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Original Research

Solubilization of Dairy Waste-Activated Sludge by Ultrasonic Wave Irradiation

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Abstract

As one of the new techniques in the field of sludge treatment, ultrasonic wave irradiation could be employed to release nutrients from the solids to the soluble phase, which can subsequently be degraded by microorganisms in biological treatment. In this paper the Tehran Pegah Dairy Complex was selected to evaluate the effect of ultrasonic wave irradiation on soluble chemical oxygen demand (SCOD), nitrogen content, phosphors content, and also the causes of rising temperature of waste-activated sludge. Experimental investigation was carried out at 60, 120, 240, and 480 s contact times, and at output powers of 100, 300, and 500 W with 20 kHz operational frequency. Results revealed that the highest temperature was found around 12ºC at 500W output power and 480s contact time. Hydrolysis of chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN), and total phosphorus (TP) also showed increasing trends at all different contact times. At 480 s contact time, the maximum of TP and TKN release was about 116% and 170%, which was observed at 500W and 100W. The maximum release of COD was found around 640%, which was observed at 300W output power.

Keywords: sludge treatment, ultrasonic wave irradiation, release nutrients, dairy wastewater, waste-activated sludge, hydrolysis

Introduction

The purification of wastewater produces large amounts of sludge, estimated at 5% to 25% of the total volume of the treated effluent [1]. Compared to other industries, the dairy industries generate large amounts of wastewater [2]. As the biggest Iranian dairy manufacturer, Tehran Pegah Dairy Complex generates around 3,500 cubic meters of wastewater. The activated sludge process is the most widely used biological wastewater treatment. During aerobic biological treatment, (such as the activated sludge process) only about 35% of the organics are actually mineralized into carbon dioxide and water by microbes [3]. One of the most serious challenges in biological wastewater treatment has proven to be the production of waste-activated sludge. Waste-activated sludge needs additional processing for disposal, but the most challenging aspect appears to be the digestion difficulty of waste-activated sludge due to rate-limiting cell lysis [4]. This is because the cell wall and membrane of prokaryotic organisms are composed of complex organic materials such as peptidoglycan, teichoic acid, and complex polysaccharides, which are recalcitrant to biodegradation [5]. The low digestibility, therefore, requires a long retention time in the range of 30-60 days during biological treatment [6]. Waste-activated sludge containing large amounts of nutrients, such as nitrogen and phosphors, could be used as a nutrient balance in biological treatments. When acoustic energy is supplied to a liquid, gas bubbles form, which is called cavitation [7-10].

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Cavitation will result in:

(a) generating chemical reactions due to a locally high temperature (~4500ºC) and high pressure (~500 bar)
(b) extreme shear forces in the liquid and thereby mechanically attacking components
(c) the formation of highly reactive radicals ($H^{\cdot}$ and $OH^{\cdot}$), which can further facilitate chemical reactions for destroying organic contaminants
(d) the additional destruction of specific compounds [9, 11-13]. Cavitation has a major role to play in sludge solubilization.

In wastewater treatment plants anaerobic digestion is generally applied to a mixture of primary and secondary sludge. The anaerobic digestion process is achieved at different stages: hydrolysis, acidogenesis, and methanogenesis (the rate limiting stage is the hydrolysis) [14]. While entering the "soluble state," the substrates are more readily available for the microorganisms, which play an important role in waste water treatment plants. It is worth noting that the microorganisms in biological treatment can only break down the substrates in soluble state. Furthermore, the number of microorganisms that could generate extra cellular enzymes for hydrolyses of the substrate is limited. Therefore, hydrolysis is a rate-limiting step in biological treatment [15]. In order to improve hydrolysis and anaerobic digestion performance, several pretreatments should be taken into account: mechanical, thermal, and chemical or biological. These pretreatments cause the lyses or disintegration of sludge cells [16, 17]. According to Wang et al. [2], ultrasonic lyses is the most interesting process for methane generation enhancement. Retention time in the digester can also be reduced [18], which allows for more compact plants.

Bougrier and other authors [19-21] suggest that the COD release needs to be correlated with specific energy input defined as the energy input per unit of sludge (as TS). The following equation (1) was accordingly used to calculate specific energy.

$$E_{SPE} = \frac{P \times T}{V \times TS} \quad (1)$$

...where E_{SPE} is the specific energy input in kWs/kg TS (kJ/kg TS) [L2T-2], P the ultrasonic power in kW [ML2T-3], T the ultrasonic duration in seconds [T], V the volume of sonificated sludge in liters [L3], and TS the total solids concentration in kg/L [ML-3].

Methodology

Research Methods

The precipitated waste, activated sludge, or return sludge was collected from Tehran Pegah Dairy Complex on a sample basis. The sludge sample was sorted at 4ºC storage room to prevent biodegradation and was transferred to the lab to apply ultrasonic waves. Samples were analyzed immediately for TKN, TP, COD, SCOD, temperature and soluble part of TKN, and TP. TP, COD, and SCOD were measured using a Spectro-photometer model DR/2800, HACH. All the analyses were carried out based on standard methods [22].

Table 1. Specifications of ultrasonic equipment.

Cientz-Iid Sonificator System	
Maximum energy output (W)	950 W
Duty ratio	0.1-99.9%
Amplitude pole	Φ6
Electricity supply	220 V, 50 Hz.
Frequency (kHz)	20
Dimensions of soundbox proof	220×400×290 mm
Cable	UNF 1/2 – in.-20
Working time	Adjustable (1 s-99h, 99min)

Ultrasonic Equipment

The waste-activated sludge samples were sonificated through the use of a Scientz-IID system (manufactured by Ningbo Scientz Biotechnology Co. Ltd.). The ultrasound unit consists of a converter (or transducer), booster, and a sonotrode. A converter basically converts electrical energy into ultrasound energy (or vibration). The booster is a mechanical amplifier that promotes high amplitude generated by the converter. The sonotrode is a specifically designed instrument that transfers the ultrasonic energy to the sludge. The components of sonotrode are shown in Fig. 1. The full specification of the apparatus is presented in Table 1. The operational frequency and the maximal energy output were 20 kHz and 950W, respectively. Fig. 2 shows the ultrasonic equipment with different accessories.

Operational Conditions of Sonification

The most important factor in the cost optimization of the ultrasonic wave irradiation process in operating condi-

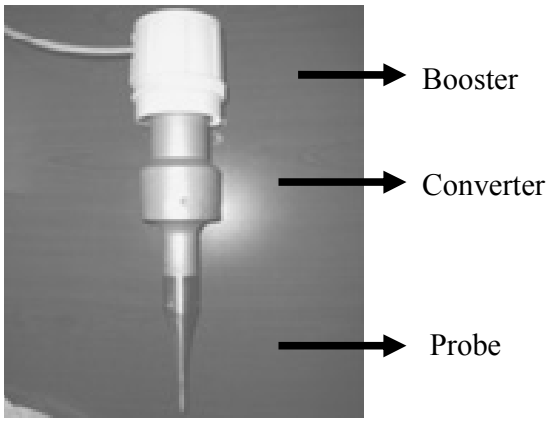

Fig. 1. Component of sonotrode.

tions will be a comparison of the output parameters of the ultrasonic system with sludge characteristics. Therefore, different contact times (60, 120, 240, and 480s) were adopted for this investigation and the ultrasonic energy outputs were set at 100, 300, and 500W. The investigation was carried out with a fixed 300 ml volume of dairy waste activated sludge and at ambient temperature in all sonification operations. The characteristics of samples used for sonification were stable throughout the experiments. Ultrasonic irradiation was applied to sludge using a sonotrode, with 6mm diameter immersed at a depth of 20 mm.

Waste Activated Sludge Container for Ultrasonic System

Three acoustic streaming regions, formed in sludge during sonification, were affected by container shape, particularly the largest region, called Eckart streaming – a large stream that terminates to better and more distribution of ultrasonic energy and an accelerated heating dissipation [23, 24] with a circulating current. This region was defined by the shape of the beaker container and size and length of the ultrasonic wave in the sludge samples. Therefore, in order to generate large Eckart streaming and better distribution of ultrasonic energy, a 300 ml sample of the sludge was placed in a glass beaker (700 ml volume and 7.2 cm internal diameter).

Results and Discussion

The application of ultrasounds to sludge processing appears to be intriguing because of the possibility of obtaining significant effects of releasing nutrients into soluble form without the necessity to apply additional components (e.g. chemical agents, simplicity of the construction, and operation of reactors as well as air tightening of the installation) [25-30]. Microorganisms in biological treatment are able to degrade only the soluble form of substrates. When substrates are in insoluble form, microorganisms are forced to hydrolyze the substrate, which turns out to be a limiting step in biological treatment [15].

In modern wastewater treatment plants this process thus seems to be of great significance. The characteristics of waste-activated sludge, collected from the dairy complex,

Fig. 2. The vessel in ultrasonic system.

Table 2. Characteristics of waste activated sludge from Tehran Pegah Dairy Complex.

Parameter	Rage of data	Mean	Standard Deviation
COD (mg/l)	19,580-21,211	20,297	833.15
SCOD (mg/l)	784-820	800	18.23
TKN (mg/l)	1,095-1,170	1,130	37.74
Soluble TKN (mg/l)	334-379	360	23.30
TP (mg/l)	253-276.5	265.5	11.82
Soluble TP (mg/l)	16.5-22	19.6	2.84
Temperature (ºC)	18.5-19.5	19	0.5

Table 3. Increased dairy waste-activated sludge temperature in different sonification conditions.

Sonification times (seconds)	Temperature increase in sludge samples (ºC)		
	Power 100 W	Power 300 W	Power 500 W
0 (Control)	-	-	-
60	2	2	2
120	3	3	3
240	4	5	5
480	9	11	12

have mean soluble COD, TKN, and TP of about 800, 19.6, and 360 mg/l, respectively (Table 2).

Sludge disposal makes up 60% of capital cost and more than 50% of operational cost in wastewater treatment plants [31, 32]. The acoustic streaming is the occurred mechanism when the sludge is sonificated. The main benefit of streaming in sludge processing is mixing, which facilitates uniform distribution of ultrasound energy within the sludge mass and convection of the liquid and distribution of any heating that occurs [33].

Effects of Ultrasonic Wave Irradiation on Temperature

One of the most important effects of applying ultrasonic wave irradiation into sludge samples could be a gradual rise in temperature because of the violent collapse of cavitation bubbles creating an extremely high local temperature (~4,500ºC). In this study, the temperature in all sludge samples was monitored before and after applying ultrasonic waves. In all sonification conditions a rise in temperature in sludge samples was observed as measured simultaneously by a Scientz-IID temperature sensor system. Table 3 illustrates temperature rising in different sonification conditions.

Maximum rise in temperature was measured around 12ºC, at 500 W output power, and 480s contact time. In all

different powers, temperature rise demonstrates an increasing trend.

With a longer contact time of 480s a rise in temperature is obtained, which is bigger than any other contact time. It thus indicates that contact time plays a significant role in temperature increase.

Minimum rise in temperature was measured around 2ºC at 60s contact time, and output powers of 100, 300, and 500 W. Table 3 compares the percentage of temperature rising in dairy waste-activated sludge at different sonification times and powers. Monitoring the temperature seems to be important since it improves solubilization. While the thermophillic biological process is employed in sludge treatment, such temperature rising could enhance the efficiency of treatment. Moreover, in the case of using a mesophillic biological process in sludge treatment, high temperature should be avoided in such units.

Effects of Ultrasonic Wave on Chemical Component Solubilization

According to the characteristics of the waste-activated sludge (Table 2), more than 96% of chemical oxygen demand (COD) is insoluble. In fact, all released organic matter is lumped together and measured as an increase in SCOD. Fig. 3 illustrates the percentage of SCOD level of increase at different contact times (60, 120, 240, and 480s).

The maximum release of SCOD was found around 640% observed at 300 W output power and the contact time of 480s. In all, different powers of 100, 300, and 500W, with increasing sonification times, hydrolyses of COD shows increasing trends. The expected value at 480s contact time and 500W output power is relatively lower compared to 240s contact time. This could be due to consuming some part of COD release because of the dominant free active radicals during sonification with 500W of power. But at the power of 300W, compared to the power of 100W, increasing COD level is observed to have higher trends, expectedly at 240s contact time. This could be related to the nature of organic compounds in dairy waste activated sludge that breaks down in this power and turns into soluble form. Regarding the saving of energy and optimizing ultrasonic energy, the best power for 60 and 480s appears to be 300W, and any further increase in power will have the opposite effect. At 120s contact time the same increasing trends were observed, but at 240s contact time better solublization efficiency could be achieved at 500W output power. According to Khanal et al. [3] better disintegration of biological and non-biological solids was achieved at longer sonification times.

Effect of Ultrasonic Waves on TKN

The presence of nitrogen and phosphors are essential for the growth of microorganisms known as nutrients or biostimulants. Trace quantities of other elements, such as irons, are also needed for biological growth. Nitrogen and phosphorus are, however, the most important nutrients. As nitrogen is an integral part of the synthesis of protein, nitrogen data will be required to evaluate the treatability of

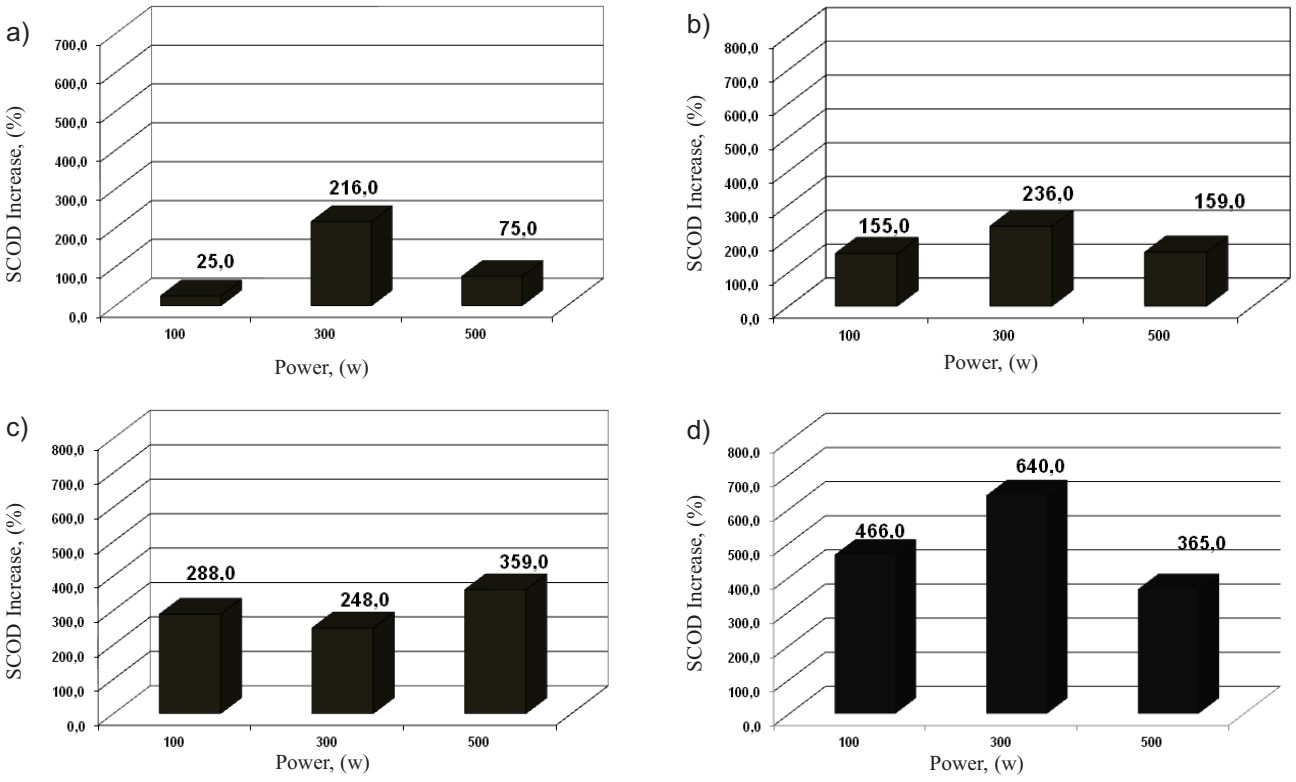

Fig. 3. Percentage of released COD from dairy waste-activated sludge at different powers versus different contact times at (a) 60s, (b) 120s, (c) 240s, and (d) 480s.

wastewater by activated sludge process. An insufficient amount of nitrogen may necessitate nitrogen being added to make wastewater treatable. Total Kjeldahl nitrogen (TKN) includes organic nitrogen, ammonia, and ammonium. The organic fraction consists of a complex mixture of compounds, including amino acids, amino sugars, and proteins (e.g. polymers of amino acids) [6]. On the other hand, balancing nitrogen in biological units is important for achieving high rates of removal. Waste-activated sludge contains a large amount of nitrogenic components, but the major part may be considered, as in insoluble form. According to the characteristics of the waste-activated sludge (Table 2), more than 68% of TKN is in insoluble form. Microorganisms in biological treatment are able to degrade only in the soluble form of substrates. If substrates are in insoluble form, microorganisms are forced to hydrolyze the substrates, which is the limiting step in biological treatment [15].

Therefore, solubilizing the waste-activated sludge and consequently supplying nitrogen components to the biological treatments units would accelerate the treatment process. Moreover, there could be a significant cut in operational cost in sludge disposal. Fig. 4 illustrates the percentage of increasing soluble TKN levels at different contact times of 60, 120, 240, and 480s.

The maximum value of TKN release was found with at 100W power and contact time of 480s, which was around 171%. With increasing sonofication time in all powers, behavior of TKN hydrolysis was apparently developing. Although at 300W power achieved values are relatively lower in comparison to 100W. This could be due to the oxidation of some part of released TKN. This phenomenon may occur due to the domination of free active radicals during sonification at 500W power compared to 300W, TKN level observed to have higher increasing trends, which could be related to the nature of nitrogen compounds in dairy waste-activated sludge that breaks down in this power and goes into the soluble phase.

According to Fig. 4, at 240s contact time the best power appears to be 500W. However, at 480s contact time and output power of 100W shows the highest solubilized TKN efficiency.

Effects of Ultrasonic Wave on Release TP

According to the characteristics of the waste-activated sludge, more than 92% of TP is in insoluble form. Phosphor is one of the initial substrates for the initiation of microbial activity in biological treatment. Furthermore, the usual forms of phosphor found in waste-activated sludge include orthophosphate, polyphosphate, and organic phosphate. The orthophosphate (e.g. PO_4^{-3}, HPO_4^{-3}, $H_2PO_4^{-}$) is available in biological metabolism without further breakdown. The polyphosphate includes molecules with two or more phosphor atoms, oxygen atoms, and in some cases hydrogen atoms combined in a complex molecule. Polyphosphates

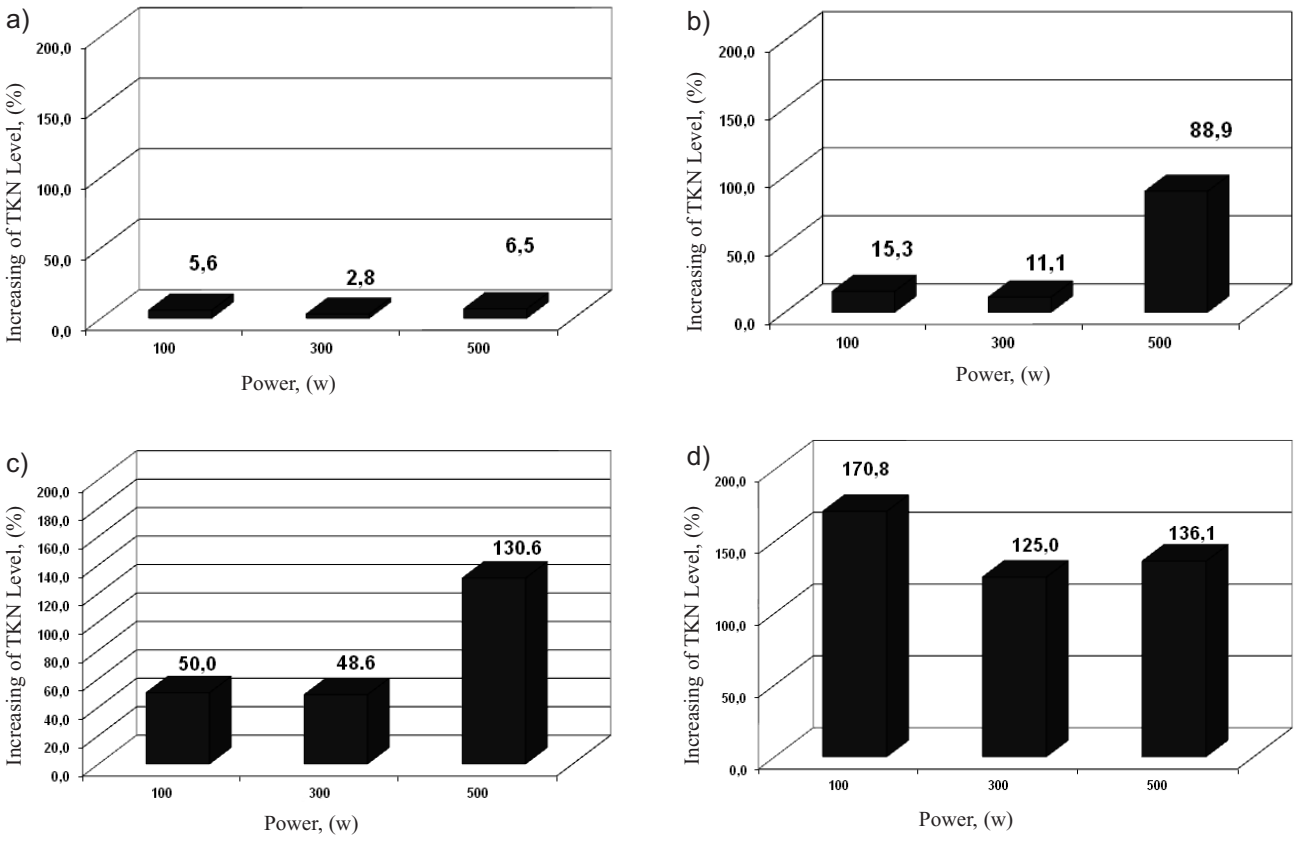

Fig. 4. Percentage of released TKN from waste-activated dairy sludge at different powers versus different contact times at (a) 60s, (b) 120s, (c) 240s, and (d) 480s.

undergo hydrolysis in aqueous solution and revert to the orthophosphate form; however, this hydrolysis is quite slow [6]. Tehran Pegah Dairy Complex has problems in balancing phosphor in biological treatment. As shown in the waste activated sludge characteristics (Table 2), only about 8% of TP exists in soluble form. Therefore, it is important to release TP from sludge in order to balance the N/P ratio for achieving an acceptable efficiency in subsequent treatment. Fig. 5 illustrates the percentage of increase in TP level, at different sonification contact times (60, 120, 240, and 480s respectively) with different applied powers.

Maximum release TP is observed at 500W and 480s contact time. At all different powers, releasing TP shows an increasing trend. All the obtained values (at 500W of power compared to 300) shows a less increasing rate apart from the contact time of 480s. This could be related to the activities of free active radicals and the consumption of part of the released TP at 500W during sonification. However, at the contact time of 480s, with the rise in power more TP is obtained. This could be related to the nature of phosphoric compounds in waste-activated dairy sludge, which breaks down at this power (500 W) and goes into soluble form. Therefore, if the solubility of phosphoric compounds is taken into account, it appears to be better to use low power (e.g 100 and 300 W) and long sonification times (e.g. 120 and 480s). Whenever a portion of TP is changed into soluble state, the application of the highest power (500 W) is inductive in this transformation. Hence contact plays a major role, since some phosphoric compounds may break dawn at a longer sonification period.

Conclusions

- According to the characteristics of waste-activated dairy sludge in TPDC, we used the rough estimate of the average values of 800, 360, and 19 mg/l for COD, TKN, and TP (existing in soluble state), respectively.
- Monitoring temperature is important during sonification, since it affects solubility. The maximum rise of sludge temperature measured was around 150% observed at the highest output power of 500W and longest contact time of 480s. At all different powers, the temperature rise shows increasing trends. Overall, the sludge temperature rise can be noticeable at all powers.
- Hydrolysis of chemical components shows increasing trends at all different contact times. Obtained values for released COD show better solubility efficiency at 300W output power and the maximum value of released COD around 640%. Nitrogen and phosphor are two important substrates in biological growth that could be supplied from waste-activated sludge to biological units. The maximum release of TP was about 116%, which was observed at the 500W output power and 480s contact time. Obtained values of this power at other contact times (e.g. 60, 120 and, 240s) were relatively lower compared to lower output power (e.g. 100 and 300 W). Therefore, the phosphoric component has shown different breakdown behaviors at 500W output power, practically after 240s contact time: a major part of TP is released and transformed to soluble form. The maximum of TKN release was about 170%, which was

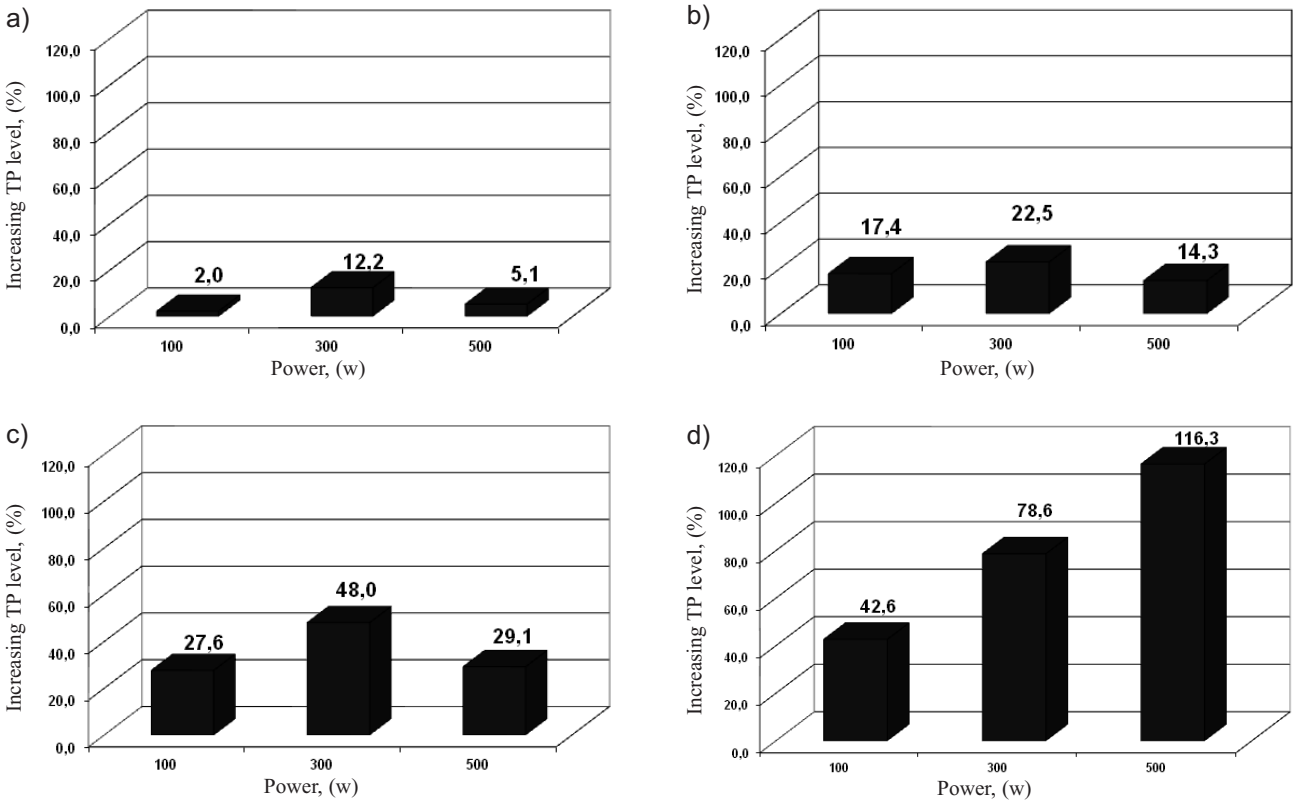

Fig. 5. Released TP from waste-activated dairy sludge at different powers versus different contact times at (a) 60s, (b) 120s, (c) 240s, and (d) 480s.

observed at the lowest output power (e.g. 100W) and longest contact time (e.g. 480s). The obtained value of released TKN at 500W output power was much bigger than at the lower output power (e.g. 100 and 300W), apart from the contact time of 480s. This could be linked to consuming some parts of released TKN, because of the free active radicals' domination during sonification.

Therefore, Ultrasonic wave irradiation causes releasing nutrients from the solid to the soluble phase, which can be degraded easily by microorganism in biological treatment.

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