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Water and detergent recovery from rinsing water in an industrial environment

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ABSTRACT

Wash water streams coming from rinsing of equipment in a detergent production site is in many cases considered as waste. On site treatment in waste water plants is possible but typically requires advanced oxidation process (AOP) technology which uses chemicals and creates a waste sludge. A new treatment approach, based on nanofiltration, has been demonstrated at industrial scale in a detergent production site in China. Wash water could be split into a concentrate stream and water fraction. The concentrate stream contains most of the valuable surfactants and has a value to recycle. The water fraction can easily be polished by MBR to feed cooling towers. As such, this production site does not discharge any process wash water and recovers all resources out of the rinsing water: both chemicals (as surfactants) as the water.

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1. Introduction

Production facilities of liquid detergents have typically 'making units', where the product is produced into bulk storage tanks, and 'packing departments' where the product is bottled and then packed for distribution to clients. In both departments, wash water is generated during cleaning and sanitization activities. This water is created during the wash-out, which is done to avoid product contamination when switching between product variants, and contains considerable amounts of surfactant which in most cases cannot be discharged into public sewer systems. Similar type of wash water is generated during the hot water sanitization which is done to avoid bacterial contamination of the liquid detergents. This wash water is in general a mixture of clean reverse osmosis (RO) quality water and detergent product and has a COD ranging from 5000 to 30,000 mg/l.

Nowadays, some of these wash water streams are treated on site before being discharged. This typically involves physical–chemical treatment and/or AOP, biological treatment and where needed additional polishing. Other wash water streams are collected and sent for an external treatment by e.g. wet oxidation or incineration with energy recovery. In both cases, the wash water is seen as a wastewater which needs to be treated. Chemicals and energy are added to transform the wash water into 'water which can be discharged' and 'sludge' which is removed as waste. The cost for the AOP chemicals and sludge removal will vary a lot from the location but can be 20 to 40 Euro per m^3 of water treated. Also external incineration will represent a significant cost.

The European E4WATER project has focused to find alternative solutions and approaches. Within this project, the wash water streams were considered as a mixture of water and product. Instead of using traditional wastewater treatment approaches, membrane technology was used to extract this product (detergent) back out of the water and to recycle this. The collected surfactants can be recycled into a variety of applications (eg. lower grade surfactants for car washes, road cleaning, …) and will generate a revenue. Addition of chemicals was avoided as this would contaminate the 'recycled product'. In addition to the recuperation of the product, the water stream was treated in order to meet the legal discharge requirements or, in the ideal case, reuse the water in the process after an additional polishing step.

In order to achieve both product and water recuperation, a two-step approach was followed. In a first step, nanofiltration (NF) has been used to split the wash water into a high and a low concentrated stream. If the concentrated stream contains half of the detergent concentration of the pure product, reuse as lower grade industrial detergent would become possible. In a second step, the permeate of the NF (containing COD, BOD and traces of detergent) was treated within an aerobic membrane bioreactor (MBR) in order to be able to discharge the water or recycle it back

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into the process.

The use of NF for the treatment of detergent containing wastewaters is well-known in literature and mainly applied for the treatment of cleaning-in-place (CIP) rinsing water. According to Kaya et al. [1,2] and Kowalska [3], NF is preferred in the removal of surfactant monomers from wastewater, while micelles can be removed by microfiltration (MF) and ultrafiltration (UF). Archer et al. [4] studied NF for model solutions containing anionic surfactants below the critical micelle concentration. Their study showed that separation performance depends on the properties of the surfactant and the interactions between surfactant and membrane, indicating the importance of the right choice of membrane case-per-case. Kaya et al. [2] investigated three different NF membranes for the treatment of single and mixture surfactant solutions. The optimal membrane was indeed different depending on the composition of the surfactant solution. Furthermore, a fast flux decline was reported in this study caused by the adsorption of the surfactants on both surface and pore walls, showing the importance of fouling in the process.

Since it is well known that the presence of detergents may induce membrane fouling, also other authors focused on this topic. Kaya et al. [1] used a two-step NF process in order to recover the product and reuse the water from a model CIP wastewater. Effects of pH, temperature and transmembrane pressure (TMP) on membrane performance and occurrence of fouling were evaluated. Best results were obtained at pH 5, room temperature and highest TMP (20 bar). A similar two-step approach was followed by Gönder et al. [5]. In the mentioned study, the Taguchi method was used to investigate which parameters affects the flux decline by membrane fouling the most. A pH above the isoelectric point of the membrane, room temperature and lower TMP (12 bar) were defined as optimal. This is also confirmed by Kertész et al. [6], although this group pointed out that there is a trade-off between flux (highest at high temperature) and retention (highest at low temperature) and between flux (highest at high TMP) and gel layer formation (also highest at high TMP, which is negative for the flux). Therefore, also in the present study, research was performed on the best conditions for the discussed wash water.

In the review paper of Suárez et al. [7], membrane technology is described as the most promising method for recovering cleaning agents. They pointed out that most work until now focused on anionic and nonionic surfactants, and that more work is needed on real industrial detergent since they include also other important ingredients. Furthermore, also a lack on larger scale data was observed. As an answer to this, within this paper, all research is performed on real wash water from one of the production lines (so called HDL, containing anionic and nonionic surfactants) of P&G. P&G is one of world's largest non-food consumer goods companies and produces a wide variety of consumer goods products which are liquid in nature (fabric detergents, dish washing liquids, hard surface cleaners, shampoos, …). At the end of the paper also data from a real industrial test case are reported, since all gathered knowledge was applied to compose a treatment train for the wash water of the liquid detergent production site of P&G in China. Due to local legal requirements, wash water from this production site cannot be discharged into the local public sewer system, even if it has been treated. All process related 'wastewater' needs to be recycled back into the process. This can be done by recycling the water back to cooling towers (if COD < 50 mg/l, surfactants < 0.5 mg/l and TDS < 1000 mg/l) or back as process water (if water meets drinking water standards). The E4WATER approach was used to meet this local 'zero liquid discharge' legal requirement by combining tubular NF with MBR and additional polishing via activated carbon and surfactant removal resins. To the best of our knowledge, it is the first time this combination of techniques is demonstrated in an industrial environment.

2. Material and methods

2.1. Nanofiltration

Nanofiltration experiments were performed at lab scale. Tubular membranes from PCI (Polyamide film with a $CaCl_2$ retention of 75%) were chosen for their advantage of lower fouling tendency, superior cleanability and the ease of membrane replacement. The tubular membrane system is based on a 12.5 mm ID × 1.2 m (length) membrane tube, which consists of a supporting tube that is coated by a selective membrane layer at the inside. A tube shows 0.072 m^2 of membrane area and is contained in a module/tube holder, which contain 18 tubes. By this way, a maximal membrane area of 1.37 m^2 is generated. The system can be applied for operating pressures of maximum 64 bar and temperatures of maximum 80 °C.

A multi-cycle filtration was evaluated with a module containing 18 tubes. A clean water flux was measured at conditions described by the supplier before and after each test in order to be able to quantify membrane fouling. Flux was measured and feed and permeate streams were sampled and analyzed at selected permeate recoveries. At the end of each filtration cycle, concentrate and permeate mixtures were sampled and analyzed. The former steps were repeated in 3 cycles with each time fresh feed.

2.2. Membrane Bioreactor tests

For the performance of MBR experiments, a bench-scale aerobic MBR was used in which different membranes can be tested simultaneously. The P&ID of the installation can be found in Fig. 1.

This set-up allows operating 6 separate filtration panels at the same time in the same sludge. The filtration protocols for the three filtration panels can be identical or different. This allows making distinction between membrane effects, and effects caused by operational differences or differences resulting from sludge quality. Operating parameters pH, temperature and TMP are logged continuously. The pH can automatically be adjusted using base and acid dosing pumps and a pH control device. Within this study the chlorinated PE membrane of Kubota was used. The properties of this membrane are summarized in Table 1.

A solution of 5% HDL (=liquid detergent to wash clothes, available on the market) was first treated by Nano Filtration. The feed solution was concentrated until a permeate recovery of 90% was reached. The first part of the trials were done with water at room temperature. At the end, the Nano filtration was done at higher temperature ($T \sim 40$ °C). Characteristics of this NF permeate are summarized in Table 2. This permeate was further treated into the MBR pilot.

During the experiments, different analyses were performed on influent, effluent and sludge on a frequency of two or three times each week. A summary is made in Table 3. For all the analytical work, validated methods (Standard methods for Water and Wastewater) were used. Flow, sludge load, hydraulic retention time (HRT), flux and transmembrane pressure (TMP) were monitored/calculated on a daily base.

2.3. Validation of treatment train in industrial environment

Based on the lab-results, a treatment train containing NF, MBR and a polishing step was selected for the treatment of rinsing water from the P&G production site in China. This site is producing HDL which is liquid detergent containing anionic and nonionic surfactants. This treatment train is schematically presented in Fig. 2.

Wash water is collected in the production department (0) and pumped to the E4WATER treatment plant buffer tank (1). A

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