Treatment of packaging board whitewater in anaerobic/aerobic biokidney

T. Alexandersson* and Å. Malmqvist**

*Industrial Electrical Engineering and Automation, Lund University, Box 118, SE–221 00 Lund, Sweden
**Anox AB, Klosterlängsvägen 11A, SE–226 47 Lund, Sweden

Abstract Whitewater from production of packaging board was treated in a combined anaerobic/aerobic biokidney, both in laboratory scale and pilot plant experiments. Both the laboratory experiments and the pilot plant trial demonstrate that a combined anaerobic/aerobic process is suitable for treating whitewater from a packaging mill. It is also possible to operate the process at the prevailing whitewater temperature. In the laboratory under mesophilic conditions the maximal organic load was 12 kg COD/m³d on the anaerobic reactor and 6.7 kg COD/m³d on the aerobic reactor. This gave a hydraulic retention time, HRT, in the anaerobic reactor of 10 hours and 2 hours in the aerobic reactor. The reduction of COD was between 85 and 90% after the first stage and the total reduction was between 88 to 93%. Under thermophilic conditions in the laboratory the organic load was slightly lower than 9.6 COD/m³d and between 10 and 16 COD/m³d, respectively. The HRT was 16.5 and 3.4 hours and the removal was around 75% after the anaerobic reactor and 87% after the total process. For the pilot plant experiment at a mill the HRT in the anaerobic step varied between 3 and 17 hours and the corresponding organic load between 4 and 44 kg COD/m³d. The HRT in the aerobic step varied between 1 and 6 hours and the organic load between 1.5 and 26 kg COD/m³d. The removal of soluble organic matter was 78% in the anaerobic step and 86% after the combined treatment at the lowest loading level. The removal efficiency at the highest loading level was about 65% in the anaerobic step and 77% after the aerobic step. In the pilot plant trial the removal efficiency was not markedly affected by the variations in whitewater composition that were caused by change of production. The variations, however, made the manual control of the nutrient dosage inadequate and resulted in large variations in effluent nutrient concentration. This demonstrates the need for an automatic nutrient dosage system. The first step towards such a system was to evaluate two different on-line instruments. Both had severe stability problems, which made them unsuitable as parts in a system for control of the nutrient dosage.

Keywords Whitewater; biological treatment; closed system; reuse; pulp and paper; control; nutrients

Introduction

The pulp and paper industries have been, and some still are, large consumers of freshwater. Due to economical and environmental reasons this consumption has decreased over time and today there exist several zero-discharge recycled paper mills (Barton et al., 1996; Habets and Knelissen, 1997) and pulp mills (Edde, 1994). However, closing a water system leads to several different problems, such as increased demand for retention aids, decreased product quality, reduced felt life and corrosion.

There are different solutions to overcome these problems. One method is to reduce the compounds in the whitewater, which the microorganisms feed on. This can be done by an in-mill biological treatment of the whitewater. It is, however, important that such a process is designed for the high temperature that is a result of the closure of the water circuits in order to avoid costly cooling.

There are several different studies of thermophilic biokidneys (Jahren and Rintala, 1997; Malmqvist et al., 1999; Robinson et al., 2001). A combined anaerobic/aerobic biokidney has previously been shown to be an attractive alternative for treatment of whitewater from recycled paper production (Habets and Knelissen, 1997). In order to have an
efficient treatment process nutrients such as nitrogen and phosphorus, which are limited in the whitewater, must be added. Since the treated water is going to be reused, it is very important that the dosing is adequately controlled so that the nutrient levels in the effluent are low. Otherwise bacterial growth in the whitewater system could be promoted.

The possibility to use an anaerobic/aerobic biological process for treating whitewater from a recycled paper mill was evaluated in laboratory scale experiments, both mesophilic and thermophilic. The normal concentration variability in the whitewater is, however, difficult to simulate in the laboratory. Therefore pilot scale trials were set up at a packaging board mill where the stability of a combined anaerobic/aerobic biokidney towards natural variations in whitewater composition was studied. The trial also included testing of two different commercially available on-line instruments in order to evaluate their possibility to be used as parts in a future control system.

Methods

Laboratory process

The laboratory scale process consisted of two sets of one anaerobic fluidised bed reactor (658 ml) followed by one aerobic suspended biofilm carrier reactor (135 ml). One set was operating at 37 °C (mesophilic) and the other at 55°C (thermophilic). Both aerobic reactors were filled to 50% with carrier material (K1). The anaerobic bed consisted of sand (Ø ~ 1 mm) and it was fluidised by recirculation of the reactor liquid from the top to the bottom. The pH in the anaerobic system was maintained around 7 by automatic addition of 1 mol l⁻¹ NaOH. Whitewater and nutrients (NH₄Cl and KH₂PO₄) were pumped to a pH control vessel (volume 90 ml). Anaerobic effluent and gas were led to a separator, where particulate matter was separated from the liquid, which was led to the aerobic reactor. The amount of gas from the separator was measured and solid material was manually removed at regular intervals.

Whitewater for laboratory experiments

The whitewater came from a paper mill, which produces fluting and testliner from recycled waste paper. The whitewater system of the mill is closed and the freshwater consumption in the mill is around 1.4 m³/ton product. The concentration of dissolved COD was 31,000 mg/l and total COD was 32 000 mg/l. Prior to use the whitewater was diluted with tap water in order to reduce the retention time in both reactors. The whitewater concentration for the mesophilic set was 30 vol-% and for the thermophilic set 40 vol-%.

Laboratory scale analysis

Samples were taken of the effluents after both the anaerobic and aerobic stages. Analyses were made on filtered (Whatman GF/A) samples. COD was determined with the Dr Lange test kit LCK 114. Ammonium-nitrogen was analysed with the Dr Lange test kits LCK 303 and 304. Ortho-phosphate-phosphorus was analysed with the Dr Lange test kit LCK 349.

Pilot plant outline

The pilot plant consisted of an anaerobic fluidised bed reactor with a total volume of 36 l and an expanded volume of 20 l. Water was recirculated from the top of the reactor to the bottom, thereby fluidising the sand bed. After the anaerobic stage, the whitewater was treated in an aerobic suspended carrier process with a volume of 13 L, filled to 50% with suspended carrier material (Natrix). The pH in the anaerobic reactor was continuously controlled to a value of 7 by automatic titration of 2 mol l⁻¹ NaOH. Whitewater and nutrients (NH₄Cl and H₃PO₄) were pumped directly to the recirculation loop on the
anaerobic stage in a fixed ratio. This ratio was changed during the trials depending on the amount of nutrients remaining in the effluent from the aerobic stage. The effluent was discharged to the wastewater system of the paper mill.

**Paper mill**
The pilot plant trials were carried out at the Munksjö Lagamill AB, Sweden, a paper mill producing testliner (35000 ton/year) and fluting (20000 ton/year) from recycled raw materials such as old corrugated cardboard and newsprint. The whitewater system of the mill is rather open and the fresh water consumption is around 14 m³/t. For the trials, treated whitewater was taken directly from the flotation unit.

**Start-up and operation of the pilot plant**
At start-up the anaerobic reactor was inoculated with 2 L anaerobic sludge from a municipal digester with a TSS of about 20 g/L. The flow of whitewater to the biological system was adjusted to give an organic load of about 5 kg COD/(m³ d) on the anaerobic reactor. The test series were started when significant gas production and removal of organic matter were observed. The hydraulic flow was then increased stepwise in order to evaluate the treatment efficiency and process stability at different loading levels.

**Sampling procedure and analyses for the pilot plant trials**
Grab samples were taken of the incoming whitewater and on the effluents after both the anaerobic and aerobic stages. Analyses were made on total, settled (30 minutes) and filtered (Whatman GF/A) samples.

Determination of COD, ammonium-nitrogen, ortho-phosphate and total phosphate were made with the same methods used in the laboratory experiments. Total suspended solids (TSS), and volatile suspended solids (VSS), were determined according to standard SS-EN 875. Total nitrogen was determined with kit LCK 238. The filterability was determined as the time it took to filter (Munktell 1003, pore size 10 μm) a defined volume of water under constant vacuum (0.9 bar).

**On-line instruments**
An automatic total oxygen demand (TOD) instrument from Ionics, model 7800 was installed to take samples of the influent whitewater. The instrument was equipped with a 75 μm filtration unit with automatic cleaning of the filter with tapwater. A small volume of filtered sample was automatically injected with nitrogen gas containing a small amount of oxygen, directly into a hot oven (900°C) where the sample was catalytically incinerated. The consumption of oxygen was measured and automatically evaluated against a calibration curve resulting in a TOD value. Besides the TOD instrument an automatic ammonium instrument from Ceric with an ion electrode was also installed to measure the effluent from the anaerobic stage. The sample was mixed with potassium hydroxide to transfer the ammonium to ammonia, which was sensed by the electrode.

**Results**

**Laboratory scale experiments**
*Mesophilic reactor set.* This process was operated for 55 days and during the first 43 days the organic load on the anaerobic reactor was around 8.6 kg COD/(m³ d), see **Figure 1**. The hydraulic retention time, HRT, in the anaerobic reactor was around 13 hours up to the flow increase on day 43 when it decreased to 10 hours. The corresponding values for the aerobic reactor were 2.7 and 2.0 hours. The increased flow led to an increase in the organic load to 12 kg COD/(m³ d). The organic load on the
aerobic reactor was during the first 26 days around 3.7 kg COD/(m³*d) and it increased to a value around 6.7 kg COD/(m³*d) from day 34 to day 55.

The COD reduction was very high in the anaerobic reactor, between 85–90%. The following aerobic step removed some additional COD so the overall reduction became 88–93%. The average COD concentration in the anaerobic effluent from the start to day 25 was around 430 mg/l. The concentration then increased in the effluent due to the removal of sludge from the anaerobic reactor. The anaerobic reduction was again improved after day 40 and remained stable after the load increase at day 43, see Figure 2. The COD removal in the aerobic reactor was stable most of the time with an average effluent COD concentration of 350 mg/l. Although the concentration in the influent to the aerobic reactor between day 30 and day 43 almost was doubled, the aerobic reactor managed to maintain the reduction.

The nutrient consumption in the anaerobic reactor was calculated to 19 (± 5) mg N/g COD$_{reduced}$ and to 2.5 (± 0.7) mg P/g COD$_{reduced}$. An equivalent consumption for the aerobic reactor could not be determined since the main part of the COD was reduced in the anaerobic reactor and consequently, the nutrient consumption in the aerobic reactor was too low to be detected.
**Thermophilic reactors.** The thermophilic reactor set was operated for almost 90 days and at several occasions different problems, like a clogged influent tube, occurred. The organic load on the reactors was to some extent affected by these disturbances, which can be seen in Figure 3. However, the average load on the anaerobic reactor was 9.6 kg COD/(m$^3$*d), whereas the load on the aerobic reactor varied between 10 and 16 kg COD/(m$^3$*d). The HRT in the anaerobic reactor was around 16.5 hours and 3.4 hours in the aerobic reactor.

After the initial phase the average COD concentration during day 26 to 60 in the effluent from the anaerobic reactor was around 1550 mg/l. The corresponding value for the aerobic reactor was 850 mg/l. This is equivalent to a reduction in the anaerobic reactor of 75% and 87% for the combined process, which can be seen in Figure 4. The consumption of nutrients during this period was calculated to be 24.5 (± 4.4) mg N/ g COD$_{\text{reduced}}$ for the anaerobic reactor and 37.1 (± 10.0) mg N/ g COD$_{\text{reduced}}$ for the aerobic reactor. The corresponding values for ortho-phosphate-phosphorus were 4.4 (± 0.8) mg P/ g COD$_{\text{reduced}}$ and 5.5 (± 1.6) mg P/ g COD$_{\text{reduced}}$.

**Pilot scale experiment**

The COD content in the influent whitewater varied considerably over time, Figure 5. Total COD varied between 1480 and 4240 mg/L, with a mean value of 2620 mg/L while soluble COD was between 1300 and 3780 mg/L with a mean of 2260 mg/L. The highest COD concentrations were found during periods of liner production and the lowest during production of fluting, but the variation within the individual production periods was also large.

The hydraulic flow was increased step-wise from 50 L/d to 300 L/d, Figure 6. During the first period, the HRTs, in the two biological steps were 17 and 6 hours respectively, and the corresponding organic loading around 4 and 1.5 kg COD/m$^3$*d, Figure 2. The removal of soluble COD in the anaerobic step was around 78% while the removal after the aerobic step was 86%, Figure 3. As the HRT was decreased to 10 and 3.5 hours respectively, the resulting load varied between 8 and 20 kg COD/m$^3$*d on the anaerobic step and 3 and 7 kg COD/m$^3$*d on the aerobic step. The removal of soluble COD decreased a few per cent to 76 and 82% respectively, Figure 7. At the highest loading level, the HRTs were 3-4 hours in the anaerobic and 1-1.3 hours in the aerobic step. The organic loading was 18-44 and 8-26 kg COD/m$^3$*d and the removal of soluble COD around 65 and 77% respectively. The removal of COD seemed to decrease temporarily.
Figure 4 COD reduction in percent for the thermophilic reactors

Figure 5 Total and soluble COD in the influent whitewater to the pilot plant

Figure 6 Flow rate and organic load on the anaerobic bed and aerobic process
after each increase in flow. The mean removal efficiencies at the higher loading levels would probably have stabilised, if the process had been allowed to adapt.

The difference between total, settled and soluble COD in the effluent after the aerobic treatment increased with increasing load on the process, due to increasing aerobic degradation and thereby increasing sludge production, Figure 8. The poor separability of suspended solids that sometimes was observed may be explained by the high organic loading or the low nutrient levels.

The concentration of ammonium nitrogen in the untreated whitewater was found to be very low, <0.1 mg/l while the concentration of soluble total nitrogen was between 2 and 5 mg/l. The concentration of ammonium after the anaerobic step varied between very low (< 0.1 mg/l) and high (25 mg/l) values. The concentration of phosphate phosphorus in the untreated whitewater was between <0.1 and 0.4 mg/l while the concentration after the anaerobic treatment varied between 0.3 and 10 mg/l.

Simple filtration tests carried out with total and settled water showed that the total effluent after the biological treatment required a very long filtering time, compared to the untreated whitewater, Figure 9. However, removal of some of the suspended solids through settling decreased the filtering time so that it became shorter for the treated effluents than for the untreated.

![Figure 7](image-url)  
Figure 7 Removal of soluble COD after the anaerobic and aerobic process

![Figure 8](image-url)  
Figure 8 Soluble, settled and total COD in effluent from the aerobic process
The TOD instrument was operated for a year, and was repeatedly calibrated but nevertheless failed to show stable and reliable measurements. Identified problems due to clogging of the injection tube were solved by introducing a rinse with deionised water between injections, but the measurements remained unstable. The explanation for this is probably the complex matrix of the whitewater. There were also problems with the ammonium analyser. The increase in pH caused inorganic material, probably calcium carbonate, to deposit on the electrode membrane.

Discussion
A packaging board mill is probably one of the most suitable types of production for introduction of an in-mill biological treatment since liner and fluting are not as sensitive to colour changes and decreased brightness as some other paper products. The quality demands on the treated whitewater for reuse in a packaging board mill have not yet been fully defined. However, it is known that pH and temperature, organic matter, VFAs, nutrients, amount and character of suspended solids, conductivity, and different compounds that affect consumption and function of the paper chemicals, are all important parameters.

The laboratory experiments showed that a combined anaerobic/aerobic process is able to reduce the major part of the accumulated organic compounds in the whitewater. Another important aspect is the operating temperature of the biokidney. In a closed system the whitewater temperature increases and could be as high as 55°C. In order to avoid costly cooling, the biological process should be able to operate at these elevated temperatures. The experiment demonstrated that biological treatment during thermophilic conditions could reduce around 87% of the organic matter, which is of the same magnitude as what was reduced during mesophilic conditions (between 88 and 93%).

The pilot trials at Munksjö Lagamill showed that a high removal of soluble organic matter was obtained at different loading levels in a combined anaerobic/aerobic biokidney. These results were comparable to the ones obtained in the laboratory experiments. The process also showed stable operation despite large variations in whitewater composition. The most difficult problem caused by the varying concentration of organic matter was to manually control the nutrient dosage, which resulted in an unacceptably high variation in the effluent nutrient concentration. The need for on-line instruments and a process adapted control strategy was clearly shown. The evaluation of the two on-line
instruments showed that continuous measurement in the whitewater is difficult due to the complex matrix.

The dosage of nutrients is one of the key operational parameters since a limitation, or excess, of nutrients affects the final water quality in several different ways. Suspected negative effects of a nutrient limitation include decreased removal efficiency, possible slime/zoogloeal growth, decreased separability of suspended solids, and possibly decreased runnability on the paper machine. These effects have to be compared against the negative effects of an excess dosage such as increased uncontrolled microbial growth, increased conductivity and increased need for retention aids, as well as the additional cost for the nutrients. To reach the ultimate goal, which is to keep the residual nutrient level as low as possible without creating other problems, the minimum and maximum nutrient levels have to be determined and a suitable dosage strategy developed.

Another important factor is the amount and separability of the suspended solids in the treated whitewater. The formation of biomass is inevitable in a biological process, but the amount and separability may be controlled. The sludge production is markedly lower in an anaerobic process than in an aerobic one. Therefore, by removing the main part of the organic matter in the anaerobic stage, the biomass production is minimised. The character of the biomass may be an even more important parameter than the actual amount. Biomass can be found either as free-living bacteria, or as smaller or larger aggregates. Suspended solids in the form of aggregates would interfere with the product quality as well as the production process and therefore have to be removed before reusing the water. The presence of free-living bacteria may not affect the product quality negatively, but a high number may decrease the drainage rate and therefore influence the runnability of the paper machine considerably. Therefore, some kind of removal of suspended solids will most likely be needed in all cases. The removal efficiency and the final amount and character of the remaining suspended solids will probably differ from case to case. The simple filtration tests presented here showed a marked difference between total and settled treated effluent. In this case, the rather coarse separation (settling) increased the filterability of the treated water so that it became at least as good as for the untreated whitewater. In other cases, e.g. when the separability of the suspended solids is poorer, or the demands higher, a more sophisticated separation method may be needed. The separability of the suspended solids seems to depend on the load, but also on the nutrient dosage. Nutrient limitation is known to cause a slimy and viscous biomass.

**Conclusions**

A combined anaerobic/aerobic process has the ability to degrade the major part of the COD in the whitewater from recycled packaging mills. The overall efficiency was only a few percentage units higher during mesophilic conditions as compared to thermophilic conditions.

In the experiments it was found that the nutrient requirement was a little bit higher for the thermophilic anaerobic reactor than for the mesophilic anaerobic reactor and the aerobic reactor required additional nutrients.

The pilot trials showed that the biokidney was rather insensitive towards variations in whitewater composition and organic load. The removal of soluble organic matter was somewhat dependent on the organic load, but was still high at a high organic load. The trials also showed the importance of a strict nutrient control and the need for separation of suspended solids from the treated whitewater.
Acknowledgements
We are very grateful to Alf Claesson and Åke Dahl from Munksjö Lagamill AB for letting
us do these trials at the paper mill. We also owe thanks to NUTEK for their financial sup-
port of this project.

References
TMP whitewater for internal recycle: Biological and physicochemical treatment and effects on pulp