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BIOLOGICAL SULFATE REMOVAL FROM TANNERY WASTEWATER IN A TWO-STAGE ANAEROBIC TREATMENT

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Abstract—In a long-term study, the process of biological sulfate reduction in anaerobic two-stage pilot plants treating tannery wastewater was investigated with the objective to reduce most of the sulfate in the first stage. Influence of quality and quantity of wastewater on sulfate removal in both stages of the pilot plant was tested simultaneously (multiple regression). Origin of the wastewater, chromium, chloride, sulfide and COD in the influent showed no significant effect on desulfurization. Feed flow and the concentration of sulfate in the influent, however, significantly affected sulfate removal. In the first stage, desulfurization increased with higher feed flow but the desulfurization then decreased in the second stage. The concentration of sulfate in the influent had a significant influence on the desulfurization in both stages of the pilot plants. The removal of sulfate in the first stage was approximately 30%, whereas in the second stage the desulfurization decreased with higher concentrations of sulfate in the influent.

Operational parameters were adjusted in order to restrict the biological sulfate reduction to the first stage. The statistical method employed was analysis of variance. Compared to pH 5 or 6 in the influent, a pH of 7 most increased biological sulfate reduction in the first stage. No significant influence on COD removal or volume of gas were observed. For three pilot plants operated parallel to each other, no significant difference in desulfurization was noticed. Copyright © 1996 Elsevier Science Ltd

Key words—tannery wastewater, two-stage anaerobic treatment, sulfate removal, multiple regression, analysis of variance

INTRODUCTION

Tannery wastewater is characterized by a high load of organic matter originating from the hides as well as from chemicals added during the tanning process. Primarily due to the components of the wastewater, it is difficult to treat (Bailey *et al.*, 1984). In addition to a high COD the wastewater shows high concentrations of sulfate, sulfide, chloride and—for a chromium tannery—chromium also. These components may inhibit the anaerobic process (McCarty and McKinney, 1961; Macchi *et al.*, 1991; Bailey *et al.*, 1982). Full tanners using chromium as a tanning agent show variation in quality and concentration of wastewater as well (Kabdasli *et al.*, 1993; Szpyrkowicz *et al.*, 1991; Talinli, 1994). Such variation in concentration even occurs when the wastewater is taken from an equalizing tank at a tannery (Table 1). This is due to charge operation, the diversity of raw material, and the tanning process.

For reasons of energetic economy and reduction of the surplus sludge, anaerobic treatment was selected to transform the bulk of the organic load into biogas during the pretreatment of tannery wastewater. In the first anaerobic stage, the sulfate in the wastewater can be reduced to sulfide by desulfurization (Mudrack and Kunst, 1982; Verink, 1988), which is expedient. There is nutrient competition between sulfate-reducing bacteria (SRB) and methane bacteria, with the SRB prevailing because they achieve a higher energy profit (Verink, 1988). In order to minimize this competition the desulfurization process should take place at the first acidifying stage. SRB tolerate sulfide up to concentrations of 2 g l^{-1} according to Neumann (1977), whereas Widdel and Pfennig (1981) found species that changed their appearance with a higher concentration of sulfide.

Up to now it has not been possible to avoid biological sulfate removal. When Hilton and Archer (1988) added Na_2MoO_4 for inhibition of sulfate-reducing bacteria, the methane production decreased as a consequence.

One purpose of the experiments was to find the most effective pH value in the influent for all processes—acidification, desulfurization, and methane production. Effectiveness was established with reference to gas production and COD removal of the entire pilot plant. Another purpose was to

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ascertain identical sulfate removal in the three pilot plants.

The influence of the quality of wastewater, the quantity of feed flow, and the origin (tannery) on sulfate removal were tested simultaneously (multiple regression). After that operation, the measured values of sulfate removal were adjusted by means of multiple regression referring to the mean value of the significant confounder. Analysis of variance was finally used to test, among other things, the similarity of the pilot plants and the influence of the pH value on sulfate removal.

MATERIAL AND METHODS

Tanneries

In the investigations, wastewater of three tanneries (A, B and C) was used. The objective was to evaluate the results for anaerobic treatment of wastewater from different tanneries. By means of multiple regression, it was possible to confirm a possible dependence of significant differences in sulfate reduction on the origin. The results for COD removal and gas production are reported in Genschow (1994).

Wastewater

The wastewater was taken from three different chromium tanneries. It was drawn once a week or once in 14 days from an equalization tank, with a retention time of 48 h (A and C), and 12 h (B) at the tannery. Even with composite samples taken at 2-h intervals, there was great variation of concentration in the wastewater. Tannery wastewater was used in undiluted form.

Pilot plant

Figure 1 shows the pilot plants for anaerobic treatment. Three two-stage pilot plants were operated parallel to each other. The anaerobic plant consists of a stirred tank and a suspended fixed film bed reactor in upflow-operation filled with Rasshig-rings (7.9 l). The fixed film bed reactor was regulated by a thermostatically controlled water reservoir to a temperature of 34°C. The anaerobic plant was supplied discontinuously four times in 24 h. Separation of the effluent and the gas produced was effected by a siphon that was placed in front of the effluent tank. The biogas was collected in volume-calibrated bags. The feed flow varied between 8 and 35.5 l week^{-1} for tanneries A, B and C with a mean value of 21.9 l week^{-1} and a standard deviation of 6.4. Mean value for total detention time was 3.5 days. By means of analysis of variance the plants were compared. The results for COD removal and gas production are given elsewhere (Genschow, 1994).

Analyses

Analyses of chemical oxygen demand (COD), chloride (Cl^-), and chromium (Cr_{tot}) were carried out according to DIN methods. Analyses of sulfide (S^{2-}) and sulfate (SO_4^{2-}) were performed photometrically, with composite samples being taken each day for 5 days. Samples for COD were acidified and stirred for half an hour to decrease sulfide.

Experimental operation

pH adjustment in the influent wastewater. The pH value of the influent was adjusted to 5, 6 and 7 by adding phosphoric acid.

Analyses by multiple statistical methods (gradual analysis of covariance). Confounder—parameter that causes unintentional variance of the dependent variable, for example, quality of wastewater

Dependent values—Y; in this case sulfate differences
Independent values $x_1 \ldots n_r$—here confounder
Regression coefficient—slope of the estimated function
Adjusted values—values adjusted through multiple regression
Treatment—operational parameter for analysis of variance, in this case pilot plant and pH value
Statistical reliability (S)—reliability for the validity of the significant connection

Gradual analysis of covariance

In contrast to standard analysis of covariance, a gradual analysis of variance allows for the additional analysis of the influence of the confounder. In a first step, multiple regression and adjustment were considered. In the second step, the influence of operational parameters was tested. All factors were considered for which significant connection with the dependent values (sulfate differences) have been shown. In the second step, analysis of variance was used. Parameters were the three pilot plants and pH in the influent. If confounder and treatment are independent from each other, the two steps together will correspond to the classic analysis of covariance. In any other case the influence of a treatment would have been obscured through the adjustment of the measured values to the confounder.

In order to check this assumption, analysis of variance

Table 1. Chemical quality of raw wastewater of three tanneries

Parameter	Number*	Min.	Max.	Mean value	Standard deviation
Tannery A					
COD (mg l^{-1})	130	2070	30,500	8200	6190
Sulfide (mg l^{-1})	115	11	1600	280	206
Sulfate (mg l^{-1})	52	67	3400	1000	547
Chloride (mg l^{-1})	39	4100	7400	5200	840
Chromium (mg l^{-1})	67	10	840	94	139
Tannery B					
COD (mg l^{-1})	280	861	16,800	5070	3080
Sulfide (mg l^{-1})	250	0	650	140	155
Sulfate (mg l^{-1})	279	160	3600	1250	846
Chloride (mg l^{-1})	165	0	11,800	4000	3100
Chromium (mg l^{-1})	210	0.4	226	14	32
Tannery C					
COD (mg l^{-1})	57	1690	14,700	2990	2870
Sulfide (mg l^{-1})	48	53	710	280	178
Sulfate (mg l^{-1})	24	323	1260	750	290
Chloride (mg l^{-1})	51	5600	10,500	8400	1970
Chromium (mg l^{-1})	42	0.4	460	55	126

*Each value 5-day composite sample.

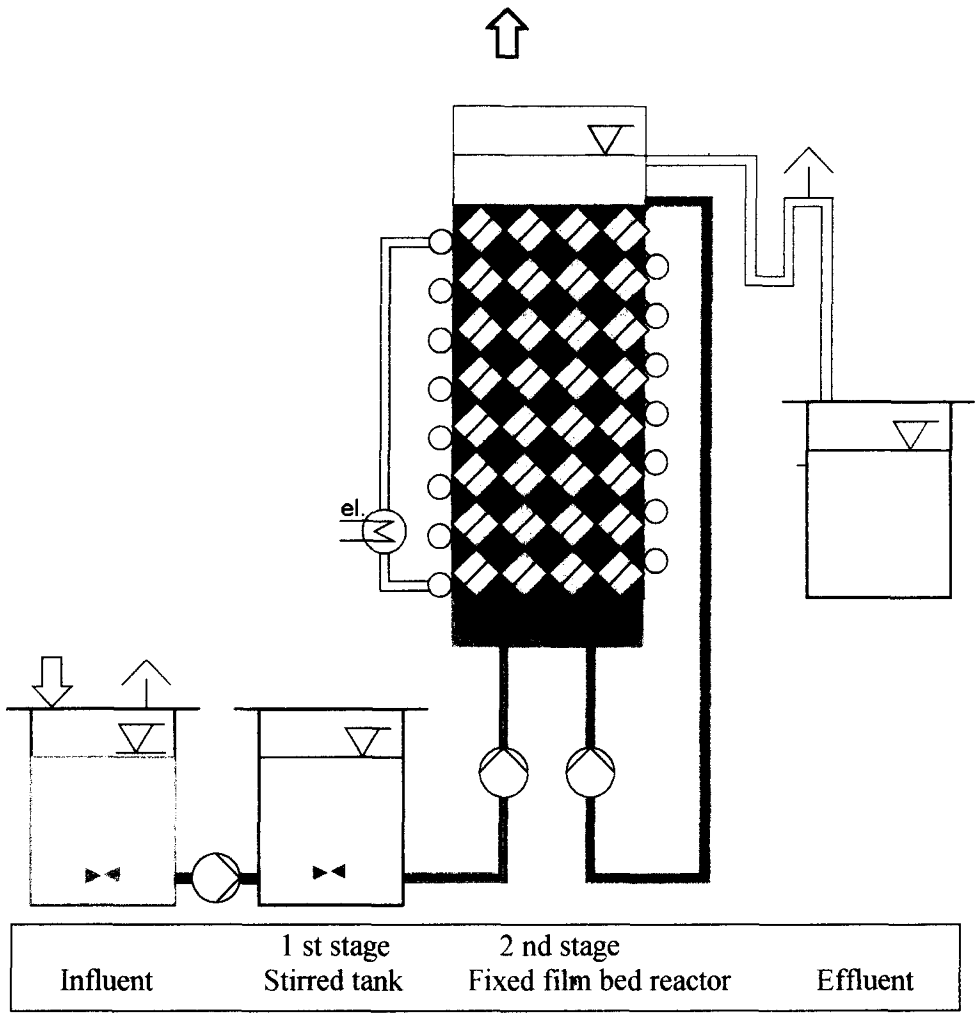

Fig. 1. Treatment scheme of the pilot plant.

can further be carried out with the original measured values. Finally, one can compare the results. This process was carried out in our experiments, but in no case was the influence of a treatment found to be obscured through adjustment. The whole procedure is described in Genschow (1994).

RESULTS AND DISCUSSION

Statistical analysis procedures

The quality of the wastewater from the three tanneries varied highly. This is expressed in the standard variation. The measured values for COD, sulfide, sulfate, chloride and chromium, their number, minimum, maximum, mean value and standard deviation are given in Table 1. Since all values for the three pilot plants are included, there are no representative values for the specific tanneries. In order to decide whether the origin or quality of the wastewater, feed flow, the difference of the pilot plants or pH in the influent were responsible for variations in sulfate removal, a statistical analysis was necessary.

Sulfate reduction was calculated with sulfate differences. Sulfate differences in the first stage are given as results of subtracting first-stage values from influent values. Differences in the second stage were formed by subtracting effluent from first-stage values. These differences were used as dependent values (Y).

The objective was to obtain information about the influence of operational parameters (e.g. pilot plants and pH in the influent) on sulfate removal. It was therefore necessary to eliminate the influence of the origin, the feed flow and the quality of wastewater on the sulfate removal. The procedure was separated into two steps.

- The influence of several parameters on sulfate removal was tested simultaneously in the first step. The result of the procedure is a regression function with a specific slope for every significant confounder.
- The adjustment of the dependent variable is included in the first step. The sulfate differences were converted to adjusted values through the equation of the regression function with the mean value of the significant confounder.

Table 2. Statistical reliability of confounders on desulfurization (multiple regression)

Confounders	Statistical reliability for the influence on desulfurization	
	1st stage	2nd stage
Sulfate (mg l^{-1})	>99.9%	>99.9%
Feed flow (l $week^{-1}$)	96.6%	97.2%
COD (mg l^{-1})*	n.s.	n.s.
Sulfide (mg l^{-1})*	n.s.	n.s.
Chromium (mg l^{-1})*	n.s.	n.s.
Chloride (mg l^{-1})*	n.s.	n.s.
Tannery (A, B, C)	n.s.	n.s.

*In the influent, n.s.: no significance.

(multiple regression). Statistical reliability for potential confounders is given in Table 2. In both stages two significant confounders were found: sulfate concentration in the influent and feed flow. The other components of the wastewater, such as chromium, chloride, sulfide and COD, showed no significant influence.

Multiple regression yields regression functions (1) and (2) which include all significant confounders. Each confounder has its specific slope.

$$1st\ stage:\ Y^* = 3.91\ X_1 + 0.296\ X_2 + a \quad (1)$$

with

Y^*: Estimation for sulfate differences (mg l^{-1}) in the first stage

X_1: Feed flow (l $week^{-1}$) $7 < X_1 < 35.5$ $\bar{x}_2 = 21.9$ $S = 96.6\%$

X_2: Sulfate (mg l^{-1}) $67 < X_2 < 3590$ $\bar{x}_1 = 1180$ $S > 99.9\%$

a: Constant term $a = -85.3$

$$2nd\ stage:\ Y^* = -5.27\ X_1 + 0.186\ X_2 + a \quad (2)$$

with

Y^*: Estimation for sulfate differences (mg l^{-1}) in the second stage

X_1: Feed flow (l $week^{-1}$) $7 < X_1 < 35.5$ $\bar{x}_2 = 21.9$ $S = 97.2\%$

X_2: Sulfate (mg l^{-1}) $67 < X_2 < 3590$ $\bar{x}_1 = 1180$ $S > 99.9\%$

a: Constant term $a = +215$

- In the second step the influence of the operational parameters on sulfate removal could be tested (analysis of variance).

Statistical reliability (S) between 95% and 99% is defined as significant. A highly significant statistical reliability is defined as $S > 99\%$.

Multiple regression

In an anaerobic digester, desulfurization is an inevitable reaction. So far there is no method of stopping sulfate reduction without at the same time inhibiting methane production (Hilton and Archer, 1988). The present goal is thus a preferably complete desulfurization without a negative influence on methane production. In a two-stage anaerobic digester, desulfurization should take place in the first stage, so that the production of methane can proceed unhindered in the second stage.

Statistical methods were carried out separately for the two stages of the pilot plant, since the objective was to increase desulfurization in the first stage, and thus to decrease the sulfate load of the second stage. Countercurrent effects in the two stages were therefore particularly relevant. In fact, differing influences in the respective stages were found.

About 250 values, each a 5-day composite sample of every parameter, were considered simultaneously for their possible influence on sulfate differences

A graphic representation of the simultaneous variation of the two significant confounders is not pertinent here. Only by keeping one confounder constant, a graphic estimation function for the other produces intelligible graphic results. It is advantageous to use the mean value as the constant confounder because the mean value is the best estimation.

The range of feed flow and sulfate in the influent are given in functions (1) and (2). For feed flow the range between 18 and 30 l $week^{-1}$ (detention time $t_R = 4.6$–2.7 d) supplies 75% of the values. 95% of the values lie between 10 and 32 l $week^{-1}$ (detention time $t_R = 8.3$–2.6 d). For sulfate in the influent, 75% of the values were found between 0.1 and 1.5 g l^{-1}, and 95% are given between 0.1 and 2.9 g l^{-1}. As a consequence, values higher than 2.9 g l^{-1} need not be considered here.

Feed flow (X_1)

For showing the dependence of sulfate differences (Y^*) on feed flow only, for sulfate the mean value ($\bar{x}_2$) was chosen as a basis for calculation, while measured values for feed flow were employed in equations (1) and (2). Figure 2 shows this setup.

With higher feed flow the sulfate differences increased in the first stage but decreased in the second. Nevertheless, the total sum of sulfate differences always lies between 600 mg l^{-1} and 680 mg l^{-1}, so that the overall sulfate reduction was always between 55% and 58% with a mean value of 1180 mg l^{-1} of sulfate in the influent.

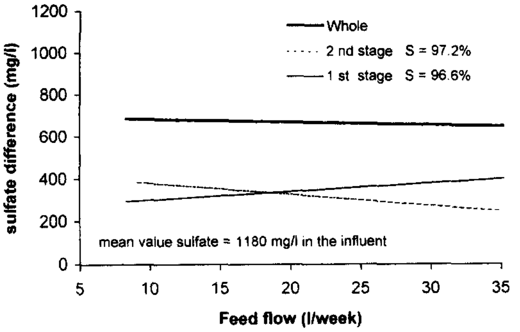

Fig. 2. Influence of feed flow on the sulfate differences [mg l^{-1}] (multiple regression).

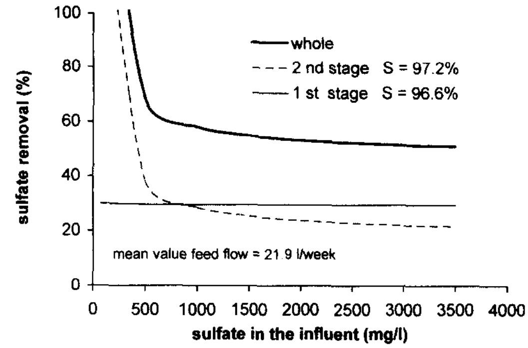

Fig. 4. Influence of sulfate in the influent on the desulfurization [%] (multiple regression).

Few studies are concerned with the influence of the quantity of the feed flow on desulfurization. Verink (1988) assumed that a high overall load of sulfate would decrease the removal rate of sulfate reduction, but attributed this result primarily to COD concentration.

The feed flow showed a significant influence on sulfate removal, whereas no such impact was noticeable for COD removal (Genschow, 1994). This may be related to the fact that the flow had a contrary influence on sulfate reduction in the two stages. The sums of sulfate differences of the two stages were roughly the same, so that COD removal caused by sulfate reduction through desulfurization was almost identical for different feed flow.

Compared to the sulfate concentration in the influent (X_2), the quantity of feed flow showed a less marked effect on sulfate reduction. Although the feed flow covered a wide range, sulfate removal (Y^*) only differed slightly.

Sulfate in the influent (X_2)

For showing the dependence of sulfate differences (Y^*) on sulfate concentration in the influent, the same procedure which had been used for feed flow was applied (Fig. 3). In both stages, higher sulfate concentrations in the influent corresponded to higher sulfate differences.

For concentrations below 1000 mg l^{-1} in the influent, calculations of sulfate differences resulted in lower values for the first stage than for the second stage. For higher concentrations, however, the estimation of sulfate differences in the first stage were higher than for the methane stage.

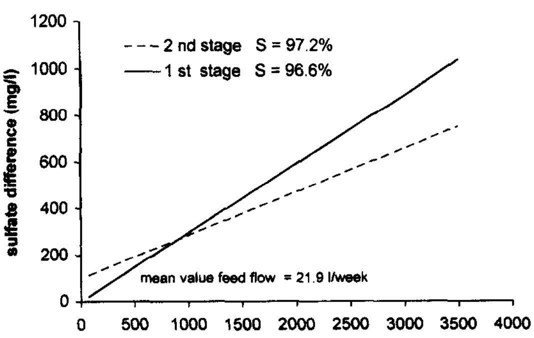

Fig. 3. Influence of sulfate in the influent on the sulfate differences [mg l^{-1}] (multiple regression).

Sulfate reduction in percent was determined by equations (1) and (2), with the value of sulfate in the influent (X_2) also serving as reference value for the second stage. This setup is given in Fig. 4. For the first stage, reduction remained constant at about 30%. Sulfate reduction in the second stage, however, decreased from 70% at low sulfate concentrations to 20% at high sulfate concentrations. When sulfate concentrations were low (<200 mg l^{-1}), the entire sulfate was reduced in the two stages, while only half of the sulfate was reduced in the pilot plant at high concentrations (sulfate >2000 mg l^{-1}).

Tanneries

No significant effect of the origin of the wastewater on desulfurization was found (Table 2), so that the results of desulfurization can be applied to other tannery wastewaters.

COD removal, however, was significantly influenced by the origin of the wastewater (Genschow, 1994). COD removal may result from three processes: acidification, desulfurization and methane production. Each process is dependent on different bacteria that require special environmental conditions. For the whole anaerobic process there may be a difference in COD removal depending on different tanning processes. These results hint at further substances in the wastewater that are not considered in multiple regression.

Analysis of variance

Pilot plants. Three similar pilot plants were operated parallel to each other. The aim of the study was to show that, under identical conditions, the plants reduced the same amount of sulfate (analysis of variance). In Table 3 the results for sulfate removal, COD removal and gas production are given. COD removal in the pilot plants differed significantly for plant 1, $\mu = 59\%$; plant 2, $\mu = 65\%$ and plant 3, $\mu = 69\%$ (with COD $\bar{x} = 5710$ mg l^{-1}). However, no

Table 3. The similarity of the pilot plants for sulfate, COD removal and gas production

Pilot plant		1	2	3
Sulfate removal	Statistical reliability (%)	n.s.	n.s.	n.s.
COD removal	Statistical reliability (%)	99.6	99.6	99.6
	COD differences (mg l^{-1})	3350	3700	3950
	COD removal (%)*	58.7	64.8	69.2
Gas production	Statistical reliability (%)	n.s.	n.s.	n.s.

*Related to mean value COD = 5710 mg l^{-1}. n.s.: no significance.

significant differences were found for gas production or sulfate removal.

pH in the influent. The pH value was varied between 5, 6 and 7 for testing the influence of pH on desulfurization and acidification. The objective was to increase COD removal and gas production.

The results of the influence of pH on sulfate removal, COD removal and gas production are given in Table 4. No significant effect on COD removal and gas production was found, but sulfate removal was significantly influenced. This result is shown in Fig. 5 in which an estimation for all adjusted sulfate differences is given.

A lower pH value of 5 led to a strong overall decrease in sulfate removal in both stages. Desulfurization almost ceased in the first stage, whereas in the second stage higher sulfate differences were found, with sulfate differences twice as high as for pH 7. At pH 7, however, a higher removal of sulfate in the first stage was followed by decreased removal in the methane reactor.

Both stages showed opposing tendencies. The lower the pH value, the smaller the amount of sulfate reduced in the first stage, and thus the higher the sulfate removal in the second stage. Differences between pH 5 and 6 in the influent were much more striking than between pH 6 and 7.

Hydrolysis needs a slightly acidic milieu. Acidogenic bacteria have their optimum environmental conditions at a pH of 5–6, but they allow for a wide range of pH (Hwang and Brauer, 1987). Methane bacteria require a small range of pH between 6.8 and 7.5 (Mudrack and Kunst, 1991; Temper *et al*., 1986), whereas ideal environmental conditions for SRB lie at pH values between 7.5 and 7.9 (Mudrack and Kunst, 1991) but they tolerate pH values between 6 and 9 (Widdel and Pfennig, 1981). A pH below 6 leads to a massive inhibition of desulfurization (Verink, 1988).

The example of adjusting the pH values showed very clearly the problem of predicting the effects of an experimental operation. Although the desired effect—the improvement of desulfurization in the first stage at pH 7—was achieved, increased removal of COD and a higher gas production were not accomplished. This may, however, also be attributed to decreased acidification at higher pH values which also neutralized the positive effect of improved desulfurization in the first stage.

The pH value should therefore be adjusted to 7. One may thus reduce costs and at the same time avoid a further increase in salinity.

CONCLUSIONS

Anaerobic treatment of tannery wastewater containing a high load of sulfate is possible. Biological sulfate removal was determined with statistical methods. Sulfate removal was significantly influenced by the feed flow, the detention time, and by the concentration of sulfate in the influent. The influence of other confounders observed simultaneously—the origin (tannery A, B or C), chromium, chloride, sulfide and COD in the influent—was not significant.

The feed flow covered a wide range, but overall sulfate reduction remained between 55 and 58% (related to a mean value of sulfate in the influent = 1180 mg l^{-1}) and differed slightly for both stages.

Independent of the sulfate concentration in the influent, 30% of the sulfate was reduced in the first stage. In the second stage, however, the percentage of desulfurization decreased with higher concentrations of sulfate in the influent.

A pH of 7 led to a noticeable decrease in sulfate reduction in the second stage. It was not possible, however, to restrict desulfurization to the first stage. The objective of increasing the overall COD removal or gas production was not accomplished. Since the

Table 4. The influence of pH on sulfate, COD removal and gas production

pH in the influent		5	6	7
Sulfate removal	Statistical reliability (%)	>99.9	>99.9	>99.9
1st stage	SO_4^{2-} differences (mg l^{-1})	54	445	528
	SO_4^{2-} removal (%)	5	38	45
Sulfate removal	Statistical reliability (%)	>99.9	>99.9	>99.9
2nd stage	SO_4^{2-} differences (mg l^{-1})	503	288	235
	SO_4^{2-} removal (%)	43	24	20
COD removal	Statistical reliability (%)	n.s.	n.s.	n.s.
Gas production	Statistical reliability (%)	n.s.	n.s.	n.s.

*Related to mean values of sulfate = 1180 mg l^{-1}. n.s.: no significance.

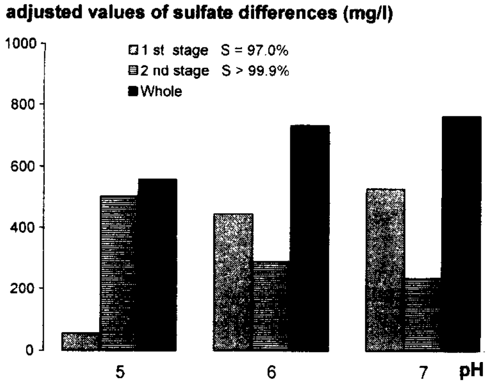

Fig. 5. Influence of pH in the influent on the sulfate differences [mg l^{-1}] (adjusted values, analysis of variance).

pH of tannery wastewater normally lies between 8 and 11, an adjustment to pH 7 produces a minimum of salinity and lowers costs.

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