Full Length Research Paper

Optimization of coagulation-flocculation process for pastas industry effluent using response surface methodology

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The goal of this study was to characterize and then optimize the treatment process of effluent generated from couscous and pasta industry. The crude effluent is characterized by a high level of organic concentration (from 2269 to 4383 mg/l). The optimization of coagulation-flocculation process was then performed to minimize chemical oxygen demand (COD). The experiments were carried out using the response surface methodology (RSM). A central composite design (CCD) was used to evaluate the effects and the interactions of three factors; coagulant dosage, flocculant dosage and pH on the wastewater treatment efficiency. Quadratic models was developed for the response variables and it successfully describes the experimental data (R^2= 0.728). Analysis of variance (ANOVA) showed that only the coefficients corresponding to the flocculant dosage were significant (p < 0.0001). In order to take into account the variability of the initial organic concentration of the effluent, the CCD was repeated thrice by using a wastewater having initially different organic charges (530, 1775 and 2722 mg/l). Whatever was the initial organic charge of the wastewater, the optimal coagulant dosage was found constant (about 50 mg/l) and the optimal pH value was about 11.0. On the contrary, the optimal flocculant dosage varied linearly (from 24 to 50 mg/l) with the initial COD (from 529 to 2722 mg/l). These optimal conditions allow a COD removal varying from 78 to 93%. Irrigation and germination tests were carried out with treated and untreated wastewater and they showed that these effluents could be adequately recycled in culture irrigation.

Key words: Couscous and pasta industry effluent, COD removal, central composite design, response surface methodology (RSM), irrigation tests.

INTRODUCTION

Several wastewater treatment methods are available as on date in order to ensure good quality effluent before disposal into the municipal sewer systems (Drogui et al., 2008). The wastewaters vary in terms of the pollutant composition depending on the origin of the industry. The disposal of such effluents in the environment will lead to surface and groundwater contamination: increase in chemical oxygen demand, eutrophisation, ecosystem imbalance and human health risks (Bektas et al., 2004).

The pasta industry produces wastewater with high concentrations of organic compounds that are not completely removed by preliminary treatments or biological process (Roa-Morales et al., 2007). The high organic load of pasta industry represents a major environmental problem. With the establishment of more stringent regulations concerning wastewater treatment, there has been a growing interest in the development of

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new technologies and procedures for the decontamination of water (Beltran de Heredia et al., 2004).

In a general way, biological processes (aerobic or anaerobic systems) are widely used for the treatment of agro-industry wastewaters; which contain high concentration of biodegradable organic matter (in term of biological oxygen demand) (Satyanarayan et al., 2005). Even though biological processes are effective and economical long, hydraulic retention time and large area requirements make sometimes these processes less attractive than physic-chemical treatments, which require shorter retention time (Drogui et al., 2008). However, treated water coming from treatment plant does not comply with environmental discharge limits. The obstinacy of organic pollution in wastewaters can cause an environmental problem. Besides, biological treatment system cannot respond quickly to the large variation in effluent quantity and BOD/COD value. Therefore, new wastewater treatment methods are needed. Coagulation/ flocculation process is one of the most efficient methods that is widely applied for industrial wastewater treatment as it is efficient and simple to operate (Jian-Ping et al., 2007; Sadri Moghaddam et al., 2010; Trinh and Kang, 2011). Chemical coagulation followed by sedimentation is a proven technique for the treatment of high suspended solids wastewater especially those formed by the colloidal matters. Research and practical applications have shown that coagulation will lower the pollution load and could generate an adequate water removal (Ahmad et al., 2005; 2007; Khouni et al, 2011).

The limitations of coagulation/flocculation are that it is not always effective and there are problems associated with sludge disposal; hence, the interest in coagulant recovery stems, not only from the potential savings in chemical costs, but also from substantial reductions in sludge volume. Some researches have been conducted to show the feasibility of recycling the coagulants (Najafi and Movahed, 2009).

Coagulation is mainly done with inorganic metal salts (aluminum and ferric sulphates and chlorides) and polyelectrolytes of various structures (polyacrylamides, chitosan, polysaccharides) (Aguilar et al., 2005). According to Duran and Georgy (2003) aluminum and iron salts are widely used as coagulants in water and wastewater treatment and in some other applications. Their mode of action is generally explained in terms of two distinct mechanisms: charge neutralization of negatively charged colloids by cationic hydrolysis products and incorporation of impurities in an amorphous hydroxide precipitate so-called sweep flocculation. It was found that the praestol anionic flocculant used simultaneously with aluminum sulfate at the water-treatment plants provides efficient purification of water and wastewater (Kurenkov et al., 2001).

In coagulation-flocculation process, many factors can influence its efficiency, such as the type and dosage of coagulant/flocculant (Desjardins et al., 2002), pH (Dominguez et al., 2005; Najafi and Movahed, 2009), mixing speed and time (Gurse et al., 2003; Amirtharajah and O'mella, 1990), temperature and retention time (Zhu et al., 2004), etc.

The optimization of these factors may significantly increase the process efficiency. In conventional multi-factors experiments, optimization is usually carried out by varying a single factor, while keeping all other factors fixed at a specific set of conditions. It is not only time-consuming, but also usually incapable of reaching the true optimum due to ignoring the interactions among variables. On the other hand, the response surface methodology (RSM) has been proposed to determine the influences of individual factors and their interactive influences. The RSM is a statistical technique for designing experiments, building models, evaluating the effects of several factors and searching optimum conditions for desirable responses. With RSM, the interactions of possible influencing parameters on treatment efficiency can be evaluated with a limited number of planned experiments (Biles and Swain, 1980; Jian-Ping et al., 2007; Wang et al., 2007).

The main objective of this work was to optimize the coagulation-flocculation process of a pasta industry wastewater and to investigate the interactive effects of experimental factors, including coagulant dosage, flocculant dosage and pH. The response surface methodology (RSM) using a central composite design (CCD) is selected for the optimization.

MATERIALS AND METHODS

Wastewater and physicochemical characterization

The wastewater used in this study was taken from a couscous and pasta manufacture in Sfax, (southern of Tunisia). The industry has implanted until 1998 a wastewater treatment station. The aim of this treatment plant was to treat wastewaters generated by the industrial activity using coagulation-floculation process. Wastewater treatment plant has a feed tank where industrial effluent are collected and pumped to the coagulation tank and then to the flocculation tank. The treated effluent was finally collected in decantation tank. Samples were collected within about three months from February to April 2010. Sample collection, preservation and measurements were done in accordance with the standard methods for the examination of water and wastewater (APHA, 1992). Physicochemical characterization (pH, T, COD, BOD_5, TSS, TS, VS and TKN-N) was carried out immediately after samples arrived in the laboratory.

A multifunction pH-meter Testo model (ECOMETP25-Korea South) was used to measure pH (precision 0.01) and temperature (precision 0.01). The physicochemical analyses were performed in triplicate. The results are expressed as mean value (MV) ± error deviation (ED). The coefficient of variation was calculated as:

$$CV = \frac{ED}{MV} \times 100 \text{, where } SER = \sqrt{\frac{\Sigma \left(MV - \overline{MV}\right)^2}{n-1}}$$

Before realizing the optimization of the coagulation-flocculation

Table 1. Levels of the variable tested in the 2^3 central composite design.

Variables (factors)	Range and levels		
	+ α	0	- α
X_1: Coagulant dosage (mg/l)	30	50	70
X_2 : Flocculant dosage (mg/l)	25	40	55
X_3: pH	7	8,5	10

process, an evaluation of the functioning of the actual effluent treatment station during one month was performed. Physico-chemical analyses (pH, T, COD, BOD$_5$, TSS, TS, VS, TKN-N, NH$_4^+$-N, N$_{org}$-N, chloride ions and P$_t$) were performed upstream and downstream of the wastewater treatment plant (feed tank and after decantation tank). For each analysis two samples were taken each week during a month from the feed tank and out of the decantation tank. The average values of physicochemical parameters of wastewater recorded during a month were calculated on the eight measurements (2 samples × 4 weeks). This investigation allows the variability of physicochemical characterization of the wastewater before and after the coagulation/flocculation step.

Coagulation-flocculation

Industrial grade aluminum sulphate (Al$_2$(SO$_4$)$_3$ 14H$_2$O) was used as the coagulant and praestol® 2500 TR (high molecular, non-ionic polyelectrolyte based on poly- acrylamide, density : approx. 650 kg/m^{3}were, viscosity in tap water: 50 mPa/s) was used as the flocculant aid, respectively. The chemical compounds were obtained from Fisher Scientific U.K. Ltd. They were prepared by dissolving the powder with distilled water.

The coagulation-flocculation experiments were carried according to Ghafari et al. (2009) using the jar tests in 500 ml beakers. The sample was immediately stirred at a constant speed of 200 rpm for 2 min (rapid mixing), followed by a slow stirring at 40 rpm for 10 min (slow mixing). After preliminary investigations, the setting time was fixed at 30 min.

Experimental design and data analysis

A central composite design, CCD, with nine replicates at the central points was employed to fit a second-order polynomial model and to obtain an experimental error for this study. The CCD was applied with three design factors and three levels. The factors are the coagulant dosage (X_1), the flocculant dosage (X_2) and initial pH of the wastewater (X_3). The coded levels and the natural values of the factors set in this statistical experiment are shown in Table 1. The central values (zero level) chosen for the experimental design were as follows: coagulant dosage (50 mg/l); flocculant dosage, (40 mg/l); pH (8.50) (experiments trials from 15 to 23, Table 2).

COD reduction percentage (removal) was selected as the dependent variable. The response variable was fitted by a second-order model in the form of quadratic polynomial equation:

$$y = b0 + \sum_{i=1}^{k} biXi + \sum_{i=1}^{k} biiXi + \sum_{i}^{i<j} \sum_{j} bijXiXj$$

Where, y is the response variable to be modeled; X_i, and X_j the independent variables which influence y; b_0, b_i, b_{ii} and b_{ij} are the offset terms, the ith linear coefficient, the quadratic coefficient and

the ijth interaction coefficient, respectively. The actual design used in this work is presented in Table 2.

Analysis of variances (ANOVA) was used for graphical analyses of the data to obtain the interaction between the process variables and the responses. The quality of the fit polynomial model was expressed by the coefficient of determination R^2 and its statistical significance was checked by the Fisher F-test. Model terms were selected or rejected based on the P value (probability) with 95% confidence level. Three-dimensional plots and their respective contour plots were obtained based on the effects of the levels of two factors.

The experimental design, regression and statistical analysis were performed, by NemrodW® software (Mathieu et al., 2000). The optimum values of the selected variables were obtained by using the desirability function available in NemrodW® and also by analyzing the response surface contour plots.

Germination essays

Germination test was performed by using treated (optimal conditions of treatment) and untreated wastewater according to Zucconi (1981). Tap water was taken as a control (pH: 6.5, turbidity: 25 NTU, conductivity: 400 µS/cm, chloride concentration: 0.1 mg/l). The germination indexes were determined for two vegetable species alfalfa (Medicago sativa) and chickpea (Cicer arietinum). Ten seeds of each vegetable were placed on Petri dishes containing a filter paper and were irrigated with 10 ml of water. The Petri dishes were then placed during six days at 25 ± 1°C in darkness.

A germination index percentage (GI) was calculated by counting the number of germinating seeds and the average sum of seed's root elongation in a sample as related to the control (Gouider et al., 2010). Results were finally expressed according to the following formula:

GI = Number of germinated seeds in sample/number of germinated seeds in control × average of root lengths in sample/average of root lengths in control × 100.

A seed was considered germinated when its root length exceeded 5 mm. For root length below 5 mm, it was considered equal to 0 and the seed was not considered as germinated.

Irrigations tests

Two seeds species alfalfa (M. sativa) and fescue (Festuca arundinacea) were selected for irrigation experiment. A series of green house assays were carried out using the mentioned earlier irrigation waters (tap water, treated and untreated water) (optimal conditions).

Pots of 160 mm in diameters and 140 mm in height were filled with soil incorporated with peat (½ soil and ½ peat). Two seeds were sown uniformly in each pot to a depth of about 10 mm. All experiments were carried out in two replicates for each irrigation

Table 2. Design matrix and results of the central composite design. The factors are expressed in coded values and the optimization parameter in percent reduction. Y for percent COD reduction.

Run number	Factor			Response
	Coagulant dosage (mg/l) (X_1)	Flocculant dosage (mg/l) (X_2)	pH (X_3)	COD reduction (Y) (%)
1	30	25	7	68
2	70	25	7	65.96
3	30	55	7	53.19
4	70	55	7	55.44
5	30	25	10	80.85
6	70	25	10	85.1
7	30	55	10	42.55
8	70	55	10	34.04
9	16	40	8.5	57.45
10	84	40	8.5	59.57
11	50	15	8.5	61.7
12	50	65	8.5	42.55
13	50	40	6	46.8
14	50	40	11	91.48
15	50	40	8.5	63.17
16	50	40	8.5	53.19
17	50	40	8.5	68.08
18	50	40	8.5	74.46
19	50	40	8.5	70.8
20	50	40	8.5	78.43
21	50	40	8.5	71.5
22	50	40	8.5	72.98
23	50	40	8.5	74.6

water. Pots were placed in greenhouse for 30 days. Each pot was irrigated with 150 ml three times a week.

The growth and health of plants were monitored qualitatively and quantitatively during the experimental period.

RESULTS AND DISCUSSION

Evaluation of the physicochemical characteristics of the industrial wastewaters

Figures 1 and 2 show the evolutions of physicochemical parameters measured on the wastewater taken from the feed tank (FT) and after the decantation tank (DT) during a month. Figure 1a show the pH values of the wastewater measured on the upstream and decantation tanks. The pH values varied from 5.57 to 8.45 in the upstream tank. This could be attributed to the variability of chemical composition of the water discharged by this industrial activity, whereas the treated wastewater collected after the decantation tank presents a pH value ranging from 4.65 to 7.00. pH values are sometimes out of the ranges fixed by standards and this variability could be attributed to the alkalinization step which is arbitrarily realized.

The temperature of the wastewater (Figure 1b) varied from 21.3 to 32.64°C ± 2.75. These values are acceptable. The control of the wastewater temperature is important in the treatment process. In fact, an increase of temperature partially destroys the current fauna and flora in the environment. It induces the increase of some products toxic power and decreases consequently, the quantity of dissolved oxygen in the medium (Derwich et al., 2010).

The chloride content measured in the feed tank and after decantation tank is shown in Figure 1c. This parameter varied significantly and had a coefficient of variation ranging from 30 to 56%. The analyses of chloride content in different stages of the treatment station show that the high chloride level was due to the mix of wastewater with the water of the softener. The highest chloride content reached 4118 mg/l. A high chloride content of the wastewater is not recommended because it induces the inhibition of micro-organisms growth and the corrosion of the installation (Maxime, 2002).

The wastewater collected from the upstream of the station presents a COD value varying from 2269 and 4383 mg/l (CV of 24%) (Figure 1d). The high organic concentration of the effluent is essentially due to the presence of couscous seeds during washing water process. The COD value measured downstream of the
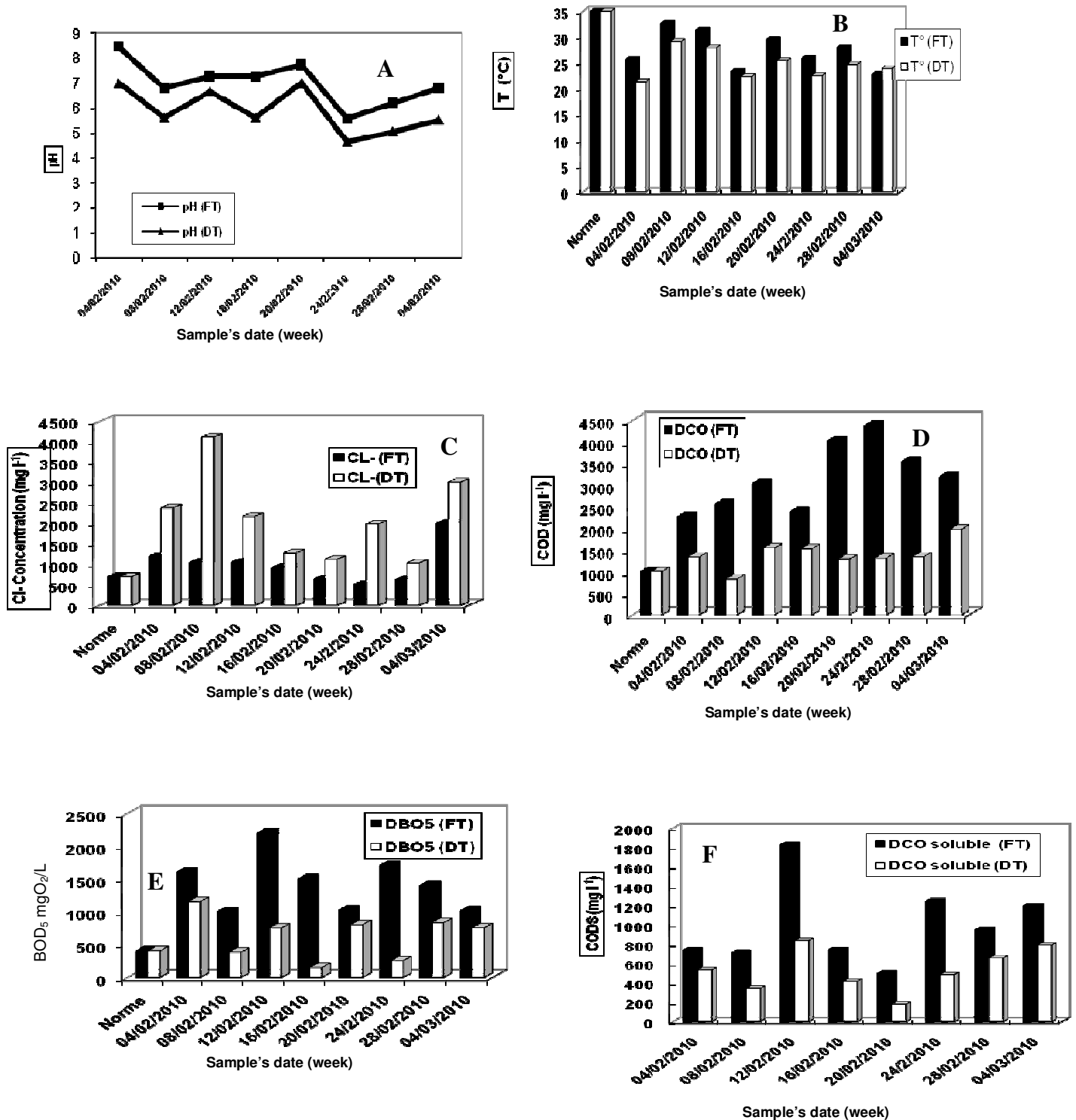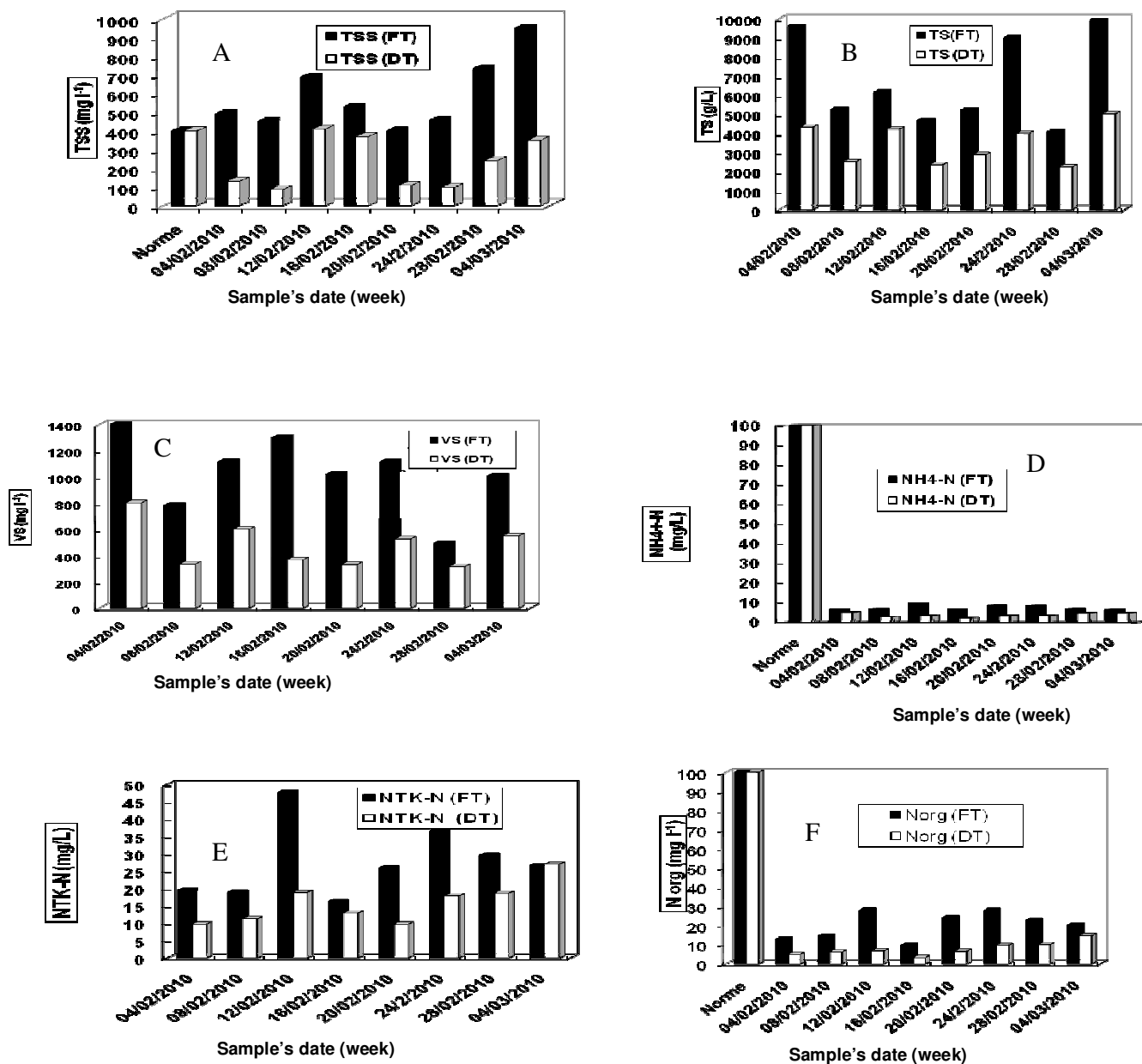
Figure 1. Evolutions of physic-chemical parameters measured on the wastewater taken from the feed tank and after the decantation tank during a month. a, pH; b, T; c, Cl; d, COD; e, COD_S; f, BOD_5.

treatment station varied from 1340 to 1973 mg/l indicating that the organic charge decreased. This value is not conformed to legislative values. The mean values of COD soluble measured on the feed tank and decantation tank were respectively about 988.63 ± 396 mg/l and 643.84 ± 224 mg/l with a CODs/COD ratio of 0.27 (Figure 1e). The

Figure 2. Evolutions of physic-chemical parameters measured on the wastewater taken from the feed tank and after the decantation tank during a month. a, TSS; b, TS; c, VS; d, NH_4^+-N; e, N_{org}-N; f, NTK-N.

high value of the COD was due to TSS present in the wastewaters.

The BOD_5 value measured on the downstream of the treatment plant (Figure 1f) was about 633 ± 340 mg O_2/l. The BOD_5/COD ratio was relatively constant and equal to 0.6 ± 0.1. This value is sharply lower than 5, which indicate the biodegradable character of the effluent (Khannous, 2005).

Figure 2 shows the variations of the parameters TSS (Figure 2a), TS (Figure 2b), VS (Figure 2c), N_{org} (Figure 2d) NH_4^+-N (Figure 2e) and TKN (Figure 2f) measured on feed tank and decantation tank.

The TSS measured on feed tank was about 810 ± 190 mg/l with a CV of 24% and reached a value of 86 mg/l after the treatment station due to the preliminary treatments (screen, sand removal, etc). The TS values ranged from 6500 ± 2269 mg/l to 3590 ± 1070 mg/l, upstream and downstream of the station, respectively (Figure 2b). The VS (Figure 2c) was about 970 ± 270 mg/l upstream of the station and 475 ± 135 mg/l downstream of the station indicating that the TS has an organic character. The last observation was confirmed by the analysis of starch content and the dry matter at the upstream and downstream of the station (6.02 ± 0.21 and

Table 3. Characterization of industrial wastewaters.

Industrial wastewater	Average value	CV (%)
pH	6.81±0.834	12.20
COD_s (mg/l)	988.63±396	40.00
COD_t (mg/l)	3176.49±770	24.0
BOD_5 (mg/l)	633±340	53.70
TSS (mg/l)	810±190	23.45
TS (mg/l)	6500±2269	34.90
VS (mg/l)	970±270	27.83
N-TKN (mg/l)	30±9	30.00
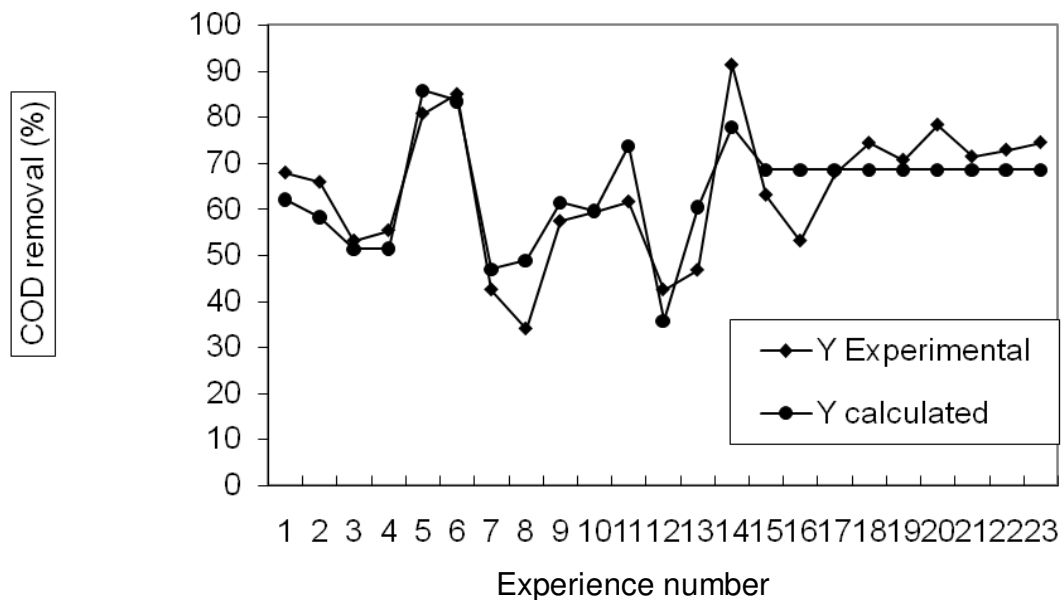
Figure 3. Variation of predicted COD removal versus experimental COD removal.

2.77 ± 0.81, respectively for starch content and 6.5 ± 2.2 and 3.59 ± 1 mg/l for dry matter).

The wastewater presents a low value of total nitrogen (30 ±9 mg/l) and ammoniacal nitrogen (7 ± 1 mg/l) at the upstream of the station. The total nitrogen measured out of the station was about 23.57 ± 5 mg/l (Figure 2f) and is represented essentially by organic nitrogen (ammoniacal nitrogen is about 10.26 ± 1 mg/l (Figure 2e) and organic nitrogen is about 17.65 ± 3 mg/l (Figure 2d).

The analysis of physicochemical parameters of the treated and untreated effluent showed a significant variability of some parameters (Table 3) and particularly the COD (CV= 24%). The industrial station of wastewater treatment did not function adequately and the coagulation/flocculation process should be optimized.

A preliminary study on the effect of type and dosage of coagulant, flocculant, mixing speed, time, temperature and pH on the coagulation-flocculation process was carried out in order to determine the most critical factors and their regions of interest.

Mathematical model

Table 4 shows the values of model coefficients and the corresponding statistical parameters. Only the co-efficients b0 and b2 corresponding to the flocculant dosage were significant.

ANOVA analysis

Table 5 shows the result of the ANOVA analysis for the model. The model equation adequately describes the response surfaces of COD removal in the interval of investigation. In fact, the R^2 value was high (0.728) and the experimental value of Fisher was superior to critical one. Figure 3 illustrates the adequacy between the predicted and experimental values of COD in the experimental investigated region. The model was validated according to the method of Lack of file showing that the error due to the lack of fit is not significant (Mathieu et al., 2000).

Table 4. Coefficients values and statistical parameters obtained for the model.

Coefficient	Coefficient value	Standard deviation	P value
b0	68.584	3.18	< 0.01
b1	-0.52	2.676	84.9
b2	-11.359	2.688	0.0847
b3	5.240	2.734	7.6
b11	2.783	2.346	25.5
b22	-4.966	2.325	5.1
b33	0.229	2.568	93
b12	1.015	2.868	72.9
b13	0.394	3.437	91
b23	-6.959	3.409	6.1

Table 5. Statistical parameters obtained from the ANOVA test performed for the model.

Source of variation	Sum of square (SS)	Degree of freedom (ddl)	Average square	Fisher number	Signification	R^2
Regression	3.84007 E +0003	9	4.26675 E+0002	4.1756	0.859***	0.728
Residus	1.43056 E+0003	14	1.02182 E+0002			

Optimization conditions

The overlay plot was generated by superimposing the contours for the various response surfaces. By defining the desired limits of the COD removal, the shaded portion of the overlay plot defined the permissible values of the dependent variables as shown in Figure 4.

The optimum values of selected variables were obtained by solving the regression equations by using the desirable function available in NemrodW®. The optimum values of the tested variables were as follows; coagulant dosage = 49 mg/l, flocculant dosage = 24 mg/l^{-1} and pH 10.6, while the response predicted was COD removal = 93%.

A verification of the results using the set of optimized parameters was accomplished by performing the experiments incorporating the optimized variables. The experiments were conducted in triplicate. The average COD removal value obtained through the experiment was 93%. These experimental findings were in close agreement with the model prediction.

In order to take into account the variability of the organic concentration of the effluent, the CCD was repeated thrice by using a wastewater having initially different organic concentration (530, 1775 and 2722 mg/l). The obtained optimal conditions are presented in Table 6.

It can be noticed that whatever the initial organic concentration of the waste water was, the optimal coagulant dosage was constant (about 50 mg/l), whereas the optimal flocculant dosage varied linearly (from 24 to 50 mg/l for initial COD varying from 529 to 2722 mg/l), (Figure 5) and should be determined according to the initial organic concentration; the optimal pH value was about 11.0.

## Reuse of the treated wastewater in irrigation

Germination and irrigation tests

Figure 6 shows the results of the germination tests performed on alfalfa (*M. sativa*) and chickpea (*C. arietinum*) by using treated wastewater according to the optimal conditions, untreated water and tap water for irrigation. Tap water was taken as the control assay. The results show that both treated and untreated water allowed a high germination index when compared with the control. The treated wastewater allowed an increase of the index of 82.77 and 7% for alfalfa and chickpea, respectively. The use of treated and untreated wastewater on the irrigation test shows also that they improve the vegetative development of the stem height when compared with the control (Figure 7). Consequently, the treated and untreated wastewater of pasta industry could be recycled in defined conditions in the improvement
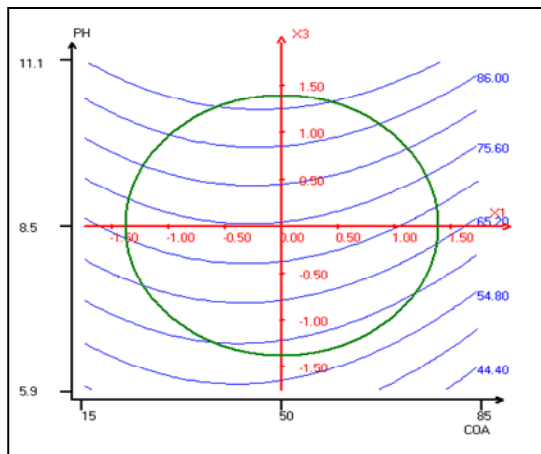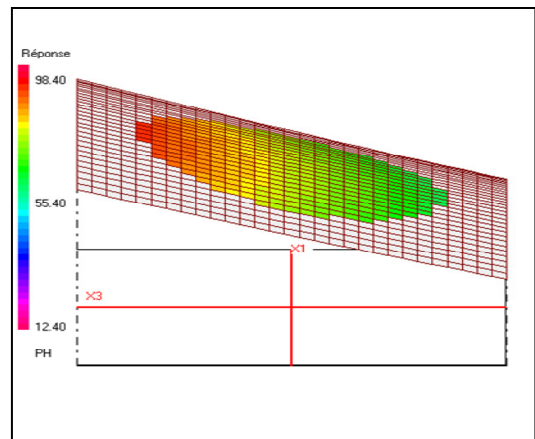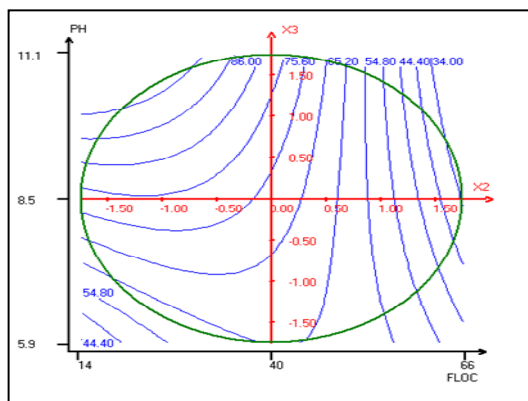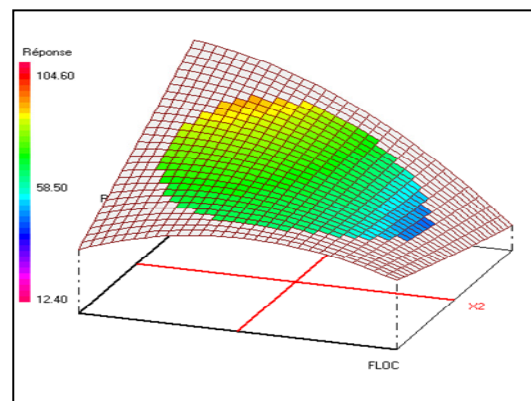
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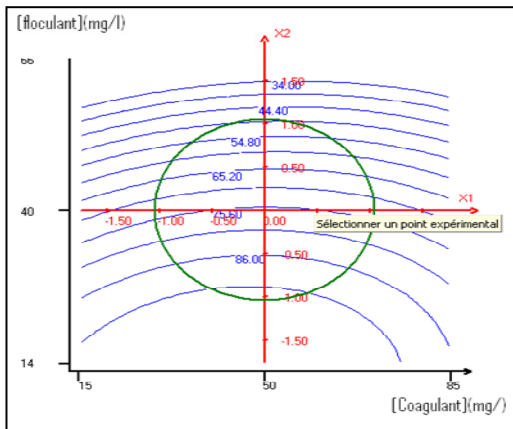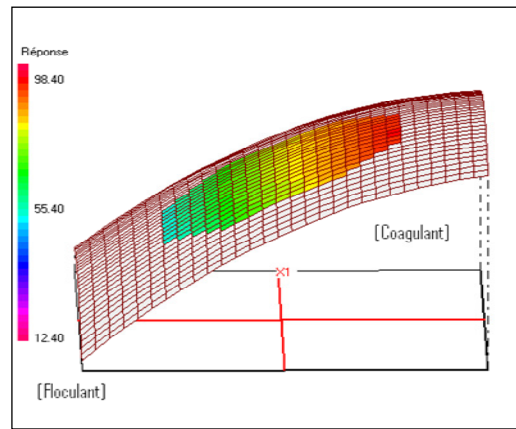
(1)   (2)

B

(2)   (1)

C

(1)   (2)

Figure 4. Variation of response Y (COD removal). A, Effect of coagulant and flocculant dosage; B, effect of coagulant dosage and pH; C, effect of flocculant dosage and pH; (1) isoresponse curve; 2, 3D design expert plot.

Table 6. Optimal conditions of coagulation-flocculation obtained for the different initial organic concentration.

Initial organic concentration	COD_{FT} (mg/l)	Coagulant (mg/l)	Flocculant (mg/l)	pH	COD removal (%)
Experimental design - Charge I	529.6	49	24	10.6	93.04
Experimental design - Charge II	1775	50	34	11	86 .79
Experimental design -Charge III	2722	50	40	11	77.78
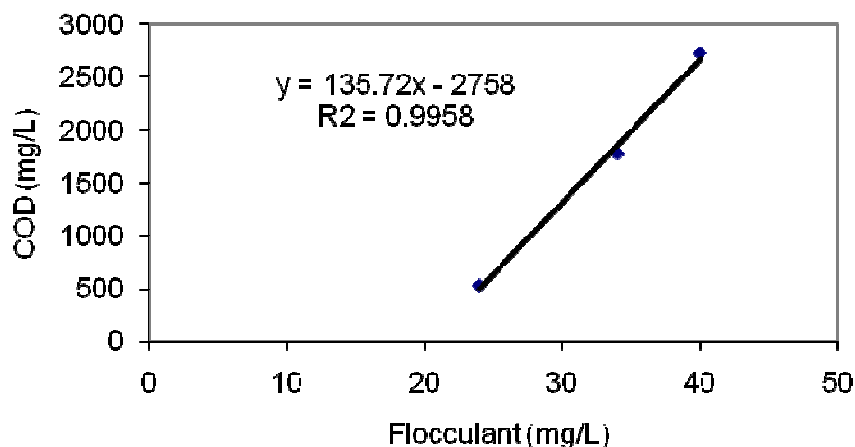
Figure 5. Linear variation of flocculant dosage versus initial COD.
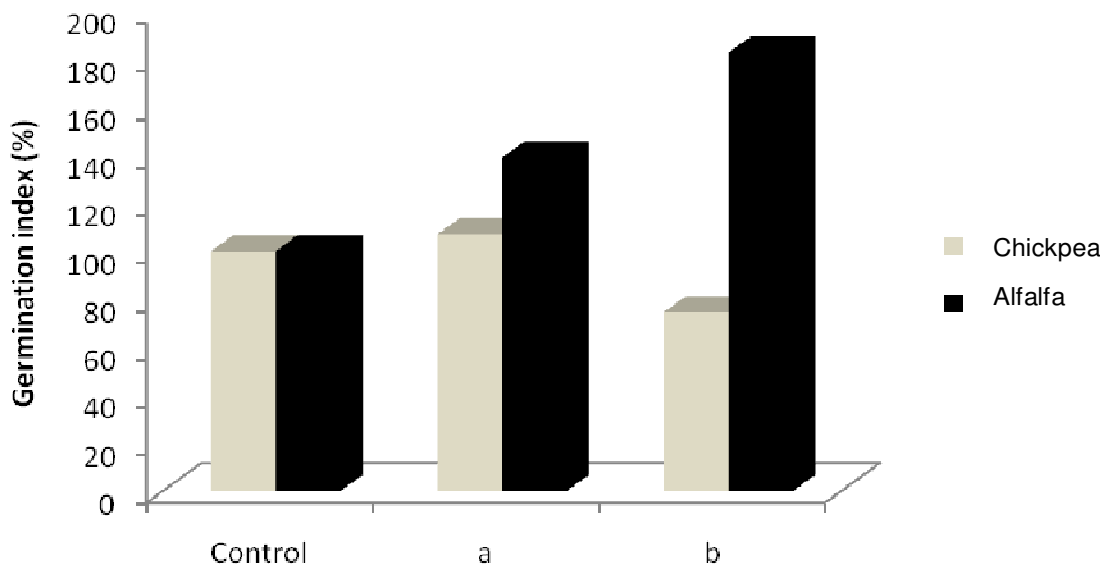
Figure 6. Germination index percentage of chickpea and alfalfa seeds determined on tap water as control, treated wastewaters (a) and untreated wastewaters (b).

of efficiency of some crops and this provides energy conservation. Nevertheless, it is necessary to control the microbial contamination.

Conclusion

Optimization of coagulation-flocculation process with respect to COD removal for the treatment of couscous and pasta industry wastewater was investigated. Response surface methodology using CCD was applied to determine the optimum operating conditions for maximum COD removal. The CDD was in triplicate by using different wastewater organic concentrations. The coagulant dosage and pH were both not significant in terms of higher COD removal yield, whereas flocculant
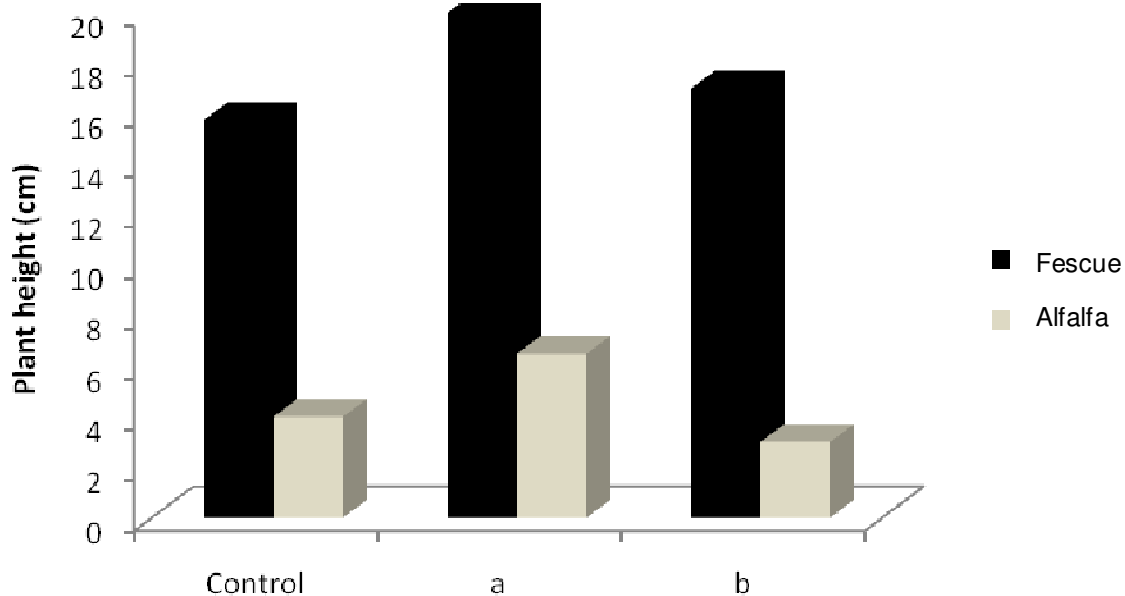
Figure 7. Plant growth of fescue and alfalfa after 30 days irrigation with tap water as control, treated wastewaters (a) and untreated wastewaters (b).

dosage is an important factor influencing COD removal. The reduced quadratic models developed using RSM for COD recovery can be used for prediction within the ranges of the factors investigated (R^2= 0.728). By applying RSM, the optimum region for the coagulation-flocculation process operation is located. The optimum conditions of coagulant dosage and pH were constant whatever the initial organic concentration of the effluent was (coagulant dosage = 50 mg/l and pH = 11), whereas the flocculant dosage increased linearly from 23 to 50 mg/l with the increase of the initial organic concentration. These optimal conditions allowed a COD removal varying from 78 to 93%. Irrigation and germination tests were carried out for alfalfa and chickpea by using treated and untreated wastewater. Interesting, the results were found for alfalfa and chickpea which encourage testing the recycle of these effluents in culture irrigation.

Abbreviation

ANOVA, Analysis of variance; **BOD$_5$**, biological oxygen demand; **COD$_s$**, chemical oxygen demand soluble; **COD$_t$**, chemical oxygen demand total; **NH$_4^+$-N**, ammoniacal nitrogen; **Norg-N**, organic nitrogen; **P$_t$**, total phosphorus; **T**, temperature; **TSS**, total suspended solid; **TS**, total solid; **TKN-N**, total Kjeldhal nitrogen; **VS**, volatile solid.

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