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Original Research

Constant Magnetic Field Influence on Stabilization of Excess Sludge with Fenton's Reagent

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Abstract

The aim of our study was to determine the impact of constant magnetic field (CMF) on the effectiveness of stabilization of the excess sludge by advanced oxidation process with Fenton's reagent. In presented study raw excess sludge from a dairy wastewater treatment plant was used. The investigations were conducted in three different technological systems in laboratory-scale stands. In the first stage the influence of chemical reagents (Fe^{2+}/H_2O_2, Fe^{3+}/H_2O_2) on the parameters of sewage sludge was observed. In the second stage the importance of CMF on the final technological result in the system with static crossing on magnetic field (scCMF) and in the system with cyclical crossing on magnetic field (ccCMF) was revealed. In the third stage of the experiment the effectiveness of the system with both chemical reagents and physical elements was tested. Application of the constant magnetic field let us obtain COD reduction in supernatant and volatile solids in dry mass of sewage sludge. The importance of the physical factor was shown as low doses of chemical reagents. Similar results were achieved independently of the fact if it was tested as the improving element of advanced oxidation with scCMF or with ccCMF.

Keywords: Fenton reaction, constant magnetic field, sewage sludge, stabilization, advanced oxidation

Introduction

Operation of a wastewater treatment plant is closely related to the need for proper sludge management. Sludge produced in the wastewater treatment process must be processed and afterwards reused or disposed of. Despite considerable progress regarding the methods and techniques of sludge processing, the problem of universal, economically substantiated technology of effective neutralization remains open.

An alternative to presently practiced methods may become innovative techniques used with much success at water and wastewater treatment. The methods, based on advanced oxidation process (AOP) of contaminants, can be mentioned [1-4]. One of the methods of advanced oxidation is Fenton reaction, the process that occurs while using hydrogen peroxide (H_2O_2) with the most frequently used catalyst: ferrous or ferric ions. The mechanism of the reaction leads to hydrogen peroxide breaking down to water and oxygen in the presence of Fe^{2+} or Fe^{3+} ions. Due to the reaction free radicals ($OH^{\bullet}$), a high oxidizing potential of 2.8 V is generated [5-7].

Effectiveness of the Fenton reaction and obtained results revealed that this method can be widely applied as a competitive technique for sewage sludge processing [8-10]. Literature data and the results achieved by the authors in the preliminary study created a solid base for further research of the optimization of the process, but

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factors improving the efficiency of this technology are still unknown. The priority remains care for economic use of chemical reagents and application of the elements promoting their limitation without influence on the final technological effects [11-13].

It was assumed that the factor fulfilling the above condition may be constant magnetic field (CMF). Purposefulness of the application of constant magnetic field has been confirmed in cases of water and wastewater treatment, but also in cases of sewage sludge conditioning [13, 14]. Using this physical factor as the element improving the advanced oxidation process is especially relevant for sewage sludge processing. The characteristics and properties of this medium limit the possibility of using other factors to improve the process, e.g. UV radiation. Besides, sewage sludge properties can be significantly changed by constant magnetic field.

The presented study aims at determining the impact of SMP on the effectiveness of advances in oxidation process with Fenton's reagent used for at sewage sludge stabilization.

Experimental Procedures

In the presented experiment excess sewage sludge from a dairy wastewater treatment plant was used. The properties of the excess sludge are shown in Table 1. The investigations were conducted in three different technological systems in the laboratory-scale stands (Fig. 1). All research stages and physicochemical analysis were performed at ambient temperature of about 20°C.

The first stage of the experiment involved activities for determining the influence of Fenton's reagent on changes of the parameters and properties of sewage sludge. The stage was divided on two series varying of the catalyst of Fenton reaction. In the first series classical Fenton reaction (Fe^{2+}/H_2O_2) was tested, in the second series Fe^{3+}/H_2O_2 was applied into sewage sludge mass. Doses of the reacting substances applied in the technological system are shown in Table 2.

Ferrous ions Fe^{2+}as $FeSO_4 \cdot 6H_2O$ and ferric ions Fe^{3+} as 40% solution of $Fe_2(SO_4)_3$, and H_2O_2 as 30% solution of perhydrol were dosed into the system to initiate an advanced oxidation process. The first stage of the experiment was carried out in laboratory reactor at a volume of 1.0 dm^3, equipped with magnetic stirrer. At the beginning of the experimental cycle sewage sludge was added into the reactor, and afterwards chemical reagents were dosed. Firstly Fe^{2+} or Fe^{3+} ions, and after 10 minutes H_2O_2 were introduced into the mass of sewage sludge. Within 20

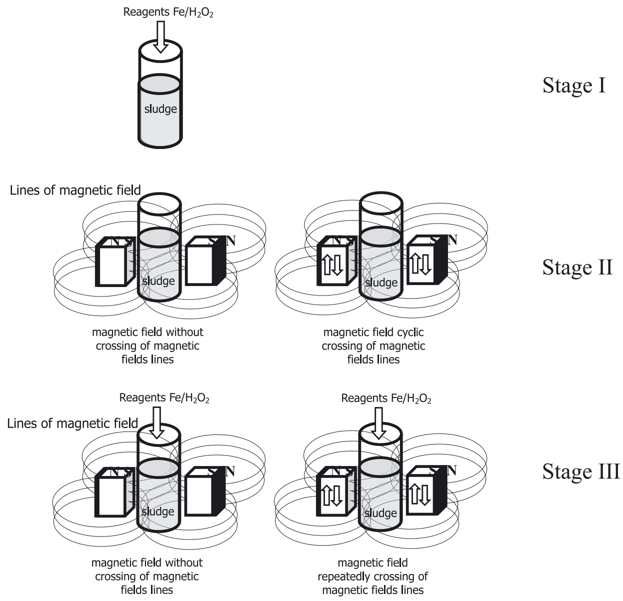

Fig. 1. Scheme of the experimental stand.

Table 1. Characteristics of the sewage sludge used in the experiment.

Parameter	Unit	min.	max.	mean
Hydration	[%]	97.62	96.95	97.28
Filtration resistivity	[$m \cdot kg^{-1}$]	$3.214 \cdot 10^5$	$4.192 \cdot 10^5$	$3.703 \cdot 10^5$
Capillary Suction Time CST	[s]	84.5	144	114
Dry mass	[$g \cdot L^{-1}$]	23.840	24.510	24.175
Mineral fraction	[$g \cdot L^{-1}$]	5.760	6.990	6.375
Volatile fraction	[$g \cdot L^{-1}$]	17.710	18.520	18.115
COD of filtrate	[mg $O_2 \cdot L^{-1}$]	562.3	701.1	631.7
Reaction	[pH]	6.98	7.40	7.19
Clostridium perfringens	[CFU·g d.m.$^{-1}$]	0.0	$10 \cdot 10^0$	$5.0 \cdot 10^0$
Coliform bacteria	[MPN·g d.m.$^{-1}$]	$2.7 \cdot 10^4$	$0.5 \cdot 10^4$	$1.6 \cdot 10^4$
Fecal coliforms	[MPN·g d.m.$^{-1}$]	$3.3 \cdot 10^3$	$3.9 \cdot 10^3$	$3.6 \cdot 10^3$

minutes of the experiment sewage sludge was mixed at 100 rpm in order to distribute all the reacting substances evenly. After this time, sludge with the reagents was left without stirring to finish the reaction.

In the second stage of the study the impact of constant magnetic field as the element influencing the properties of sewage sludge used in the experiment was determined. Depending on series of this stage the investigations were performed in the system with static crossing on magnetic field (scCMF) or in the system with cyclical crossing on magnetic field (ccCMF). In both variants constant magnetic field at the intensity of 0.6 T was generated due to magnetic liquid activator. It is built of two parts, making a cylindrical body (ring) with a definite diameter, situated directly inside the reactor. In order to improve the scope of the interaction of physical factor, the reactor involved two rings generating a constant magnetic field.

Table. 2. Chemical reagents doses used in experiment.

Doses	Fe^{2+} [$g \cdot L^{-1}$]	Fe^{3+} [$g \cdot L^{-1}$]	H_2O_2 [$g \cdot L^{-1}$]
1	0.25	0.25	1.00
2	0.50	0.50	2.00
3	0.75	0.75	3.00
4	1.00	1.00	4.00
5	1.50	1.50	6.00
6	2.00	2.00	8.00

In the first series of the experiment scCMF was generated. Activated sludge was supplied into the unmovable reactor placed in the scope of direct interaction of physical factor.

In the second series ccCMF was generated. In this part of the experiment a special device was used that let a cylinder with mass of the sludge to be cyclically introduced in the scope of interaction of constant magnetic field. It indicates that stabilized sludge was not still exposed on the interaction of physical factor during the retention time of the experiment. Sludge was exposed on magnetic field with a frequency of 20 times per min.

Final stage of the experiment included physical factor and advanced oxidation process at sewage sludge processing. To the effect, sewage sludge was introduced in the scope of the direct interaction of constant magnetic field in systems with scCMF and ccCMF. Afterwards, chemical reagents were added, and the doses were the same as in the first stage of the experiment.

Organic substances were expressed as COD in the supernatant after vacuum filtration [PN-74/C-04578.03] and volatile substances in dry mass of sewage sludge [PN-75/C-04616.01]. Retention time of the sludge in all stages of the experiment was 24 h.

The authors controlled pH of the reaction. They did not imply into sewage sludge any acidifying substances and did not correct pH of the sludge before the addition of Fenton's reagent. pH that was kept for tested sewage sludge was chosen because of applicable, technological and economic aspects. Another reason for desisting from sewage acidification was the fact that that sewage sludge reaction was corrected through the addition of iron salts,

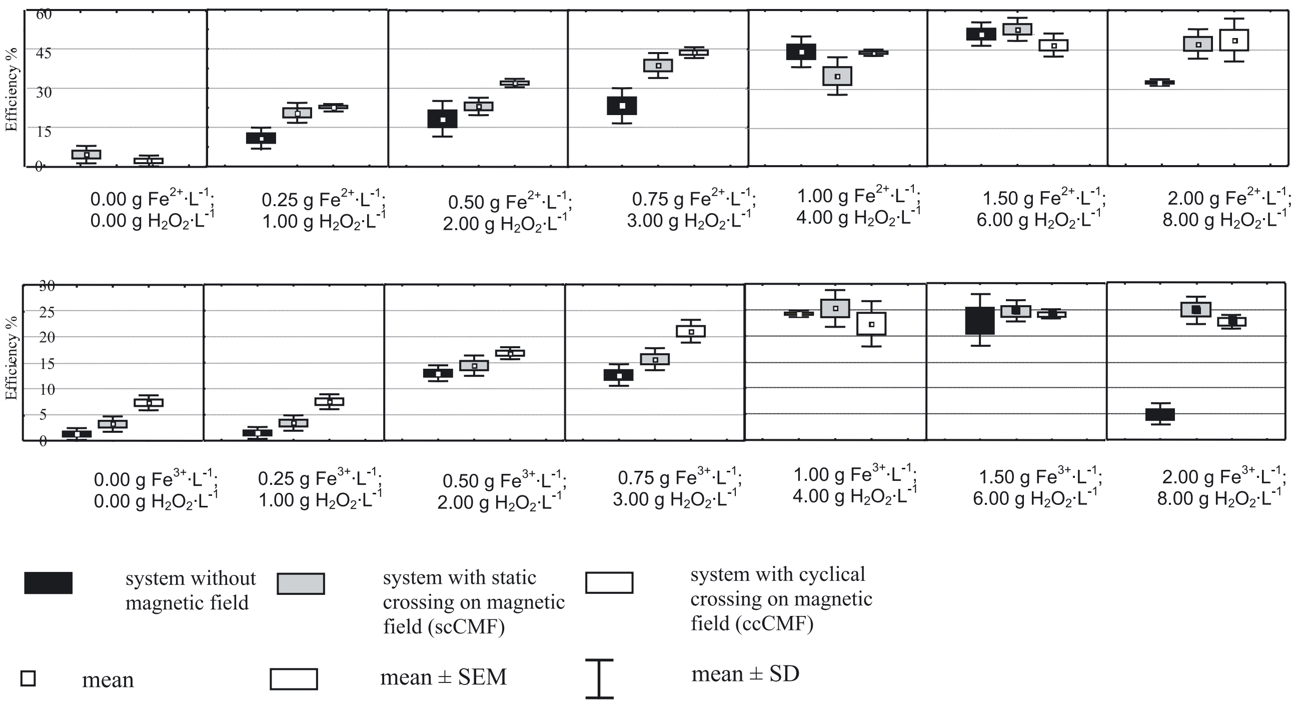

Fig. 2. Percentage efficiency of organic compounds (expressed as COD) degradation in the filtrate.

mostly $Fe_2(SO_4)_3$. Decrease in pH depended on the doses of iron salts.

Statistical analysis of the obtained results was done using variance analysis, at the assumed accuracy level ($p < 0.05$). Normal distribution was confirmed by Szapiro – Wilk test, and hypothesis concerning variance homogeneity inside the groups were verified on the basis of Leveney's test. Analysis of the differences between means from the particular groups was done using Tukey's test.

Results

Application of the lowest doses of Fenton's reagent (Fe^{2+}/H_2O_2) in the first stage of the experiment let us obtain the reduction of COD concentration in the supernatant after vacuum filtration in the range from 18.2% to 24.9%. Much more effective turned out variants with 1.0 g $Fe^{2+}\cdot L^{-1}$; 4.0 g $H_2O_2\cdot L^{-1}$ and 1.5 g $Fe^{2+}\cdot L^{-1}$; 6.0 g $H_2O_2\cdot L^{-1}$. The effectiveness of COD removal was 44.6% and 49.4%, respectively, and COD concentration in the supernatant after vacuum filtration, at the end of the cycle was on the level 390.8 mg $O_2\cdot L^{-1}$ and 353.7 mg $O_2\cdot L^{-1}$ (Fig. 2).

Improving impact of scCMF was shown at low doses of Fenton's reagent. Statistically significant differences, contrary to the first stage of the research, were observed when from 0.25 g $Fe^{2+}\cdot L^{-1}$, 1.0 g $H_2O_2\cdot L^{-1}$ to 0.75 g $Fe^{2+}\cdot L^{-1}$, 3.0 g $H_2O_2\cdot L^{-1}$ was applied into the technological systems. In these cases the effectiveness of COD removal from the filtrate ranged from 24.6% to 38.1% at the end of the cycle. The highest effectiveness of COD removal in the most effective variant was 51.5%. The result was obtained in the system with scCMF at high reagent dose – 1.50 g $Fe^{2+}\cdot L^{-1}$ and 6.0 g $H_2O_2\cdot L^{-1}$ (Fig. 2).

Similar results of organic matter reduction in the supernatant were achieved when dairy sewage sludge was cyclically exposed on magnetic field in the system with Fenton's reagent. The efficiency of COD removal ranging from 26.0% to 47.9% at the end of the cycle depending on the dose of chemical reagents. The most effective appears to be the two highest doses of Fenton's reagent. In case when 1.5 g $Fe^{2+}\cdot L^{-1}$ and 6.0 g $H_2O_2\cdot L^{-1}$ were applied into the mass of sewage sludge COD removal efficiency after 24 h was 47.3%, and in variant with 2.0 g $Fe^{2+}\cdot L^{-1}$; 8.0 g $H_2O_2\cdot L^{-1}$ the effectiveness was 47.9%. It was proved that the application of the three lowest doses of Fenton's reagent in the system with ccCMF let obtain better technological results than in the first stage and in the system with scCMF (Fig. 2).

Introduction of Fe^{3+}ions as a catalyst of advanced oxidation process did not improve the technological effect of sewage sludge stabilization in contrast to the system with Fe^{2+}/H_2O_2 both in the first and third stage of the experiment. In the best case of the first stage of the experiment COD effluent after 24 h was 502.9 mg $O_2\cdot L^{-1}$, and the effectiveness of organics removal was at 24.3% (Fig. 2).

In stage II only impact of CMF on sludge was examined. The shown statistics depend ($p<0.05$) on changing of COD of the filtrate (Fig. 2). In series with scCMF system meanly was removed of 6.1% organic compounds in the end of the technological cycle. When sludge was cyclically moved into magnetic field (ccCMF) efficiency of

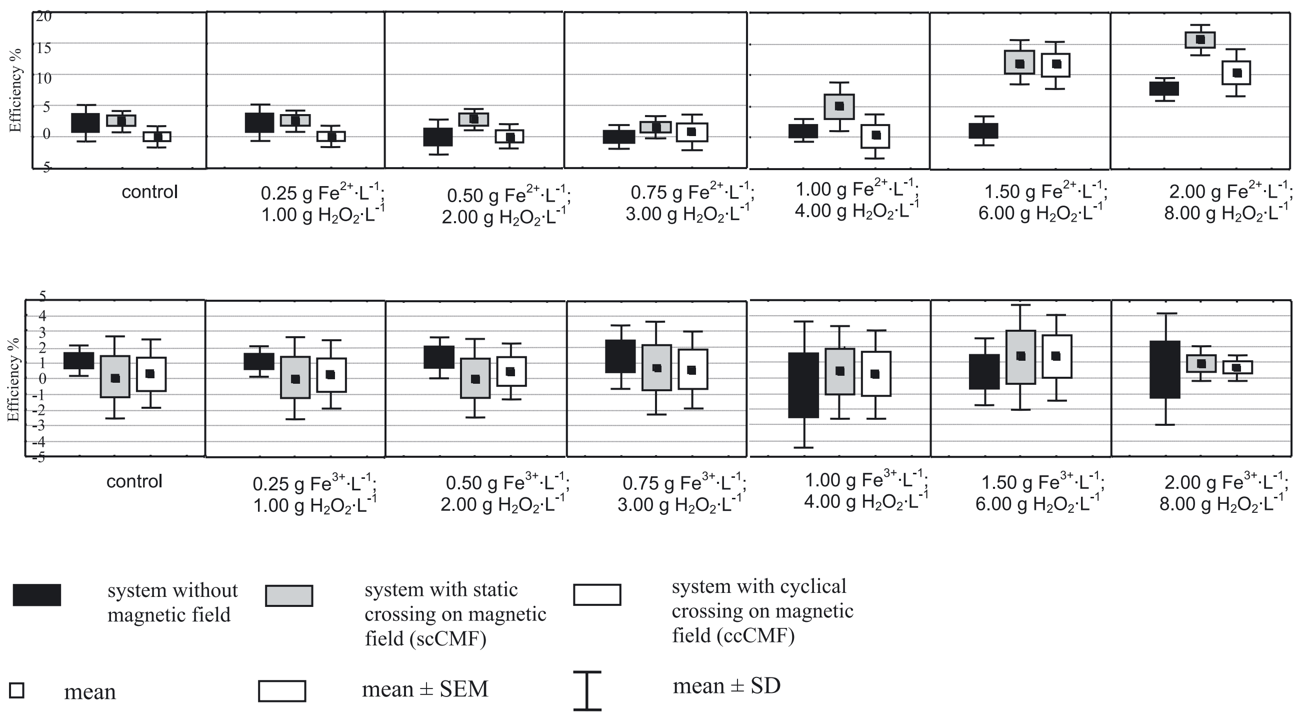

Fig. 3. Percentage efficiency of volatile fraction removed in analyzed sludge.

COD limitation was 3.1%. Use of magnetic field in both cases (ccCMF, scCMF) had no statistically significant impact on the value of volatile the fraction (Fig. 3).

The most effective variant of the third stage of the experiment after 24 h in the system with scCMF allowed us to obtain COD removal efficiency on the level of 25.3% and organic matter concentration was 486.6 mg $O_2 \cdot L^{-1}$. Application of Fe^{3+}/H_2O_2 and the system with ccCMF let achieve, in most cases, significantly higher final effect, in contrast to the first stage of the experiment. Statistical analysis proved that improving effect of the physical factor was shown in the range of doses from 0.25 g $Fe^{3+} \cdot L^{-1}$; 1.00 g $H_2O_2 \cdot L^{-1}$ to 0.75 g $Fe^{3+} \cdot L^{-1}$; 3.00 g $H_2O_2 \cdot L^{-1}$. In these cases the system with ccCMF was more effective in the range from 4.0% to 6.6%. Significant differences were observed when 2.00 g $Fe^{3+} \cdot L^{-1}$ and 8.00 g $H_2O_2 \cdot L^{-1}$ was applied into the mass of sewage sludge. Application of only chemical reagents at the conditioning and stabilization process resulted in 5.0% of COD removal effectiveness. The same dose of Fenton's reagent along with the system with constant magnetic field to improve organic matter removal to 24.8% in series I and to 22.4% in series II of the third stage of the experiment (Fig. 2).

Statistically significant removal of volatile substances was observed when dairy sewage sludge was treated by Fenton's reagent and was exposed on scCMF (Fig. 3). It was proved in cases of applications of chemical reagents from 1.0 g $Fe^{2+} \cdot L^{-1}$; 4.0 g $H_2O_2 \cdot L^{-1}$ to 2.0 g $Fe^{2+} \cdot L^{-1}$; 8.0 g $H_2O_2 \cdot L^{-1}$ into the mass of sludge. At the end of the technological cycle volatile matter concentration in analyzed samples ranged from 17.510 $g \cdot L^{-1}$ to 15.540 $g \cdot L^{-1}$. at the initial value in raw sludge – 18.115 $g \cdot L^{-1}$. Therefore, the effectiveness of volatile substances removal ranged from 4.9% to 15.6% (Fig. 3).

The lowest technological effect was observed in series II of the third stage of the experiment. Statistically significant difference, in contrast to the chemical stage, was revealed in variant with chemical reagents – 1.50 g $Fe^{2+} \cdot L^{-1}$ and 6.00 g $H_2O_2 \cdot L^{-1}$. Volatile matter concentration was 16.090 $g \cdot L^{-1}$.

In any technological variants with Fe^{3+} ions applying into dairy sewage sludge there was no significant result of the removal of volatile substances in tested mass of the sludge.

Reaction of sludge was close to neutral in range from pH 6.98 to pH 7.40 average pH 7.19 (Table 1, Fig. 4). Dozing of Fe^{3+}/H_2O_2 changed this parameter in the clearest way. Addition of this type of inorganic coagulant influenced the decrease of pH and was connected to the increased dose of the chemical reagent. In successive technological variant analyzed sludge reaction decrease in range from pH 6.91 to pH 3.43 in the first stage of experiment after one-day retention. When Fe^{2+}/H_2O_2 was used, reaction of wastewater sludge decreased from pH 6.98 to 4.72 in the end of research in first stage. In the third part of experiment value of this parameter was in range pH 7.13 to pH 4.97 in series I and pH 7.07 to 5.18 in series II (Fig. 4).

Discussion of Results

The increase in the effectiveness of organic substance removal in supernatant after vacuum filtration assured by the application of CMF into the system could be a result of several physical and chemical mechanisms. There is a high probability that constant magnetic field was the element influencing the processes of $OH^{\bullet}$ generation [14-16].

It was found that free radicals have one or more nonpaired electrons whose quantum number of spin is -1/2 or +1/2. During the reaction of two free radicals, unpaired electrons can have the same spin which is defined as a

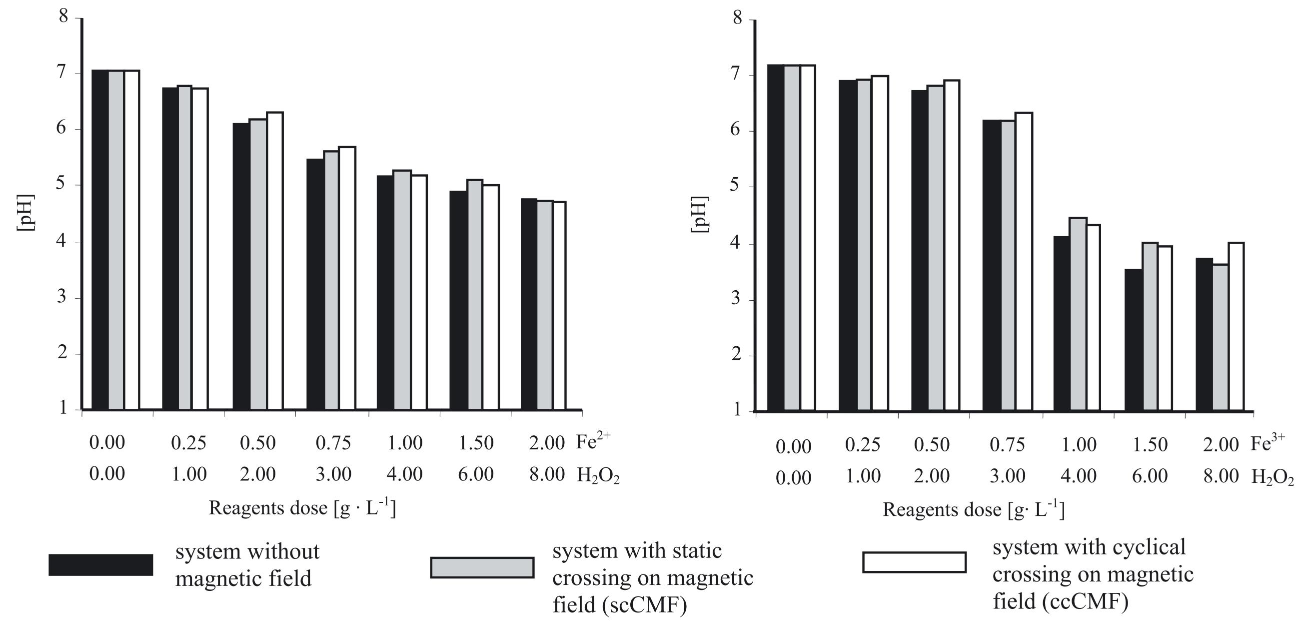

Fig. 4. Changes of pH value during the experiment.

"triplet configuration". In cases when the spin is opposing, it is defined as "singlet configuration". Free radicals possessing a triplet configuration do not create bonds. Through the intersystem crossing, the triplet configuration can be transformed to the singlet configuration, which makes the creation of the bonds between free radicals possible. Such a possibility was revealed for free radicals that posses unpaired electrons with opposing spin in direct contact. The CMF can cause a decrease in intersystem crossing efficiency which leads to a decrease in the amount of radicals that are transformed to singlet configuration with simultaneous maintenance on the same level or even increase in free radicals' total quantity. For these reasons, this physical factor is thought to be the element responsible for generating hydroxyl radicals $OH^{\bullet}$ [14, 15].

Positive integration of Fenton reaction and constant magnetic field are confirmed by the results of pretreatment of different kinds of wastewater. Advanced oxidation process improved by constant magnetic field resulted in about three-fold limitation of the amount of used reagents at the same effectiveness of pollutants' removal from dairy wastewater. The process was fast and the impact of constant magnetic field was shown after several minutes of exposure to wastewater. The experiment revealed that there is a possibility of application of lower doses of chemical reagents at the same effectiveness of wastewater treatment when CMF was introduced [14].

Evidently, higher COD removal in the supernatant after vacuum filtration of tested sludge, ensured by CMF implementation into the technological system, could result in improvement of coagulation and sedimentation, and organic matter precipitation to the sludge. It was more probably because in other experiments the application of a physical factor to technological system improved sewage sludge dewatering in contrary to application chemical reagents only. The impact of physical factors on improving pollutant coagulation and dewatering sewage sludge was shown in the literature references [17, 18].

Low efficiency of the oxidation of volatile matter in dry mass of sewage sludge can be explained by the fact that hydroxyl radicals $OH^{\bullet}$ as highly reactive and slowly mobile forms, firstly affected on the organic substances suspended in supernatant. High organics concentration in supernatant probably were the basis substrates of oxidation due to advanced oxidation process. Another reason that part of organic matter was removed from supernatant after vacuum filtration was precipitated due to coagulation of iron salts and deposit in dry mass of sludge. Fenton reaction involves two parallel processes, coagulation and oxidation, influencing changes of organic matter concentration, especially in the supernatant. Certainly the reduction of COD in supernatant after vacum filtration is partly the result of coagulation and sedimentation of organic matter that is deposited in dry mass of sludge. Presented in manuscript, correlations are proved by previous studies and papers concerning the influence of advanced oxidation on the ratio of the particular fractions of dry mass of sewage sludge [12, 19, 20].

Probably, longer hydraulic retention time in tanks and application of higher doses of chemical reagents let us obtain more effective final results. The authors was conscious that high operating cost directly resulted from high doses of chemical reagents that would disqualify proposed technology as expensive and economically unfounded. Carrying out the experiment would have educating aspect only, and it would not be a chance to put the proposed technology into practice.

The correlation was often presented by the literature date concerning the impact of advanced oxidation process on the ratio of the particular fraction contents in dry mass of sewage sludge. The study under sewage sludge stabilization by Fenton's reagent with the catalyst of $FeCl_3$ let obtain, after 24 h of retention time, the concentration of volatile matter in the range of 12.988 $g \cdot L^{-1}$ to 12.142 $g \cdot L^{-1}$ depending on the reagents doses, at the initial concentration 15.228 $g \cdot L^{-1}$. Even the application of constant magnetic field did not improve final technological effects [13].

Different effects concerning organic substances' transformation in dry mass of sewage sludge were observed when the advanced oxidation process was associated with typical aerobic stabilization with compressed air [19]. In this technology of sewage sludge processing the retention time of the sludge in tanks was about 20 days. Chemical reagents were dosed into the system during the first 8 days of the experiment in the amount of 2.5 g $H_2O_2 \cdot L^{-1}$ and 0.6 g $Fe^{2+} \cdot L^{-1}$ and the sum was 20 g $H_2O_2 \cdot L^{-1}$ and 5.0 g $Fe^{2+} \cdot L^{-1}$, respectively. At the end of the experimental cycle in the reactor supported by compressed air and H_2O_2 the efficiency of volatile matter reduction in dry mass of sewage sludge was 58.2%. However, in the reactor with Fe^{2+}/H_2O_2 the effectiveness increased to 80.6%.

Similarly, application of the longer retention time in the reactor and higher doses of chemical reagents introduced into the sewage sludge could let obtain much higher final technological effects. Authors have proved that the increase in operation cost directly associated with high amounts of chemical reagents might classified this method as expensive and economically poor. The study could be educational, but there would be no chance to apply this technology in practice.

It is generally accepted that increase of Fenton reaction dose influenced the waste oxidation effect. Required doses of those oxidants are held in a wide range and it is connected with the kind of waste and expected level of treatment [19, 21].

In the experiment doses of the reacting substances applied in the technological system ranged from 0.25 g $Fe \cdot L^{-1}$; 1.0 g $H_2O_2 \cdot L^{-1}$ to 2.0 g $Fe \cdot L^{-1}$; 8.0 g $H_2O_2 \cdot L^{-1}$. Doses of chemical reagents, applied in the experiment, were comparable or even lower, contrary to the doses presented in the literature studies concerning advanced oxidation in sewage sludge conditioning and stabilization [8, 9, 20]. Moreover, presented results proved that organic pollutant oxidation and reduction was effective in the supernatant after vacuum filtration but organic matter oxidation in dry mass of sewage sludge was not observed. It seems to be necessary to use higher doses of Fenton's reagent. However, higher doses of Fenton's reagent could cause an increase in operating costs, and it could influence the competition using Fenton reaction and conventional methods of sewage sludge treatment. The authors expected a more significant impact on the obtained results of the stabilization of sewage sludge.

The optimal pH for various substrate oxidation would range from pH 2.0 to pH 4.0, or even from pH 5.0 to pH 7.0 [22-24]. In some cases the effectiveness of Fenton reaction is the same in a wide range of the reaction. Such a relation was observed for advanced oxidation process used for the treatment of landfill leachate. Fixed reaction rate and highly efficient results was observed in the wide range of the reaction from pH 3.0 to pH 9.0 [11]. Fixed reaction rate and highly efficient results were observed in the wide range of the reaction from pH 3.0 to pH 9.0 during decolourization of some kind of synthetic dyes [24]. Base on the bibliographic data presented above during the experiment, authors did not correct pH of the sludge before the addition of Fenton's reagent.

Conclusions

- The reduction of volatile matter in mass of sewage sludge was observed merely in the range of low doses of Fenton's reagents (Fe^{2+}/H_2O_2).
- Application of constant magnetic field in the technological system improve the efficiency of COD removal in the supernatant, the impact of physical element is especially significant in the range of low doses of chemical reagents.
- Similar results were achieved independently if physical element was tested as the element improving advanced oxidation process in the system with scCMF or ccCMF.
- There were no changes in sewage sludge properties if constant magnetic field was the only element affecting sewage sludge.

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