American Journal of Chemical Engineering
2016; 4(2): 30-37
http://www.sciencepublishinggroup.com/j/ajche
doi: 10.11648/j.ajche.20160402.11
ISSN: 2330-8605 (Print); ISSN: 2330-8613 (Online)

Research Article

An Alternative Arrangement for the Alum Sludge Management: Minimising Waste with Low-Cost Solar Techniques

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To cite this article:

Maha A. Tony, Aghareed M. Tayeb, Yaqian Zhao. An Alternative Arrangement for the Alum Sludge Management: Minimising Waste with Low-Cost Solar Techniques. *American Journal of Chemical Engineering*. Vol. 4, No. 2, 2016, pp. 30-37. doi: 10.11648/j.ajche.20160402.11

Received: January 22, 2016; **Accepted**: January 30, 2016; **Published**: March 31, 2016

Abstract: Alum sludge produced from the drinking water treatment plants was used to investigate the improvement capabilities in its dewatering properties. The sludge was passed through a laboratory solar still towards reducing the sludge volume during the dewatering process. A number of parameters describing the distillate and the sludge were measured at the end of each experiment in order to determine the process behaviour under conditions of relatively high solar radiation which is reached to 1014 W/m^2 and temperature levels which reaches inside the still to a maximum of 87°C. Conventional chemical conditioners in augmentation with the solar dewatering such as polyelectrolyte, and advanced conditioner like photo-Fenton's reagent were used in the sludge conditioning studies. Experimental results indicated that 10 mg/L of anionic polyelectrolyte conditioner enhance the dewateraibility in the terms of SRF reduction to 97%. However, it reached to 78% when the Fenton's reagent is added. It is realized that dewatering is accelerated when the polymer is added compared to that of Fenton's reagent conditioning; Fenton's reagent offers a more environmentally safe option. Moreover, the volume of distillate collected is nearly a double fold increase in the case of the conditioning with the Fenton's reagent rather than that for the organic polymer. In addition, the turbidity of the supernatant are: 3.4, 2.7 and 247 NTU for polyelectrolyte, Fenton's reagent treatment and for the raw sludge, respectively. Furthermore, the optimum influencing variables of Fenton's reagent is evaluated by applying Box-Behnken experimental design based on the response surface methodology (RSM), i.e. Fe^{2+}, H_2O_2 and pH are 50 mg/L, 600 mg/l and 8.5, respectively.

Keywords: Alum Sludge, Dewatering and Conditioning, Fenton's Reagent, Organic Polymers, Solar Still, RSM

1. Introduction

Alum sludge is produced in large amounts from the drinking water treatment plants as a result of using aluminium sulphate as a primary coagulant. The characteristics of this sludge make it difficult to dewater. Therefore, effective conditioning should be applied to improve its dewaterability. Traditionally, chemical conditioning prepares the sludge for a better and more economical treatment. Such chemicals of ferric salts, lime and alum especially the organic polymers have widely been applied to improve the dewatering properties before the mechanical dewatering of the sludge [1, 2]. However, these polymers may have unknown potential risks to the environment from a long term point of view [2]. This leads to searching for a more economical and feasible solution regarding alternative conditioning technique.

Consequently, the advanced oxidation processes, especially Fenton's reactions have been emerged as an alternative conditioning technique [2-4].

Although the Fenton's reagent, which is a solution of hydrogen peroxide and iron catalyst, had been applied for wastewater sludge conditioning [3, 9-12], our previous studies to investigate alum sludge conditioning with Fenton's reagent ($FeCl_2$ and H_2O_2) have demonstrated a considerable success of 47% reduction in the specific resistance to filtration (SRF) [2, 13, 14].

The key procedure in sludge treatment is water removal. Currently the most general form of sludge disposal is sanitary landfilling which necessitate the sludge to be dewatered to 35% of solids [15]. Significance to expelled moisture by the application of means of solar energy has been applied for the dewatering of wastewater sludge by using solar still [15, 16] and solar dryers [17] especially in the region of "solar-rich" countries, such as The Middle East region. Moreover, interest in the simple solar still has been mainly due to the simplicity of its construction and design, besides the low operational and maintenance costs. The results reveal that the sludge containing water can be dewatered to a certain extent [15]. However, according to the literature the process seems not applied thus far for alum sludge dewatering. Furthermore, the augmentation between photo-Fenton's reagent and solar dewatering is not explored in the literature. Since there may be some disadvantages when applying the solar dewatering procedure in some places. For example, the weather is not always sunny and the temperature of sunrays cannot be controlled [18]. However, Egypt has endowed with a high solar intensity; the annual global radiation is between 7 and 9 GJ/m^2. In south of Egypt, winters are mild and summers are hot during daytime and warm at night, with maximum temperatures ranging from 34°C to 40.8°C. Sunshine is available on the ground surface well over 90% of the day time [19, 20].

Factors to control the Fenton reaction process are the amounts of Fe^{2+} and H_2O_2, and the operating pH. Response surface methodology (RSM) is a technique used for the modelling and analysis of problems in which a response of interest is influenced by several variables with the objective of optimizing this response [21]. Although this powerful experimental design tool has been increasingly applied in many fields to investigate the process conditions that provide enhanced treatment of contaminated soils and different wastewaters [22-25], it has not been well exploited to optimize the solar photo-Fenton oxidation variables in sludge treatment according to the literature.

In this study, photo-Fenton reagent was employed to condition alum sludge from the drinking water treatment collected from a local water treatment plant in Egypt. Furthermore, the combination of Fenton's reagent conditioning and solar energy dewatering is explored for the dewatering of the alum sludge. Focuses are placed on the effectiveness of Fenton reaction in improving sludge dewaterability and the optimization of Fenton reaction conditions (Fe^{2+}, H_2O_2 and pH) using RSM to achieve the maximum SRF reduction of the sludge. Moreover, investigation on dewatering by applying the polyelectrolyte dose was also conducted for the comparative purpose. Here we attempt to utilize solar energy in order to evaporate and collect the water of the sludge through the solar still. The incident radiation and the temperatures that developed are monitored, whereas key parameters of the sludge and the distillate are determined at the end of the study.

2. Materials and Methods

2.1. Experimental Materials

Alum sludge samples used during this study were taken from a water treatment plant, Kedwan Station in Minia city, in the south of Egypt. In the plant the treatment process uses aluminium sulphate to treat raw water taken from The River Nile. Principle properties of the alum sludge are given in Table 1. Fenton reagent, as the conditioner, are prepared by making a solution from Fe^{2+}, namely, Ferrous Oxalate ($Fe(C_2O_4)$) and Commercial H_2O_2 (30% by wt.) was used. Sulfuric acid and sodium hydroxide are used for pH adjustment of the sludge samples. Magnafloc LT-25 (0.01% by wt.) was also used for the sludge conditioning tests for comparative purpose.

Table 1. *Properties of alum sludge used in this study.*

Parameters	Unit	Value
Suspend solid (SS)	mg/l	2,364
pH		8.5
SRF	m/kg	2.24×10^{13}
Turbidity (supernatant)	NTU	274
Moisture Content	%	97

2.2. Basics of Solar Still and Process Description

The solar still used for the present study is similar to the ones employed for seawater desalination. The basic principles of solar still are to use natural evaporation and condensation. The sun's energy heats water in the sludge to the point of evaporation. As the water evaporates, water vapour rises, condensing on the glass surface for collection. The intensity of solar energy falling on the still is the single most important parameter affecting production. It consists of three basic parts: the water basin, the transparent and the distillate trough (see Fig. 1). The water basin is 80 cm in width and 125 cm in length. It was constructed from a galvanized steel sheet with a thickness of 0.001 m. The sides and the bottom of the basin were painted black in order to increase the absorption of the incident solar radiation whereas the transparent cover, made of Plexiglas (0.003 m thickness), had a slope of 30°. Finally a thermal insulation sheet was placed around the basin in order to reduce any thermal losses. An initial quantity of 20 L of the alum sludge is placed to the still in the solar basin and left outdoors. However, when Fenton's reagent or polyelectrolyete were added, as conditioners, the sludge is subjected for 30 s rapid mixing in order to promote flocculation before the sludge is introduced into the still. The sludge is stirred manually in the solar still at definite times in order to homogenize the solution.

The experimental setup is located at the Solar Energy Lab., Chemical Engineering Department of Minia University, Egypt, where the latitude of the town is 28° and it is 130 m above sea level. The region usually enjoys mild, sunny and dry seasons [26, 27]. Tests were carried out during the period of May to September 2015. Intensity of solar radiation was measured by a solarimeter (Epply Black-and-White Solarimeter, Model 8-48). Experiments were started at 9.00 am and continued till the exact reaction time needed during the day time. For other experiments those need more than one day the sludge was left in the still for more than one day.

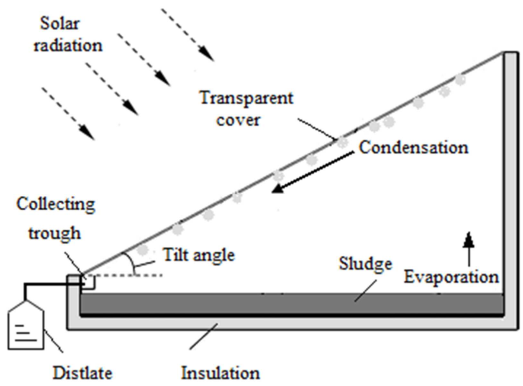


Figure 1. *Schematic diagram of the experimental set-up.*

2.3. Experimental Design and Optimizing

Factors to control the Fenton reaction process are the amounts of Fe^{2+} and H_2O_2, and pH. Optimizing such amounts plays a key role towards the success of the Fenton process. Through the classical method, optimization involves the changing of one variable, say amount of Fe^{2+}, at a time while fixing all other variables at one level and studying the effect of the variable on the response. This is an extremely time-consuming, expensive, and complicated process for a multi-variable/parameter system. To overcome this difficulty, a statistical-based technique commonly called the response surface method (RSM) as a powerful experimental design tool has been used.

Box–Behnken design [18], which is the standard of RSM, was selected for the optimization of photo-Fenton's parameters. This factorial design was employed to fit the second-order polynomial models and to obtain an experimental error for this study. Since different variables have different limits of variation and may have different units, the significance of their effects on response can only be compared after they are coded. For statistical calculations, the three independent variables, i.e. initial Fe^{2+}, H_2O_2 and pH, were coded as A, B, C, respectively and their ranges and levels are presented in Table 2.

Table 2. *Experimental range and levels of the independent variables.*

Variable	Range and levels		
	-1	0	1
A, initial Fe^{2+} (mg/l)	20	40	60
B, inital H_2O_2 (mg/l)	200	400	600
C, pH	3.5	6.0	8.5

2.4. Analytical Methods

Dewaterability of the sludge under solar, Fenton and polymer conditioning is demonstrated. Samples from both the distillate and the alum sludge were collected and brought to the laboratory for further analysis.

Three samples from the sludge were taken at different time intervals to measure its dewaterability evaluated by SRF, which was measured using a standard SRF test. A change of sludge's SRF before and after conditioning and the percentage reduction is monitored via the following equation:

$$\eta(\%) = \frac{SRF_0 - SRF}{SRF_0} \times 100 \quad (1)$$

where η is the % SRF reduction and SRF0 and SRF are, respectively, the SRF of alum sludge before and after conditioning. Furthermore, the settling test was used to evaluate the sludge dewaterability. A 100 mL of the sludge is poured into a graduated cylinder and the floc/liquid interface was observed to record the settling test.

The distillate collected in a volumetric flask was measured, then its turbidity (using a HACH 2100N IS Turbidimeter) and pH (using a digital pH-meter, model pH-206) is recorded and compared with that of the supernatant of raw sludge.

3. Results and Discussion

3.1. Performance Analysis and Temperature Profile for the Still

Solar radiation is an important parameter with the highest effect on the productivity as it provides the energy requirement for water evaporation from the sludge. Fig. 2(a) illustrates the solar radiation during the day.

Throughout the experiment relatively high levels of solar radiation prevailed, with maximum values in the range of 1014 W/m^2, whereas ambient temperature also remained high. As it can be seen in the figure the mean ambient air temperature during the day is varied from 25 to 39°C. Fig. 2(a) presents the variation of temperatures and horizontal solar radiation for the day of the experiment. Before sunrise, the ambient air temperature is 24–25°C which then rises to 38–39°C at midday. The temperatures of the solar still follow at higher levels: the air temperature inside the still reaches a maximum of 83°C, whereas the temperature of the sludge inside the basin reaches a maximum of 87°C.

An obvious behavior of increase in the temperature with the increase in the solar radiation can be noticed and consequently the reduction in the SRF of the sludge is achieved, as shown in Fig. 2(b), whereas after the midday there is a steady decrease of SRF since there is no replenishment of alum sludge under the batch operation of the experiment. At the end of the day, all the remaining sludge inside the basin is sludge cake.

It is clear from the results that both solar radiation and temperature have a significant influence on the evaporation rate and sludge dewatering. When the solar radiation and ambient temperature are low at 767 W/m^2 and 25°C, respectively, the SRF reduction ranged from 22 to 48%. However, it reached to 67% for the higher solar radiation (1014 W/m^2) and ambient temperature (39°C). With the onset of solar radiation and the increase of temperatures the evaporation and subsequent condensation started and it continued throughout the day. However, when solar radiation had dropped to zero and condensation was halted, the evaporation stops. Thus, the high temperature makes the water evaporation from the sludge and the still allows the water transport out of the unit. These are in accordance with previous observations concerning the evaporation of the sewage sludge using solar techniques [28].

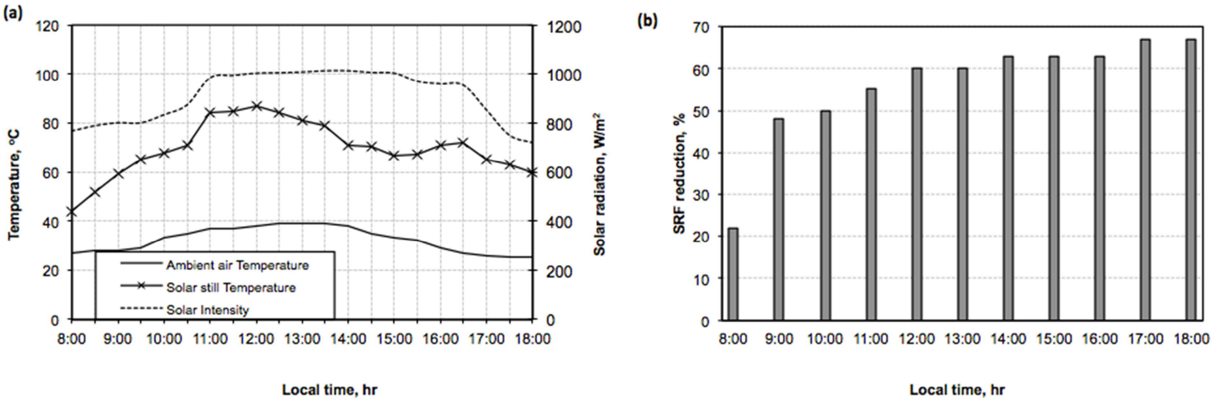


Figure 2. *Effect of temperature during a typical day (11July 2015) on the sludge dewatering: (a) Temperatures and solar radiation variation; (b) SRF reduction %.*

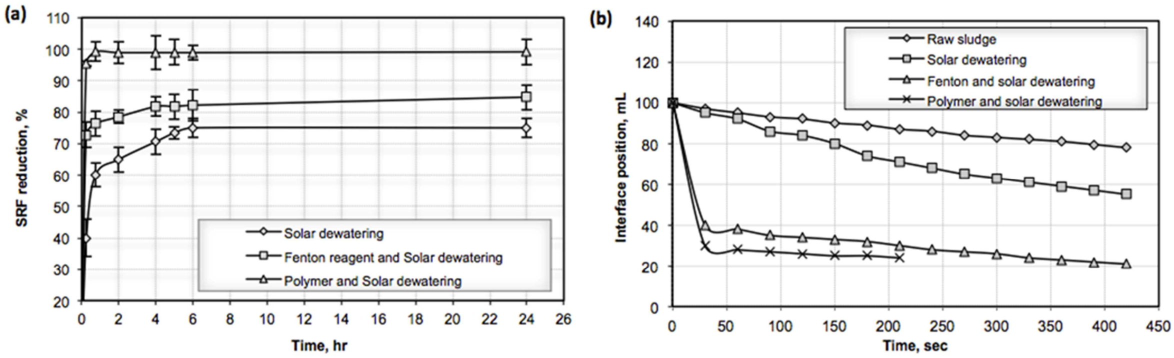


Figure 3. *Alum sludge dewatering using solar and solar-chemical dewatering techniques (a) SRF reduction; (b) settling test.*

3.2. Dewatering of Alum Sludge in a Solar Still

For the purpose of comparison between solar dewatering and chemical-solar dewatering for the alum sludge, experiments were carried out in three manners. They are solar dewatering and solar dewatering with Fenton reagent condition and polymer conditioning, respectively. The experimental conditions are based on earlier works by the authors, in which the optimum polyelectrolyte dose of 10 mg/L is added and the range of the Fenton's reagent is chosen [2, 13]. Fig. 3 jointly illustrates the effectiveness of alum sludge dewatering in the solar still, together with a separate Fenton's regent and polymer addition for comparing such techniques. Initially, raw alum sludge is subjected to the solar still and the reaction time is investigated. For the Fenton's reagent experiment, the doses of the reagent were: $[Fe^{2+}]$ = 40 mg/L; $[H_2O_2]$=100 mg/L. They were added to the sludge and the starting pH of the sludge 8.5 without any adjustment; then the sludge was poured into the solar still. Furthermore, 10 mg/L of anionic LT-25 polymer was added in another experiment then the sludge is left in the still.

Fig. 3 illustrates the SRF and settling test during the alum sludge dewatering in solar and solar-chemical conditioning. In order to provide the insight into such the characteristics, measurements of the distillate turbidity is provided. Examination of the results shows that for all the treatment methods, % SRF reduction increased with the exposure time, then the reduction becomes slow during the day. It was observed that the treatment resulted in 67, 74 and 97% of the SRF reduction for solar dewatering, solar Fenton's reagent dewatering and solar polyelectrolyete dewatering, respectively. The microscope observation clarifies that, the sludge dewatering behaviour is accelerated and the SRF reduction is increased when using a conditioner.

In the solar dewatering technique without a conditioner, only the solar radiation is the responsible of the dewatering by evaporating water in the sludge and the remaining is the dry solids in the sludge [17]. It has been observed from the experiments that the remaining in the still after the day was slurry from the sludge cake.

In the case of polymer conditioning, the mechanism of the reaction is different as the polymers have the function of charge neutralization and inter particle or primary flocs bridging. These values notably have a remarkable difference with that of the raw sludge. This change is similar to the results reported in the literature [1, 2].

In the case of solar Fenton's dewatering/conditioning, it was observed that the number and size of alum sludge flocks decreased by the solar photo-Fenton reaction. This is consistent with the observation for the sludge minimization by Fenton's reagent [2]. The dewatering is carried out when the •OH radicals produced by photo-Fenton reaction attacked the sludge and broke up the sludge flocs. During the sludge dewatering, H_2O_2 was consumed by the photo-Fenton reaction. Therefore H_2O_2 concentration continuously decreased with time. Dissolved total Fe ion (Fe^{2+} plus Fe^{3+}) concentration in the sludge rapidly decreased just after the initiation of the photo-Fenton reaction and was rather little during the photo-Fenton reaction. It might be due to the reason that most Fe ions were entrapped to the sludge [2].

As the sludge lost its water in the form of distillate, the total volume of distilled water in mL produced by the solar still was collected (Fig. 4). The turbidity of the distillate collected was measured and compared with the supernatant of the raw sludge. It was found that, the turbidity of the distillate collected during the dewatering of the sludge using polyelectrolyte is 3.4 NTU; however, it is 2.7 NTU for treating it with Fenton's reagent, compared to 247 NTU for the supernatant of the raw sludge.

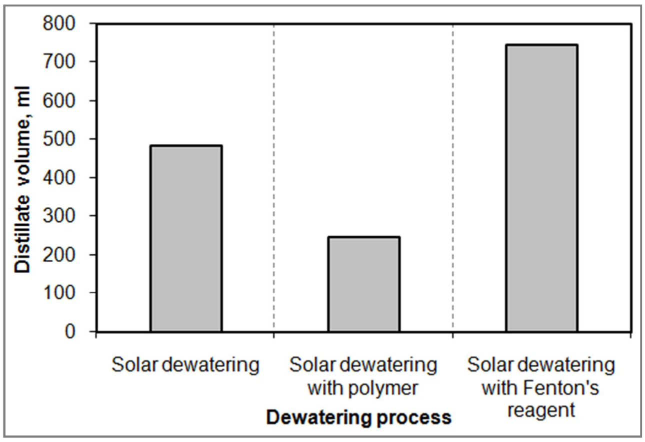


Figure 4. *Distillate volume variation during the experiment.*

The distillate shows increased contamination due to the intense production of vapours carrying volatile organic compounds inside the still, rendering it useless for further usage. However, in the case of the Fenton's reagent, it showed lower NTU value, meaning Fenton's reagent helps in oxidising the organic pollutants in the sludge, thus the distillate collected is in low turbid. The experiment showed the feasibility of the solar dewatering of the alum sludge especially in the case of using Fenton's reagent as a conditioner, thus providing a possible solution regarding the handling of the excess volumes produced in a typical drinking water treatment plant with secondary treatment.

The obvious and largest flocs were observed when the polymers were added. However, Fenton's reagent does not show particle bridging in contrast to the synthetic polyelectrolytes. Consequently, small floc-like appearance will be expected. This may be the reason of the SRF reduction is maximized when the polyelectrolyte is applied as a conditioner besides the solar dewatering. However, notwithstanding the SRF reduction increases, Fenton's reagent has a potential advantage of eliminating the perceived long term risk associated with polymer residual in the environment [13].

3.3. Optimising Fenton's Reagent Variables Using SRM

The main objective of the optimization is to determine the optimum values of solar-Fenton's reagent variables for SRF reduction efficiency from the model obtained via experiment. Fifteen runs of the experiment were required for a complete set of optimising requirement using various ratios of Fe^{2+}/ H_2O_2 and different pH values.

The coefficients of the model equation and their statistical significance were evaluated using Box-Behnken factorial design. The experimental design and the SRF reduction rate (%) of the sludge obtained from the experiments and as the responses predicted are shown in Table 3.

Table 3. *Factorial design of alum sludge treated by solar photo-Fenton's reagent.*

Run no.	Coded factors			Response (η, %)	
	A	B	C	Experimental	Predicted
1	-1	-1	0	41.76	41.71
2	-1	1	0	36.88	36.82
3	1	-1	0	70.15	70.09
4	1	1	0	65.28	59.79
5	0	-1	-1	61.27	72.10
6	0	-1	1	76.12	65.29
7	0	1	-1	60.40	71.24
8	0	1	1	67.21	56.39
9	-1	0	-1	20.43	20.44
10	1	0	-1	50.52	61.35
11	-1	0	1	32.95	32.97
12	1	0	1	59.65	54.24
13	0	0	0	62.98	62.50
14	0	0	0	62.98	62.50
15	0	0	0	62.98	62.50

The regression model for the % SRF reduction of the sludge in terms of coded factors is given by the following second-order fitting polynomial equation.

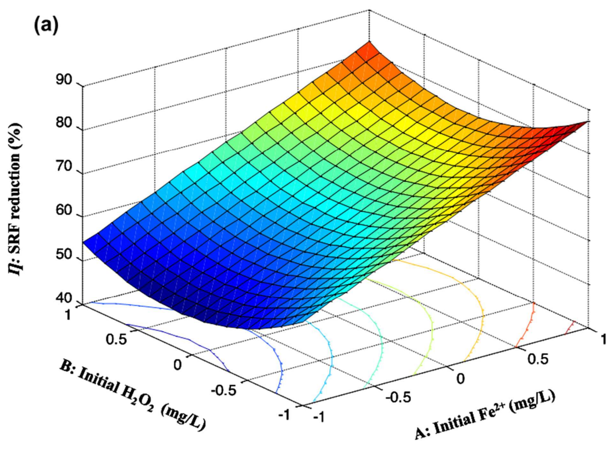
$$\eta(\%) = 62.57 + 14.19X_1 - 2.44X_2 + 5.41X_3 - 17.21X_1^2 - 0.85X_1X_3 + 8.16X_2^2 - 2.01X_2X_3 - 4.47X_3^2 \quad (2)$$

The analysis of variance (ANOVA results) incorporating the lack-of-fit test using the software of Statistical Analysis System [20] is applied to evaluate the model (data not shown). Based on the lack-of-fit analysis, the second-order response model appeared to adequately fit the data (P-value>0.05). Based on the test for significance, the second-order regression model is significant (P-value<0.05), and this model explains about 99% of the data variability. The measured and the predicted data show that the proposed empirical model is suitable for predicting %SRF reduction, revealing a reasonably good agreement. This confirms that the predicted model can be used to navigate the design space defined by the Box-Behnken design.

The graphical representations of the model (Eq. (2)) facilitate an examination of the effects of the experimental factors on the responses, 3D surface graphs and contour plots between the factors were obtained using the MATLAB 7.0 software and are presented in Fig. 5. This figure illustrates the responses of different experimental variables and can be used to identify the major interactions between the variables [23]. The 3D surface graph and contour plot in Fig. 5a shows that the reduction of SRF increased with the increase in the Fe^{2+} and H_2O_2 concentrations. Nevertheless, by the further increase in the reagent doses, an opposite effect appears as the SRF reduction decreased. It appears that the H_2O_2 and Fe^{2+} concentrations plays an important role in Fenton reaction since excess addition of H_2O_2 may lead to the negative impact on sludge dewaterability. The reason may be attributed to the amount of hydroxyl radicals. When H_2O_2 and Fe^{2+} concentrations increase until a critical concentration, a so-called scavenging effect occurred. Several references are available with concern of the hydroxyl radical production on the effect of Fenton reaction [24, 29]. The response surface in Fig. 5a shows a curvature. This indicate that the interaction effect between Fe^{2+} concentration and H_2O_2 dosage on SRF reduction is greatly pronounced, as confirmed by significance test.

Fig. 5b illustrates the response surface assuming the initial pH and the Fe^{2+} concentration as independent factors. For a constant Fe^{2+} dose, the SRF reduction increased with the increase in the initial pH from 3.5 to 8.5. It is evident that there is an obvious interaction between the initial pH and the initial Fe^{2+} concentration. The optimum pH with the higher removal is 8.5 for the original pH of the raw sludge. It is interesting that this finding in our research for the original raw sludge optimum pH medium is consistent the previous finding in the literature [2, 30].

It is known that sub-optimal pH can decrease the amount of hydroxyl radicals, which is supposed to be the driving force towards the development the sludge dewatering [7, 31]. The high %SRF reduction efficiency achieved at the relatively high initial pH implies lower costs for pH adjustment and lower amounts of sulfate introduced.



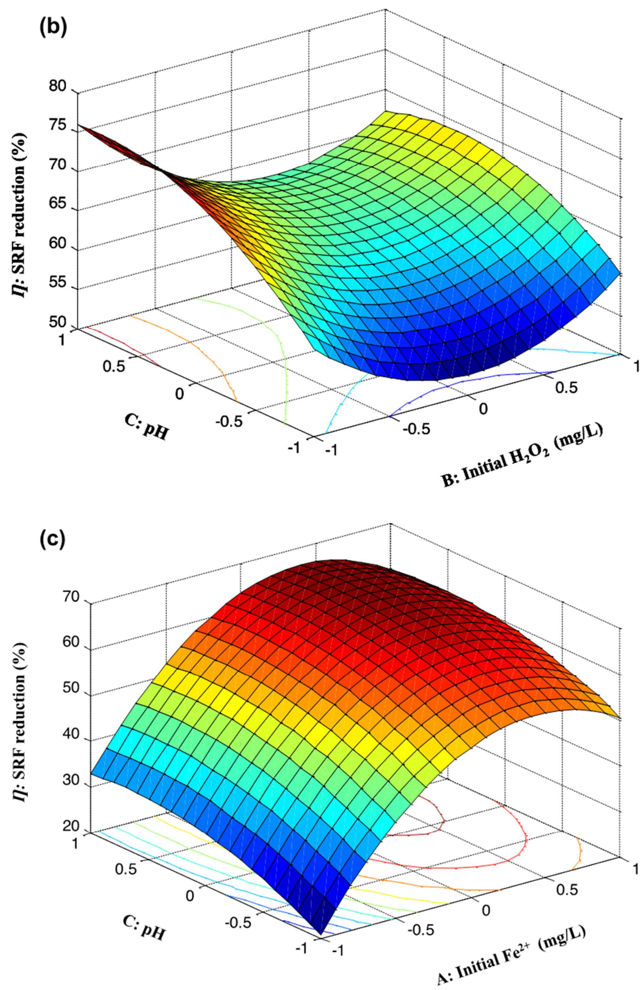


Figure 5. *Response surface and contour plot for alum sludge dewatering: (a) coded Fe^{2+} and H_2O_2 vs. predicted %SRF; (b) coded Fe^{2+} and pH vs. predicted %SRF; (c) coded H_2O_2 and pH vs. %SRF.*

Several reports [9, 32, 33] on Fenton treatment of sludge claimed that the optimum initial pH was in the acidic range 2.5–4.0. This discrepancy between these findings and the results of our work is probably due to the characteristics variability in the sludge and difference in the process mechanism.

Fig. 5b also clearly shows a curvature in the three-dimensional plot. As shown in Fig. 5, an improved %SRF reduction is observed when $[Fe^{2+}]$ increased. However, an increase in $[Fe^{2+}]$ beyond the optimum region resulted in decreasing the %SRF reduction. This is mainly due to the fact that the excess of Fe^{2+} could negatively affect the coagulation–flocculation process and scavenge hydroxyl radicals generated through the reaction of Fenton's reagents [34, 35].

The response surface as a function of the factors initial pH and $[H_2O_2]$ is shown in Fig. 5c. At a high initial pH, the %SRF reduction increased dramatically with increased $[H_2O_2]$. The largest SRF reduction occurred when a high $[H_2O_2]$ together with a high initial pH was used, while the poorest %SRF occurred when the $[H_2O_2]$ and the initial pH were at their low level. At a high initial pH (8.5), there is an optimum $[H_2O_2]$.

This finding also indicates that the interaction between initial pH and [H_2O_2] is obvious. Such a finding is available in the literature confirming this [2, 8].

Thus, it is reasonable to believe that model is accepted as regression coefficient is more than 80% and the optimum values are recorded in Table 4. Then these values are used to conduct an experiment and the SRF reduction is in a good agreement with the predicted one which is 77%.

Table 4. *Optimum value of the process parameter for maximum efficiency.*

Parameters	Optimum values
η (SRF reduction rate %)	78
Fe^{2+} (mg/l)	50
H_2O_2 (mg/l)	600
pH	8.5

4. Conclusions

The proposed solar still dewatering process was efficient for extended dewatering and volume reduction of alum sludge. The SRF of the dewatered sludge decreased to reach to a reduction up to 67% within one daytime. The sludge lost all its water, which was collected as distillate from the still. It is realised that, the system performance is increased by the proper use of a chemical conditioner besides the solar dewatering. Maximum SRF reduction for the sludge by using the solar still is 98% by using polyelectrolyete conditioner and 78% for Fenton's reagent conditioner. The experiment showed the feasibility of the solar dewatering of the alum sludge that points towards a possible solution regarding the handling of the excess volumes produced in a typical drinking water treatment plants.

Acknowledgements

The authors wish to appreciate the Water Treatment plant in Kedwan, Elminia, in the south of Egypt, for providing the sludge samples.

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