total solids (24 400 mg/dm^3), although in both cases it is sludge from the treatment of municipal wastewater.

3.2. TOTAL AND FECAL COLIFORMS

For electrooxidation, the quantity of total coliforms was affected by time ($p = 0.020$), current intensity ($p < 0.0001$), pH ($p < 0.0001$), and, additionally, by the interaction between pH and current intensity ($p < 0.0001$). The lowest concentration of total coliforms was obtained after applying a current of 0.2 A with pH 5 (Fig. 1a), obtaining a value of 8.71log MPN/cm^3 which is 0.83 logarithmic units lower than that of the untreated sludge.

For the sonication treatment, the interactions between the factors were statistically significant for total coliforms: time–temperature ($p = 0.025$), time–pH ($p = 0.007$), and temperature–pH ($p < 0.0001$). Accordingly, the lowest concentration of total coliforms in units of log MPN/cm^3 was 6.55 at 45 °C with pH 8.5 for 50 min, as shown in Fig. 1.

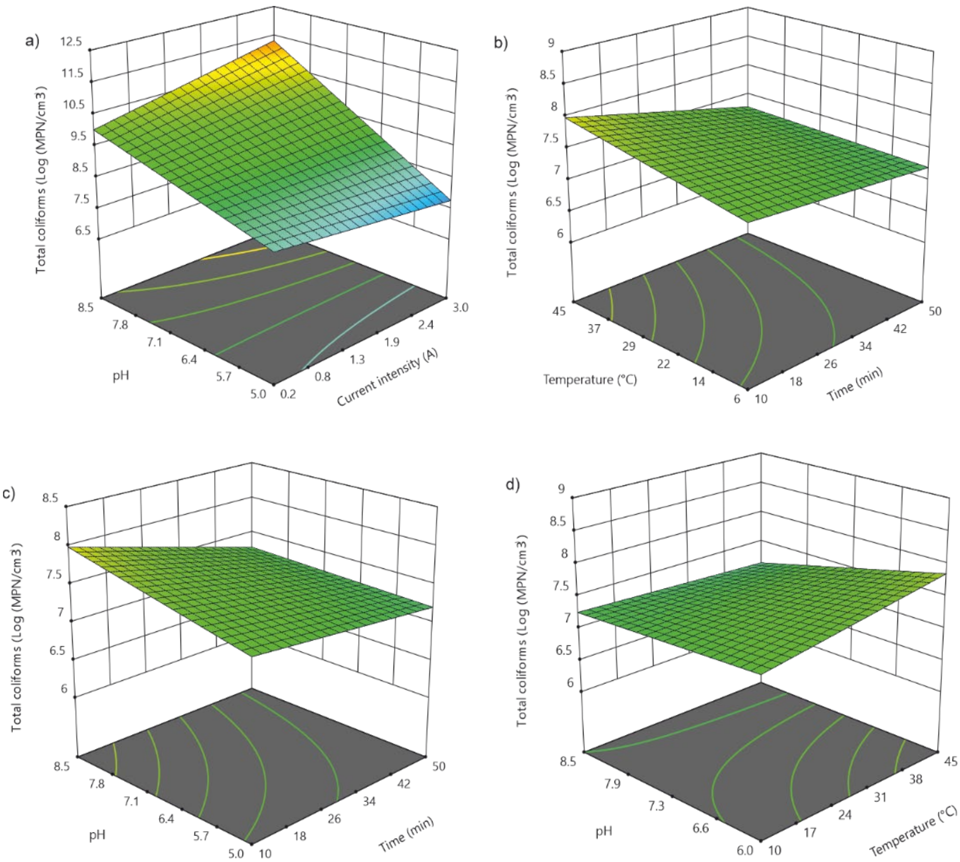
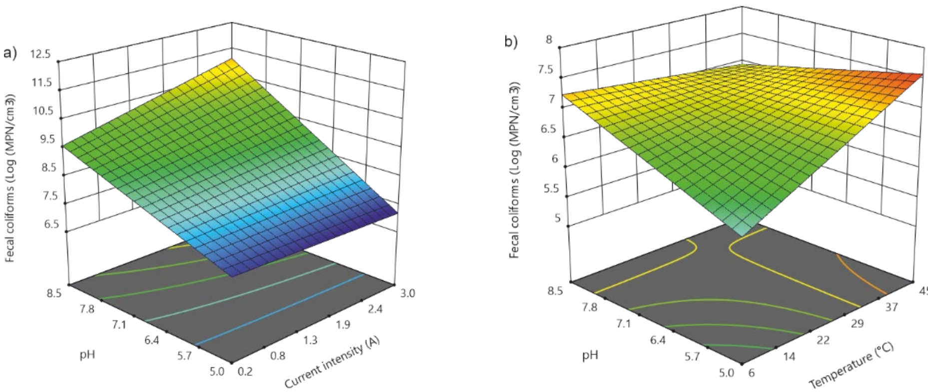


Fig. 1. Total coliform concentrations in primary sludge:
a) electrooxidation treatment as a function of pH and current intensity,
b) sonication treatment (40 kHz, 80 W) as a function of temperature and time,
c) sonication treatment (40 kHz, 80 W) as a function of pH and time
d) sonication treatment (40 kHz, 80 W) as a function of temperature and pH

The statistically significant factors for the decrease in fecal coliforms by the electrooxidation treatment were the intensity of current ($p = 0.002$), the pH ($p < 0.0001$), as well as the interactions of these factors ($p < 0.0001$). The lowest concentration of fecal coliforms was obtained by applying a current intensity of 0.2 A with pH 5 and 3 A with pH 5 (Fig. 2a), obtaining a decrease in fecal coliforms in 2.58 logarithmic units relative to untreated sludge. The disinfection efficiency obtained in the present study was higher compared to that which was reported in the study by Wang et al. [21], where the reduction of 2 logarithmic units of *E. coli* was indicated after applying 4 V, but for a shorter time (5 min), and the total inactivation in 20 min. This was performed in an electrochemical cell with a stainless steel cathode and an anode composed of BiO_x/TiO_2. A decrease in coliforms of sewage sludge by the electrooxidation treatment is because oxidants as hydrogen peroxide (H_2O_2), hydroxyl radicals ($^{\bullet}OH$), hypochlorous acid, and hypochlorite (HOCl and OCl^-) can be produced in the electrochemical process, breaking down the cellular membrane of microorganisms. On the other hand, the electric force created by the input charges can cause the dissolution of the cytoplasm [22].



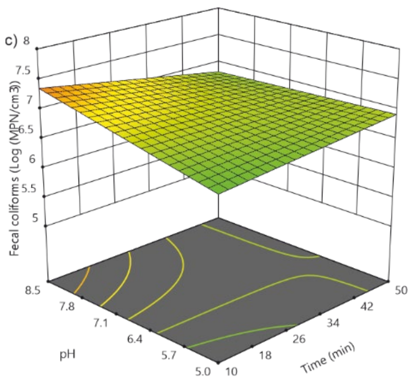


Fig. 2. Coliform concentrations in primary sludge: a) electrooxidation treatment as a function of pH and current intensity, b) sonication treatment (40 kHz, 80 W) as a function of pH and temperature, c) sonication treatment (40 kHz, 80 W) as a function of pH and time

For the sonication treatment, the factors temperature ($p < 0.0001$), pH ($p = 0.008$), as well as the interaction between them ($p < 0.0001$), were statistically significant on the concentration of fecal coliforms. The interactions between temperature and time, as well as pH and time, were also statistically significant ($p < 0.0001$). Figures 2b and 2c show that the lowest concentration of fecal coliforms (5.72log MPN/cm^3) was obtained by applying sonication for 10 min, at 6 °C with pH 5. In contrast to the results obtained in this research, Antoniadis et al. [23] observed that at high power (450 W) and lower frequency (24 kHz), the colonies of *E. coli* in synthetic wastewater were almost eliminated, as was the total microbiological load in municipal wastewater in short sonication times. Reports indicate that low sonication frequencies (ca. 20 kHz) remove fecal coliforms and other pathogens from sludge, due to cavitation-induced shear stress [24], because ultrasound damages the cell walls and cytoplasmic membranes of microorganisms, although the efficiency of ultrasound depends on the type of cells that are present (whether or not they have a capsule, for example) and, therefore, depends on the mechanical properties of the cells [21].

3.3. CHEMICAL OXYGEN DEMAND (COD)

The COD of the treatments tested with electrooxidation was significantly influenced by the current intensity ($p = 0.003$), the pH ($p < 0.001$), and the interaction of these two variables ($p = 0.006$). As shown in Fig. 3a, the lowest COD value (7381.67 mg/dm^3) was obtained by applying 3 A with a pH of 5, which represents a removal of 58.59% of COD in the sludge. This indicates that the higher the current intensity, and the lower the pH value, the greater the COD removal. Under acidic pH conditions, hydroxyl ion-removing compounds such as carbonate and bicarbonate are depleted, resulting in greater oxidation of organic compounds [25].

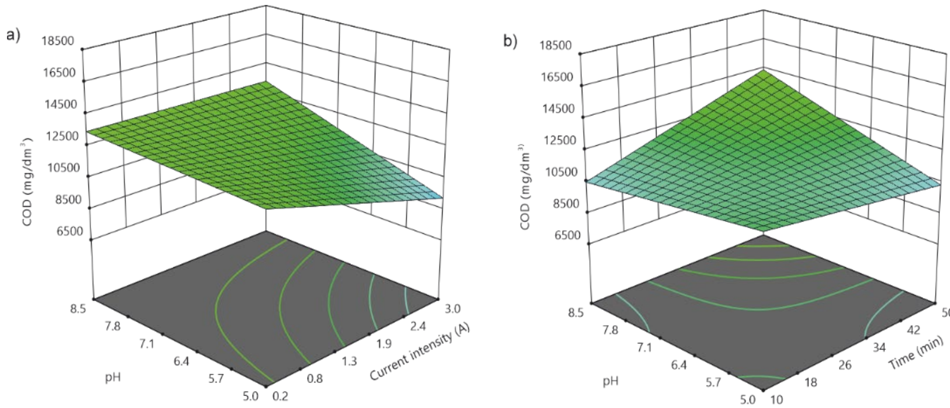


Fig. 3. COD in primary sludge: a) electrooxidation treatment as a function of pH and current intensity, b) sonication treatment (40 kHz, 80 W) as a function of pH and time

According to Rahmani et al. [26], hydroxyl radicals in the medium react with the steel electrode, preventing them from efficiently oxidizing organic materials. Another possible factor that decreased efficiency during electrooxidation is the presence of solids in the medium, since Chae et al. [27] demonstrated that removing solids before electrooxidation enhances the reactions involved, resulting in greater oxidation of organic matter, measured as COD, and ammonium during the electrooxidation treatment of pig manure. For the COD of the treatments by sonication, the statistically significant factors were time ($p = 0.012$), temperature ($p < 0.0001$), and pH ($p = 0.005$), as well as the interaction of the 3 factors ($p < 0.0001$) and the interaction of time–pH ($p < 0.0001$). According to the above, the lowest COD values obtained by applying sonication at pH 5 for 10 min (Fig. 3b) were 8498.33 mg/dm^3, representing a 32.5% reduction compared to the sludge without sonication.

Flores-Miranda et al. [28] reported that longer sonication times are more effective in degrading cell membranes, disaggregating conglomerates, and transporting intracellular material to the surrounding liquid. Shorter sonication times result mainly in the disintegration of flocs. Longer sonication times supply greater specific energy to the system, which results in greater solubilisation of the COD. Also, increasing the sonication time reduces the particle size [29]. Zhang et al. [30] suggest that sludge lysis is deeper in the initial period of sonication, which may explain why low COD values are obtained after 10 min of treatment.

Table 4

Treatments to reduce coliforms in primary sludge

Process	Conditions	Decrease		
		Total coliforms (log MPN/cm^3)	Fecal coliforms (log MPN/cm^3)	COD [%]
Electrooxidation	3 A, pH 5, 50 min	2.45	2.58	58.59
Sonication	6 °C, pH 5, 10 min	2.99	3.82	Increase of 21.43

Since Mexican regulations establish that sludge and biosolids must contain less than 1000 MPN/cm^3 of fecal coliforms, the conditions in which this parameter was lower were selected, which correspond to those conditions established in Table 4. The increase in COD values in the present study could be due to the solubilization of extracellular polymeric substances, which are high molecular weight polymers as proteins and carbohydrates, and the intracellular substances of microbial cells as enzymes and DNA [10]. Additionally, Zielewicz et al. [31] claim that sonication has two effects: a first stage in which the sludge flocs are disaggregated (dispersed) and the microbial cells that are attached to the solids are released; and a second stage in which the exposed cell walls are broken. This second step is referred to as cell lysis. Therefore, the results obtained suggest that the energy applied was sufficient to disaggregate the sludge flocs and a subsequent lysis, which is reflected as an increase in the COD values.

Although electrooxidation and sonication were effective for pathogen removal, this study was limited to the lab scale. Therefore, further testing is needed to detect variations in the results when using a pilot scale. It is also necessary to develop the necessary equipment to carry out the processes in larger volumes of sewage sludge, taking into account energy consumption and other important operating variables for the system.

4. CONCLUSIONS

Sonication reduces the amount of fecal coliforms that are present in primary sludge by 31%, at 6 °C and pH 5 applied for 10 min. On the other hand, for the treatment with electrooxidation, the amount of fecal coliforms was only reduced by 16% under conditions 3 A and pH 5 for 50 min. Although the concentrations of fecal coliforms and the chemical oxygen demand in the primary sludge decreased, these parameters continue to exceed the maximum permissible limits established in Mexican regulations (< 1 000 MPN/cm^3) for primary sludge treated by sonication to be considered as stabilised sludge. Consequently, it is recommended to use additional treatments to comply with regulations.

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