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Investigation of batch anaerobic digestion and membrane filtration efficiency for the treatment of unhairing wastewater

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ABSTRACT

Abstract: *The objective of this study was to evaluate the efficiencies of the anaerobic batch digestion test and the membrane filtration process in treating unhairing wastewater from tanneries.*
During the anaerobic test, the higher initial organic load decreased the biogas yield. With an initial organic matter load of 5 g COD/L, the biogas yield was of 367 mL/g COD introduced, compared to the 250 mL/g COD introduced when the initial organic matter load was 2.7 g COD/ L. Microfiltration (MF) of unhairing wastewater using a 0.2 µm pore-size membrane resulted in high removal efficiencies, 100% for bacteria and 98.5 and 99% for turbidity in the 1st and 2nd MF series, respectively. These results confirmed the importance of MF for the removal of suspended solids (SS) from unhairing wastewater.
A microtoxicity assay showed only the anaerobic batch digestion test carried out under low initial COD was able to adequately reduce the toxicity of unhairing effluents.

I. Introduction

Production in tanneries can be divided into four main categories: (1) hide and skin storage and beamhouse operations, (2) tanning operations, (3) post-tanning operations and (4) finishing operations [1]. Tanning is one of the most polluting industrial activities in the world. Because the transformation of raw materials into the finished product occurs mainly in water, the wastewater is heavily loaded with pollutants [2]. During tannery operations, two components, the sodium sulfide used in the dehairing stage and the chromium used in the tanning stage, are potential toxicants [3].
The wastewater discharged from tanning, therefore, is highly complex, concentrated, and toxic. These pollutants are expressed in terms of chemical oxygen demand (COD), biochemical oxygen demand (BOD5), total suspended solids (TSS), and total Kjeldahl nitrogen (TKN), as well as sulfur, phosphorus, and chromium compounds [4]. The wastewater, therefore, is characterized by a high load of contaminants that requires considerable treatment before it can be discharged into a body of water.
Many processes have been used in the treatment of tannery wastewaters [5], including biological [6-7], oxidative [8-9] and chemical processes [10]. Tannery wastewaters are characterized by high organic loads, the recalcitrant components of which respond to biological degradation treatment methods [11] , particularly anaerobic digestion. The

use of anaerobic processes to treat tannery wastewater offers significant advantages over aerobic processes. These include low sludge production, low energy requirements and the potential for energy recovery [12-13]. Anaerobic methods, which have received increasing attention in recent years, involve the degradation and stabilization of complex organic matter by a consortium of microorganisms leading to the production of energy-rich biogas that can be used as renewable energy [14]. Anaerobic digestion can be carried out in batch or continuous modes [15]. The choice of wastewater treatment process depends on several factors, including efficiency, cost and environmental capability [16]. Recently, membrane technology has become increasingly attractive for wastewater treatment and recycling. The main advantage of a membrane process is that the concentration and separation of pollutants is achieved without changing the physical state of the effluents and without chemical inputs. Membrane processes, such as microfiltration (MF), nanofiltration (NF), ultrafiltration (UF) and reverse osmosis (RO), have been examined for the treatment of tannery wastes including the recovery of particular chemicals (e.g., chromium) [17-19]. Additionally, UF and MF remove bacteria efficiently, with selectivity dominated by a sieving effect [20-21].

The objective of this research was to assess combined anaerobic batch digestion and the MF approach for the treatment of unhairing the wastewater generated by a Tunisian tannery. The toxicity of the treated and untreated wastewater was evaluated using a microtoxicity test based on changes in bioluminescence of Vibrio fischeri.

II. Materials and Methods

II.1. Raw wastewater

The unhairing-liming bath at a Tunisian tannery was sampled and the effluents filtered through stainless steel sieves (140 µm) to remove hair, pieces of skin and fats. Samples were stored in the dark at 4±1◦C until use.

II.2. Batch anaerobic assay

II.2.1. Inoculum

A mesophilic-anaerobic inoculum was obtained from the municipal solid waste treatment plant Ecoparc I (Barcelona, Spain) and stored in a 10 L plastic container under strict anaerobic conditions until use.

II.2.2. Substrate

Raw unhairing wastewater, from which sulfide had been completely removed by stripping with nitrogen, was used as the substrate in all batch reactors.

II.2.3. Experimental system

Anaerobic batch tests were based on the methods of Field et al. [22] with adaptations based on Angelidaki et al. [23].

The effect of the organic load on the anaerobic process was examined in batch culture experiments using initial COD concentrations of 5 and 2.7 g/L. The controls (blanks) contained only inoculum and tap water and were used to determine biogas production due to depletion of the residual biodegradable organic material and endogenous respiration.

All samples had an initial pH of 7.5; a solution of 15 M sodium bicarbonate was used as buffer. One hundred and fifty mL of wastewater were inoculated with 37 mL of inoculum (VS 5 g/L) and bottles were filled with tap water to a final volume of 0.6 L. The mixture was incubated in 1 L aluminum bottles (traveller SIGG®, Spain), which were purged with N_2 and tightly sealed under the conditions described below. The bottles were incubated under strictly static anaerobic conditions in a temperature-controlled chamber at 37 °C for 40 days until no further biogas production was detected.

All batch assays were carried out in triplicate; results are expressed as the means.

II.3. Microfiltration

II.3.1. Microfiltration tests pilot and membrane

A semi industrial plant (Tech-Sep, France) equipped with a Kerasep membrane was used for MF. The ceramic membrane, 0.4 m long, had 19 channels with an area of 560.10^{-4} m^2 and a mean pore diameter of 0.2 µm. MF was conducted in a crossflow mode with a feed volume of 7 L in each batch.

After optimization, the filtration parameters used were a trans-membrane pressure (TMP) of 1.6 bar and a cross-flow velocity (U) of 2.25 m/s at a constant temperature of 20°C.

II.3.2. Membrane cleaning and laws

After each experiment, the membrane was chemically cleaned. The cleaning procedure was carried out in successive cycles, the number of which was dependent on the membrane and the effluent quality. Each cycle comprised an initial warm water rinse (approx. 50°C) followed by flushing at 80°C with an alkaline solution (e.g. sodium hydroxide), flushing at 60°C with acidic solution (e.g., nitric acid) and a final water rinse until a neutral pH was attained for every stream leaving the system. The cleaning effect was controlled by measuring water flux under standard conditions. The permeate flux (*J*) can be expressed by the resistances-in series model:

$$J = \frac{TMP}{\mu R_t} \quad (1)$$

Where: $TMP = \frac{P_f + P_r}{2} - P_p$ (2)

in which J is the flux ($L/h/m^2$), P is the transmembrane pressure (bar), μ *is* the dynamic viscosity of the feed (Pa.s), and P_f, P_r and P_p are, respectively, the pressure of the feed, the retentate and the permeate (bar). R_t is the total resistance to flow (m^{-1}).

II.4. Analytical methods

Chemical oxygen demand (COD), biological oxygen demand (BOD_5) total organic carbon content (TOC), pH, volatile fatty acids (VFA), suspended solids (SS), total solids (TS), volatile solids (VS), total Kjeldahl nitrogen (TKN), sulfide (S^{2-}), fats, conductivity and turbidity, were determined using standard methods APHA [24]. All results are presented as averages of duplicates with standard deviations.

Quantitative biogas production was followed by measuring the pressure increase in the headspace by means of an SMC (ISE30) Pressure Switch manometer (1 MPa, 5% accuracy) at 37 °C. Biogas production of blank (inoculum only) batches was subtracted from biogas production in each treatment to obtain the biogas production, which was then expressed in standard temperature and pressure conditions.

For the MF process, all aerobic mesophilic bacteria were enumerated on Plat count agar after an aerobic 24 h incubation at 37 °C [25].

A Microtox system obtained from Microbics Corporation was used to test for changes in toxicity of the effluent during remediation. The assay was based on the decrease in the amount of light emitted by the bioluminescent marine bacterium *Vibrio fischeri* on contact with the sample. Toxicity is inversely proportional to the intensity of light emitted after contact with toxic substances. The effective concentration, EC_{50}, is defined as the concentration that produces a 50% light reduction. EC_{50} was measured after 5 min of contact time. A color correction was applied according to the Microtox instructions. To obtain 50% inhibition, the fractions were diluted wherever necessary with purified water containing 2% NaCl. This diluent was also used as a non-toxic control. Effluent toxicity is expressed in units of EC_{50}.

III. Results and discussion

III.1.Characterization of raw unhairing wastewater

The physico-chemical parameters of the raw unhairing effluent are presented in Table 1. All samples had high COD and BOD_5, 30000 and 7600 mg/L, respectively. Unhairing effluent samples had high concentrations of TKN (1100-4500 mg/L), and sulfide (780-3500 mg S^{2-} /L).

Table 1. *Physico- chemical composition of raw unhairing wastewater*

Parameters	Values Range
pH	11 -13
Chemical oxygen demand (mg/L)	9000 - 30000
Biological oxygen demand (mg/L)	2100 - 7600
Suspended solid (mg/L)	4100 - 12000
Total solids (mg/L)	8600 - 28000
Fat (mg/L)	52 - 235
Total Kjeldahl nitrogen (mg/L)	1100 – 4500
Sulfides(mg/L)	780 -3500

III.2. Batch anaerobic tests

III.2.1. Biogas production

The biogas yield of unhairing wastewater during the incubation period (40 days) is shown in Figure 1. An initial lag phase of up to 4 days was observed in all assays. This phase may be due to the presence of toxic compounds in the unhairing wastewater that inhibit microorganism growth. Kroeker et al. [26] demonstrated that effluents can negatively affect microorganisms, shifting the microbial populations or inhibiting bacterial growth. Inhibition of bacterial growth is usually indicated by a decrease in the steady-state rate of biogas production or the accumulation of organic acids [27]. In this work, the small amount of biogas produced indicated that there was microbial inhibition during the incubation period.

Following the lag phase, biogas production increased markedly in all batches, suggesting an increase in microorganism activity. The enrichment began to adapt to the new substrate, and the rate of biogas evolution increased, most likely with an increase in the density and activity of the microbial population.

It can be observed that the biogas yield varied with the organic load applied. Therefore, at the end of incubation the biogas yield of 250 mL/g COD was obtained with an initial organic matter loading of 5 g COD/L. Nevertheless, the biogas yield was 367 mL/g COD for initial organic load of 2.7 g COD/L. These results are in contrast with the finding of Al-Masri [28] and Schievano et al. [29], who reported that higher biogas-yield is obtained at higher initial OLRs.

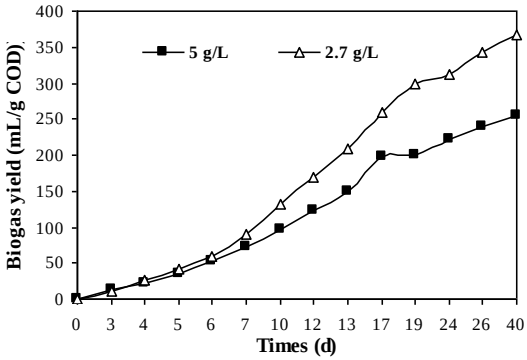

Figure 1. *The biogas yield through time over the 40 day incubation period of unhairing wastewater.*

III.2.2. VFA

The concentration of VFA was determined (Fig. 2). The major product of VFA was acetic acid, most likely due to a nearly neutral pH in the influent. On the basis of thermodynamics, Rodriguez et al. [30] predicted that acetate would be the major product of VFA at a low partial pressure of hydrogen and neutral pH, while at lower pH and/or higher hydrogen pressure, butyrate should be the major product.

The final VFA concentration (1.65 g/L) in the assay with an initial organic load of 5 g COD/L was more important than that in assay with organic load of 2.7 g COD/L (Fig.2). Demirel and Yenigün [31] explained that the rate of VFA production increased in proportion to increasing OLR.

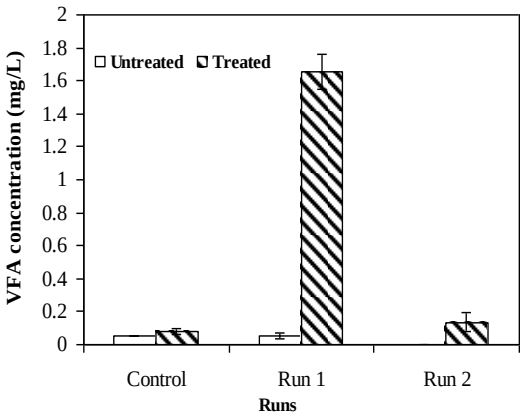

Figure 2. *VFA concentration before and after anaerobic digestion of unhairing wastewater. (Run 1: Initial Organic load=5 g COD/L; Run 2: Initial Organic load=2.7 g COD/L)Vertical bars represent the standard deviation.*

III.3. Microfiltration assay

III.3.1. Permeate flux

During MF assays, the flux declined, reaching final values of 20.38 after 89 minutes in run 1 and 35.7 L/h/m^2 after 72 min in run 2 (Fig. 3). Decreasing the initial SS concentration, therefore, increased permeate flux. Membrane fouling obviously occurred rapidly once the membrane module was put into operation, leading to these decreases. Membrane fouling plays a key role in filtration processes, and several factors may contribute to this effect, including cake formation, adsorptive fouling mechanisms and the blockage of pores [32]. The effect has also been reported by other authors who processed raw wastewater with high organic contents [33-34].

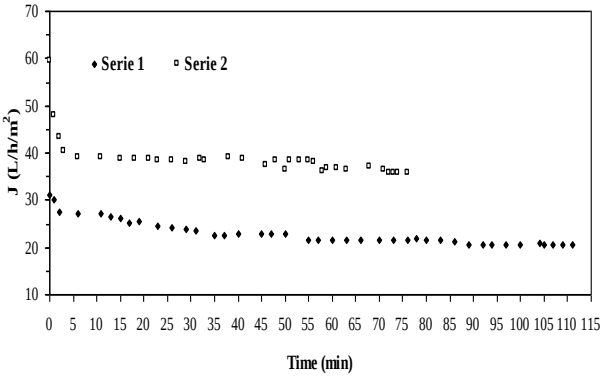

Figure 3. *Effect of initial SS concentration on permeate flux during MF of unhairing wastewater (Series 1: SS = 10.9 g/L; Series 2: SS = 5.1 g/L).*

III.3.2. Water quality results

The efficiency of the selected MF membrane was assessed on the basis of the removal of several pollutants present in the unhairing wastewater.

III.3.2.1. Non-specific parameters and microbial pollution

Conductivity and pH were not significantly affected by MF. However, there was a noticeable decrease in suspended solids after MF. SS removal efficiencies were 92.46 and 93.64 % during the 1st and the 2nd runs, respectively. Thus the MF membrane clearly presented a complete barrier to high molecular weight particles and particle-related load, whilst maintaining some permeability to dissolved compounds. Suspended solids, therefore, were in negligible quantities in the permeate, and turbidity removal were 98.5 % and 99 % at the end of the 1st and the 2nd series of MF, respectively. The total numbers of bacterial colonies found in the influent were 1727 and 586 CFU before the 1st and the 2nd assays, respectively. After the two MF assays, bacteria were completely removed (100 %). With an MF pore diameter of 0.2 µm), all bacteria should be removed from suspension.

These results support those of our previous study [35], which showed a complete elimination of bacteria from the MF permeate of treated unhairing

wastewater following treatment in an activated sludge system.

III.3.2.2. Parameters indicating organic pollution

During MF assays, there was a decrease in the organic matter content of permeate (Figure 4). COD removals were 45.7 and 19.9 % after the 1st and the 2nd series of MF, respectively. After MF assays, BOD_5 removal efficiencies were 53 and 22.7 % for series 1 and 2, respectively. These results indicate that increasing the initial SS concentration led to decreases in COD and BOD_5 after the MF assays. These removal efficiencies can vary based on the type of organic fraction in the suspended form [36].

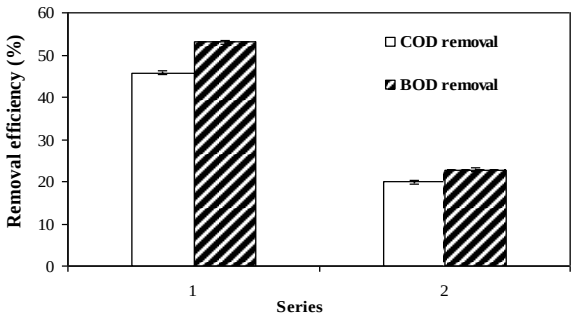

Figure 4. *COD concentration and BOD and COD removal efficiency during MF assays (Series 1: SS = 10.9 g/L; Series 2: SS = 5.1 g/L).*

III.4. Microtoxicity assay

The wastewater samples treated in this work were highly polluted with organic compounds, as indicated by high COD, BOD_5 and TKN concentrations. All measured values exceeded the limits set for tannery wastewaters discharging into receiving streams (Table 1). Therefore, raw unhairing wastewater, with an EC_{50} of 0.5 %, is toxic. Similar results were reported by Vidal et al. [37] who found that the untreated unhairing wastewater to be toxic, as indicated by exposure of *Daphnia magna* and *Daphnia pulex* to the wastewater for 24 or 48 hours.

Effluent from run 1 had an EC_{50} of 1 %; hence, both the untreated and anaerobic treated samples from run 1 were toxic. It is possible that the toxicity was caused by the organic load remaining in the treated unhairing wastewater. Alternatively, the toxicity could be due to the presence of inorganic ions, such as ammonium [37-38], in the effluent.

At the end of the incubation for run 2, the EC_{50} of the treated effluent, 6%, was not toxic. In this case the reduction in toxicity was not attributed to the efficiency of the anaerobic treatment, but rather to the low initial organic load used, which led to low remaining organic matter concentrations after anaerobic digestion.

MF also had little effect on the microtoxicity of unhairing wastewater because the EC_{50} of samples from series 1 and 2 were 1 and 2 %, respectively, after 5 min of exposure of *V. fischeri*. Thus the MF process will not contribute to detoxifying unhairing effluents.

These results showed that the toxicity in these assays was due to the wastewater characteristics and to the type of treatment process. These results agree with those of Klinkow et al. [39], who found that toxicity to *V. fischeri* changed depending on the type of effluent treatment.

The microtoxicity assays showed the need to use a combination of biological and physico-chemical processes to treat unhairing wastewaters from leather factories.

IV. Conclusion

During the batch anaerobic digestion of unhairing effluent, the cumulative volume of biogas and the VFA concentration, which had acetic acid as its major component, increased in proportion to the initial organic load in the system. The highest biogas yield from unhairing wastewater was achieved with the low organic load.

Overall, the MF of unhairing wastewater leads to an excellent reduction in turbidity, removal of suspended solids and bacteria from the effluent. The permeate flux and removal of COD and BOD_5 increased with decreasing initial SS.

The microtoxicity assay suggested that neither anaerobic digestion nor the MF processes were able to reduce toxicity of the unhairing wastewater to an acceptable level, except when a low initial organic load was applied.

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