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Continuous Biological Treatment (CBT) of Anaerobic 55 ^{o} Celsius pre-treated Membrane Concentrates of TMP Wastewater Streams from Pulp and Paper

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ABSTRACT: *Implementation of the European Framework Directive (EFD) requires integrated water resource management (IWRM), an important tool in managing available water resources in the presence of emerging constraints. Pressures caused by variability in water supply cycles, droughts, pollution, industrialization, increasing domestic and commercial demand, inadequate infrastructures, intense urbanization and population growth, and decrepit distribution networks have led to the increase exploitation of industrial wastewater. Most industrial processes have some negative impacts on the environment especially on a catchment scale i.e. water, air and soil quality. In the context of water resources and environmental protection measures, the term "sustainable development", a development that is compatible with the future gains and environmental protection is regulated more and more by law. To comply with these emission-based limit values, basically downstream or subsequent techniques, so called "end-of-pipe-techniques" are used. We found that continuous biological treatment (CBT) process presents secondary measures to purify contaminated industrial wastewater. Hence, the objectives of this research paper was to investigate the chemical oxygen demand (COD), biological oxygen demand in 5 days (BOD_5), suspended solids (SS) denoted as "food" (F) to biomass (M; microorganism) ratio (F:M), by targeting varying rate of influent stream. Our result shows the maximum elimination rate based on the effluent parameters i.e. chemical oxygen demand (COD, COD mf), suspended solids (SS), and biological oxygen demand in 5 days (BOD_5) of each continuous biologically treated wastewater. Previous experiments performed using sequential batch reactor (SBR) treatment scheme yielded lower results. The results of our experiments on industrial wastewater streams from the pulp and paper industries within Baden Württemberg, Germany have far reaching global industrial implications. This part of the experiment was focused on TMP anaerobe 55 ^{o} C treatments by continuous biological treatment (CBT) combined with activated sludge system and membrane technology has the advantage of that the separated materials are neither thermally nor chemically or biologically modified.*

Keywords - *Chemical Oxygen Demand (COD); Biological Oxygen Demand (BOD_5); Suspended Solids (SS); Continuous Biological Treatment (CBT); Thermal Mechanical Pulping (TMP); Deinking*

I. INTRODUCTION

The industrial paper production process accounts as one of the largest consumer of water resources. The amount of water and wastewater used is largely dependent on the type of product produced. The integrated water resource management (IWRM) encourages the application of new techniques in maximizing the available water resources. Techniques like water recycling and reuse are essential to mitigating the overexploitation of available resources and reducing freshwater consumption and wastewater generation from the pulp and paper industry. The intermittent application of membrane technology in the treatment process also plays an important role in decreasing freshwater withdrawal but increases the toxicity of permeate retained, an increasing source of reusable water. Two stage membrane filtration processes was used to obtain concentrated wastewater. Ultra-filtration (UF) materials and methods were used to obtain 10% concentrated permeate which was subjected to subsequent Nano-filtration (NF) to attain 18% concentrated permeate, and then stored separately. NF to UF is 1.8 times higher and was mixed to attain equal concentration and stored at 4 degree Celsius before subsequent runs. Continuous biological treatment (CBT) reactor with activated sludge process in an aerobic environment was used in industrial wastewater treatment. The aim of this paper is to explore the benefits of membrane technology as a sustainable technology used in efficiency gains in water saving in the pulp and paper manufacturing process and also to show the efficiency of CBT combined with aerated activated sludge system used in the elimination of pollutants in the membrane concentrate of TMP anaerobe and thermophilic temperature pre-treated wastewater from the pulp and paper industry.

II. THEORETICAL BASIS OF EXPERIMENTAL SETUP

A. Pulp and paper processing

The conversion of wood or recycled paper to slurry fibers is characteristic of the pulping process. Of which the following techniques are used: chemical, mechanical, or a combination of chemical and mechanical pulping. The main purpose of chemical pulping is to extract lignin, a process known as delignification. Mechanical pulping yields up to 90-98% through the stone ground process at temperature (90 – 135 ^{o} C) [1] Waste paper are shredded and mixed hot in alkaline water at pH 9-11 mixed with detergents, dispersants and various solvent. The acidity that is characteristic with wastewater from the pulp and paper industry is due to acid extract in the wood from resin acids, unsaturated fatty acids contributes a significant amount of the toxicity. The most significant of pollutants in wood based wastewater is lignin. Its presence in wastewater results in dark colored, high turbidity, high COD and low BOD_5/COD ratio due to its low biodegradability. Therefore membrane technology uses a selective barrier between two phases. A feed is separated into permeate (filtrate) and wastewater (concentrate) by a membrane under applied driving force.

B. The Need for Membrane Technology

Membrane filtration by ultrafiltration (UF) and nanofiltration (NF) applied to the pulp and paper manufacturing processes fulfill separation tasks, where the amount of dissolved substances including organic and inorganic compounds, microorganisms and turbidity should be reduced. The separation of various components of a nanofiltration and ultrafiltration is related directly to their relative transport rate within the membrane, typically 50-μm internal diameter and 100- to 200-μm outer diameter, which is determined by their diffusivity and solubility in the membrane material. In hollow fiber modules hundreds to thousands of hollow fibers are bundled together to form a module. These membranes consist of a dense film through which permeates are transported by diffusion under the driving force of a pressure, concentration, or electrical potential gradient. Dense membranes can separate permeate of similar size if their solubility in membrane material differs significantly. Symmetric membranes have nearly homogenous structure all over the thickness of the membrane. Asymmetric membranes are made up of two layers: filter and support. Ultrafiltration is considered an important treatment step in the hierarchy of conventional industrial thermal mechanical pulp and paper wastewater treatment schemes. It is the main membrane process employed by the pulp and paper industry and serves as the primary steps in membrane treatment process. The effectiveness of ultrafiltration process is measured by its extraction of high molar mass of ligneous substances. Nanofiltration removes most of the organic load and also the multivalent ions, such as calcium, iron, aluminum, silicon, magnesium and sulphate [2].

Nanofiltration was employed in TMP wastewater in order to attain high retention of lignin before immediate continuous biological treatment by means of activated sludge in an aerobic environment. The efficiency gains can attribute to the ultrafiltration in removing higher molar mass compounds and microorganism, thereby accelerating the efficiency of nanofiltration in the consequent filtration step. It is also widely believed that the choice of specific membrane filters and their design also have an influence on energy requirements and overall efficiency of the elimination of problematic constituents of industrial wastewater from the pulp and paper. Numerous studies have achieved high elimination rates of COD using sequential batch reactor when combined with activated sludge process in TMP raw wastewater.

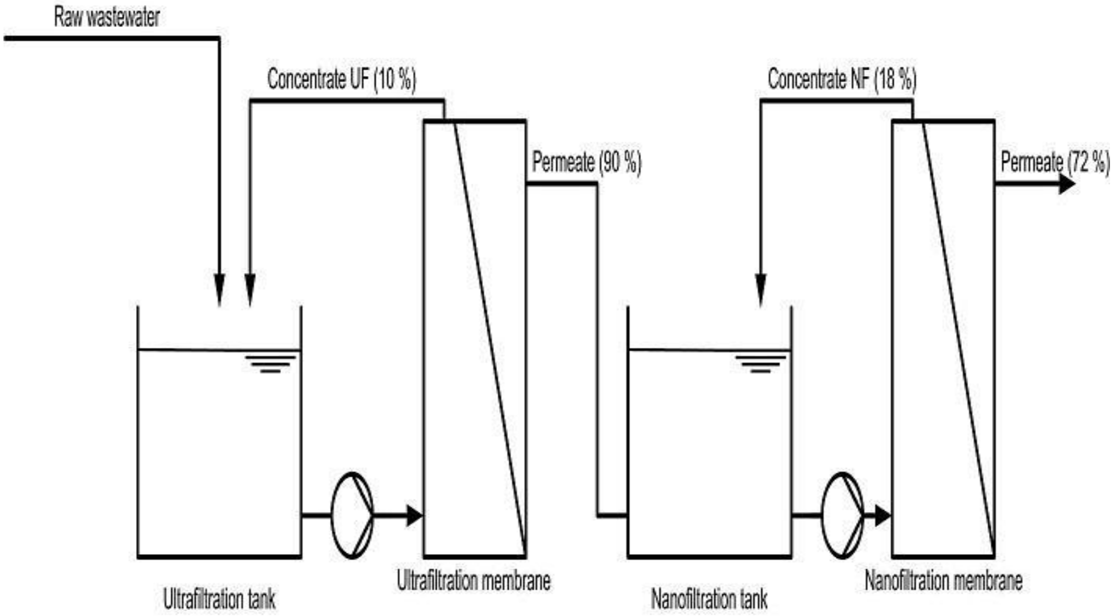

Figure 1: 2-Stage Membrane (UF-NF) Filtration Process

C. Microorganisms and Activated Sludge Growth Process

The growth of microorganism are seen in four phases; lag, log (growth), stationary and death phase with slight alteration based on varying chemical conditions in wastewater and the time needed to adapt to wastewater characteristics. Enzymes produced to sustain a stationary (steady) and growth phase are based on predation and succession of competing microorganisms' species in adaptation to changing wastewater environment, especially through substrate optimization and metabolism. This success of emerging new microorganism leads to the decline of others in wastewater.

In aerobic treatment process, microorganism through three main processes; oxidation, synthesis, and endogenous respiration uses organic matter (biomass) to produce energy. An example of this is exhibited by chemoautotrophs, Nitrosamines in the nitrification process. Microorganisms, in aerobic environment, ammonia are converted into nitrate in two steps: conversion of ammonia into nitrite, and then subsequent oxidation of nitrite into nitrate by Nitrobacterium. Chemo heterotrophs microorganism species involved in other conversions are bacteria, fungi, algae, protozoa and metazoans (flagellates or ciliates) [4]. Fungi and algae compete for food in the aerobic suspended growth process and lower pH environment gives fungi an advantage over algae in the process. Protozoan and metazoans like rotifers and nematodes' make up the secession of the activated sludge process. Objective of these organisms revolves around floc formation, saprophytes, nitrification, predation and secession. Succession by nuisance microorganism can be the cause of sludge blankets, excessive sludge bulking. Nuisance microorganism in biological reactor leads to longer adaptation periods and reduced influent rate.

A pH of 6-9 is optimal for carbonaceous removal. This paper maintained a constant pH of 7-9. TMP and De-inking wastewater had a pH of 4 or lower. Readjustment of pH was performed trough addition of Sodium hydroxide to the raw wastewater; consequently increasing the pH to enable an increase the potential for microbial growth. An acidic condition in wastewater drastically reduces microbial growth therefore antithetical to the activated sludge process.

Mixed liquor suspended solids or MLSS is a mixture of sludge and influent wastewater in the aeration tank. It contains biomass, biodegradable mixed liquor volatile suspended solids MLVSS. The MLVSS is composed of inert and activated microorganism in non-microbial organic matter. It was used in the experimental runs to derive the amount of biomass in the continuous biological reactor system. This in turn is used in continuously targeting of optimum F/M ratio. The amount of organic matter applied to the amount of activated sludge per day should be taken into account determining F/M ratio. Other determining factors of low to high F/M ratio has a direct correlation with the fluctuation in available substrates determines the growth rate of microorganisms and their ability to undergo oxidation, synthesis and endogenous respiration.

TABLE 1: TYPICAL MINIMUM SRT RANGES (METCALF & EDDY 4TH EDITION, 2004)

Treatment	SRT range, d
Removal of soluble COD in domestic wastewater	1 - 2
Conversion of particulate organics in domestic wastewater	2 -4
Develop flocculent biomass for treating domestic wastewater	1 - 3
Develop flocculent biomass for treating domestic wastewater	3 - 5
Provide complete nitrification	3 – 18
Biological phosphorus removal	2 – 4
Stabilization of activated sludge	20 – 40
Degradation of xenophobic compounds	5 - 50

III. MATERIALS AND METHODS

A. Daily Operation parameters

Daily operations are performed to ensure proper maintenance and continuous operation of the biological reactor. Parameters requiring daily maintenance are dissolved oxygen aeration, temperature, nutrients, pH, and suspended solids. Dissolved oxygen must be sufficient for the growth of microorganism in aerobic environment. Dissolved oxygen is supplied through aeration. Dissolved oxygen (DO) concentration should be maintained at 2.0 mg/L and not allowed below 0.5 mg/l. These levels promote growth and inhibit bulking and filamentous growth.

Temperature was constant in all experiment at 30-35 degrees Celsius. This promotes higher percent elimination rate observed in all measured parameters and promote nitrification. A constant thermostat was used to obtain temperature values. A minimum of three measurements was performed per experimental day.

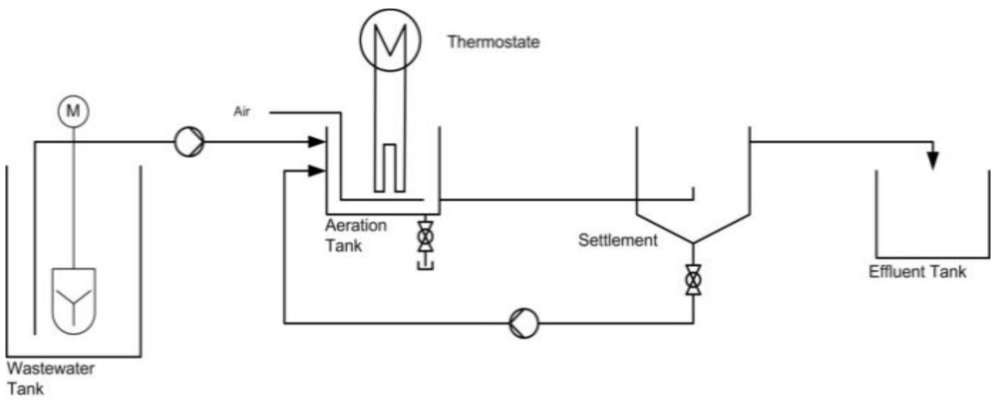

FIGURE 2: EXPERIMENTAL SETUP FOR CONTINUOUS BIOLOGICAL TREATMENT

Nutrients, nitrogen (N) and phosphorus (P) are essential nutrients required for the growth of microorganism which in turn synthesize new biomass. Beside the widely used nutrients, nitrogen and phosphorus. Microorganism also requires available macronutrients (S, K, Mg, Ca, Fe, Na, and Cl) and micronutrient (Co, Ni, Zn, Mn, Mo and Se) for growth. Nutrients in the form of urea NH_2CONH_4 and di-hydrogen potassium phosphate KH_2PO_4 are added to the influent wastewater based on measured BOD_5 concentration at the start of each run. The ratio of 100C:5N:1P is maintained for optimum growth. Therefore, for every 5 g of ammonium (N), 1 g of KH2PO4 (P) will be required to fulfill 100 g BOD_5.

For the optimal treatment of highly concentrated influent stream (1800 to 10,000 mg/l MLSS) and conversion of organic pollutants to non-pollutant requires various means of mixing and the promotion of suspended growth process of microorganism in the activated sludge [5]. The continuous biological reactor is used in all my experimental runs. The continuous biological reactor is composed of a reactor (aeration tank), sedimentation tank (denitrification tank, clarifier) and reverse sludge recycling system. For example, MLSS is the mixture of wastewater and microorganism in the activated sludge in the aeration tank until the adequate F/M ratio is reached. Continuous aeration by dissolved oxygen (DO) is used to promote microorganism growth through biological reaction. The MLSS is later separated to a liquid and solid phase in the sedimentation tank where the aeration is turned off to promote denitrification. The supernatant flows out of the top of the sedimentation tank as effluent while the settle solids at the bottom is recycled back to the aeration tank.

IV. RESULTS: FIGURES AND TABLES

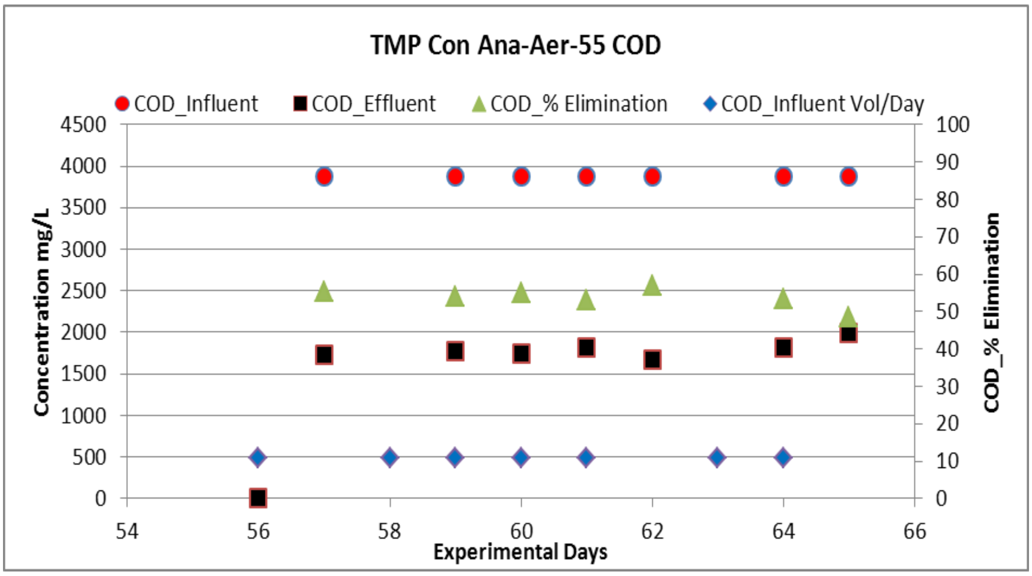

Figure 3: COD TMP Con Ana-Aer-55

The COD_5 result showed the highest elimination rate on day 62 at 55% with Influent rate of 10.9 liters per day. The COD_5 results as shown in this phase of the experiment in days 56 – 65 reveals a steady state percent elimination rate. This is largely attributed to the longer acclimation time of the microorganism in thermophilic conditions. The influent volume was constant at 10.9 liters per day as shown in Figure 3; consistent with the COD_5 elimination results. As shown in previous phase of the experiments, a longer experimentation time and higher substrate would have induced highly favorable results. An increase in the influent rate would have contributed to higher elimination rates due to acclimation of microorganisms due to the conversion of the pretreated thermophilic conditions to a more favorable mesophilic steady state. This would have been more efficacious in increasing the elimination rate of chemical oxygen demand.

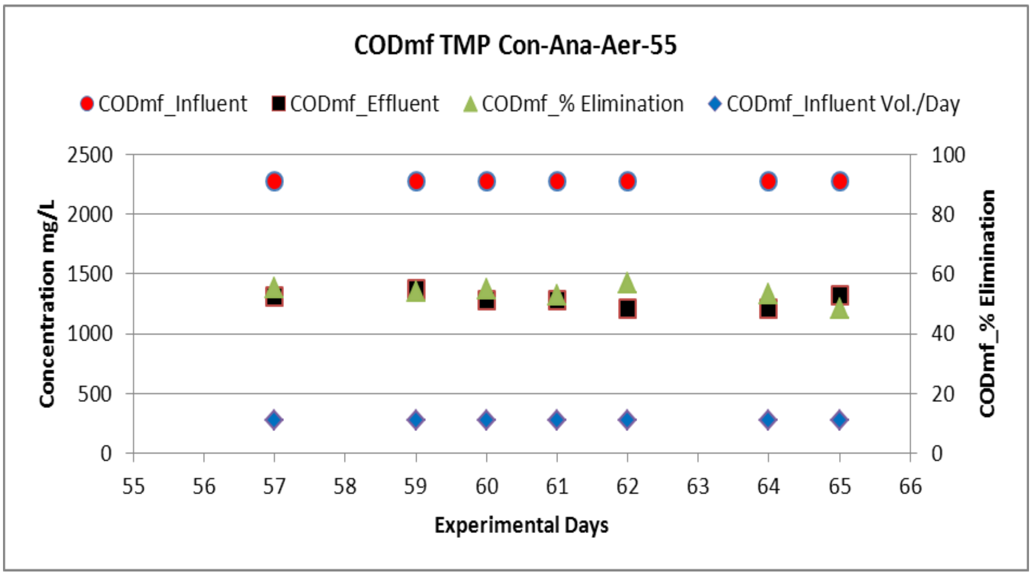

Figure 4: COD mf TMP Con-Ana-Aer-55

The soluble chemical oxygen demand or COD $_{mf}$ results showed the highest elimination rate on day 62 at 52% with Influent rate of 10.9 liters/day. The similarity of the results to COD $_{mf}$ is an indication to the accuracy of the measurement from the total COD and the filtered COD $_{mf}$ and the limitations of the microorganisms under thermophilic conditions. Hugh variation between both results is a clear indication of an ineffective activated sludge treatment reactor due to high input of suspended solids in the effluent.

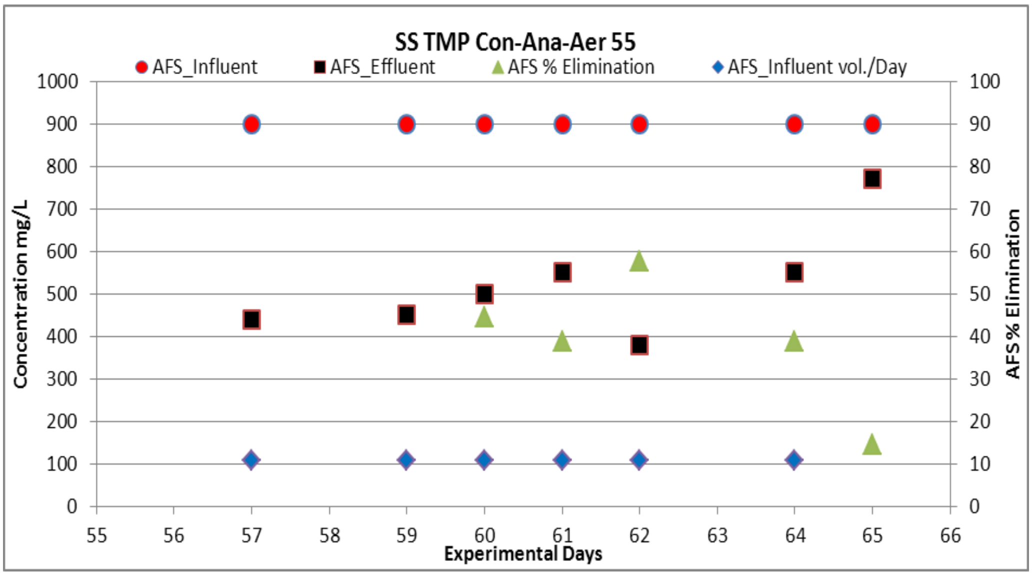

Figure 5: SS TMP Con-Ana-Aer-55

The result showed the highest elimination rate of suspended solids on day 62 at 58% with Influent rate of 10.9 liters/day. The higher than normal turbidity presence in the effluent can be attributed to many factors. The acclimation of the microorganism in thermophilic conditions were inefficient in promoting the proper adaption through continuous substrate provisions, inefficient sludge settling capacity in the secondary settling tank (SST) and growth of sludge blanket caused by nuisance microorganism. At a constant influent rate of 10.9 liters per day, possible increase of the influent rate and a prolonged experimental time could have possibly increased the suspended solids elimination rate.

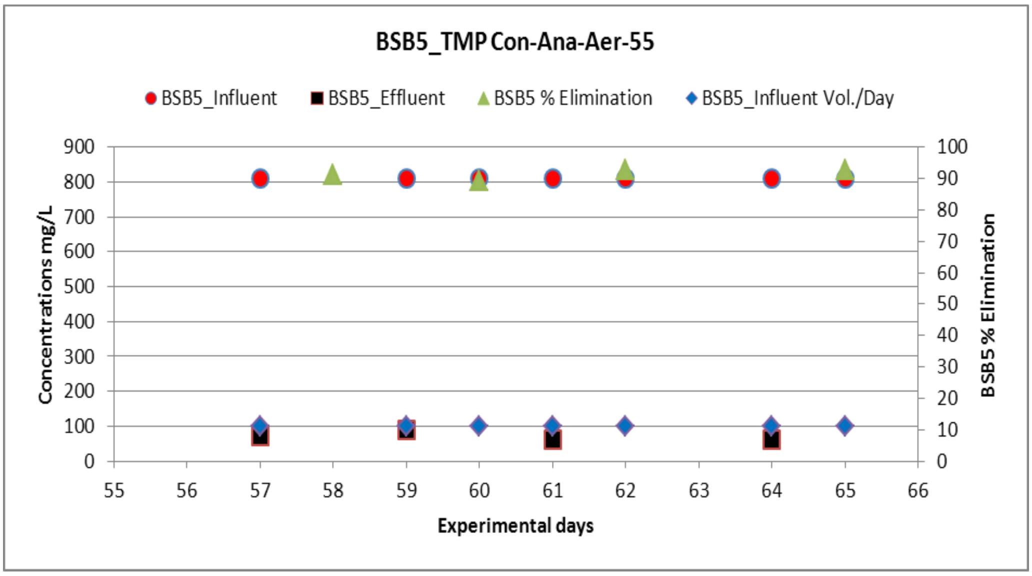

Figure 6: BSB_5 TMP Cn-Ana-Aer-55

The biological oxygen demand in 5 days or BSB_5 results shows the highest elimination rate on day 65 at 90% with influent rate of 10.9 liters per day. This result is an indication of the efficacy of the continuous batch reactor especially the secondary sedimentation tank (SST) in the sequestration of the biological oxygen demand in 5 days (BSB_5). The constant influent rate was observed in the effluent sample result and the rate of elimination remains constant throughout the experiment.

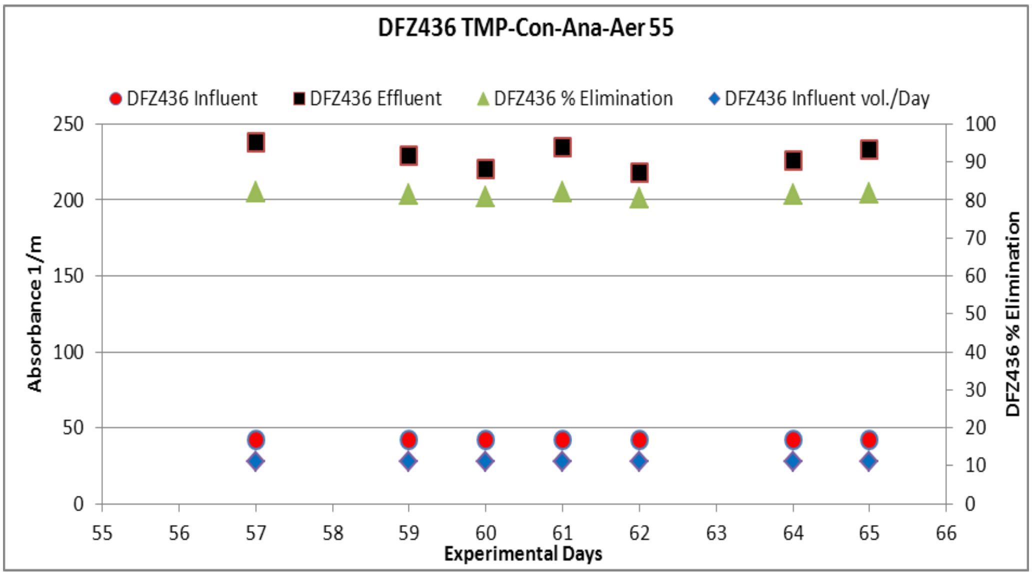

Figure 7: DFZ436 TMP-Con-Ana-Aer-55

The result shows the highest elimination rate on day 65 at 82% with Influent rate of 10.9 liters per day. The influent rates remained the same throughout the experiment and this steady trend in elimination rate of the effluent parameter is closely correlated to the constant influent rate at 10.9 liters per day. High elimination rate in absorbance is an indicator of reduction of pollutants and turbidity observed in the pretreated raw wastewater.

TABLE 2: TMP-AN55 INFLUENT AND EFFLUENT PARAMETERS

PARAMETERS	*UNIT*	*INFLUENT*	*EFFLUENT*
COD	**mg/l**	**3870**	**1990 - 1670**
COD_{mf}	**mg/l**	**2280**	**1370 - 1210**
BOD_5	**mg/l**	**810**	**88 - 60**
BOD_5/COD	**-**	**0.20**	**0.04 – 0.036**
DFZ436	**1/m**	**42.3**	**218 – 238**
DFZ525	**1/m**	**23**	**96.9 – 106**
DFZ620	**1/m**	**14.6**	**38.9 – 43.5**
SS	**mg/l**	**900**	**770 – 440**

The suspended solids or SS influent was at 900 mg/l while effluent fluctuated between 400 and 800 mg/l. This fluctuation can be attributed to flocs caused by sludge blankets which consequently trickled into the effluent sample during the experimental period. This is made evident by the very low elimination rate of 15% observed on day 65

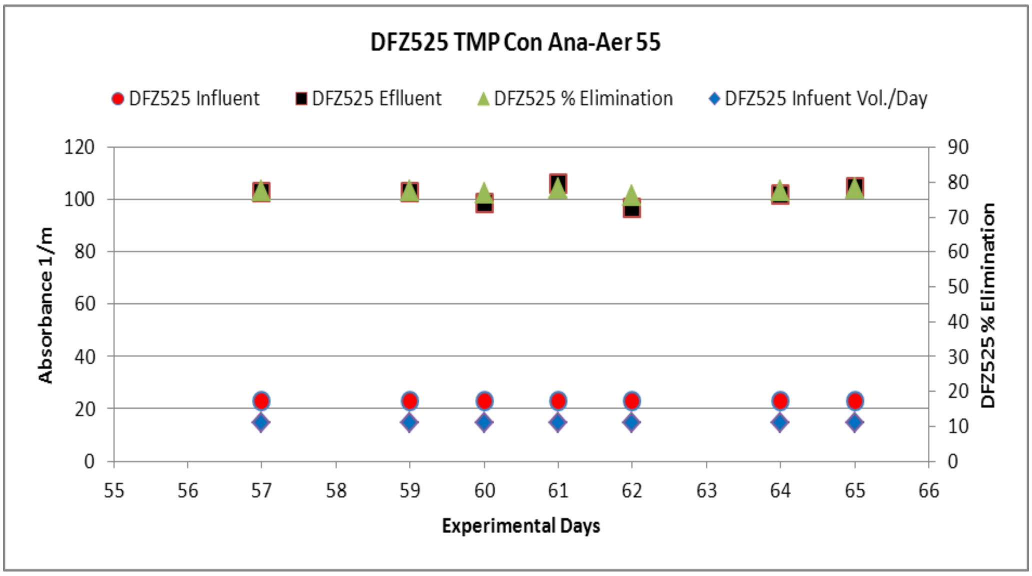

Figure 8: DFZ525 TMP-Con-Ana-Aer-55

The results showed the highest elimination rate on day 65 at 78% with Influent rate of 10.9 liters/day. The consistent rate of elimination has a positive correlation with the constant influent rate. An increase in the influent rate could have yielded a higher elimination rate. A similar elimination rate was observed in comparison to DFZ425 while DFZ620 decreased markedly to 67 % at day 61. The three markers for the examination of turbidity confirms that continuous biological treatment (CBT) is highly effective in the treatment of highly concentrated permeate and pretreated raw wastewater from the pulp and paper industry.

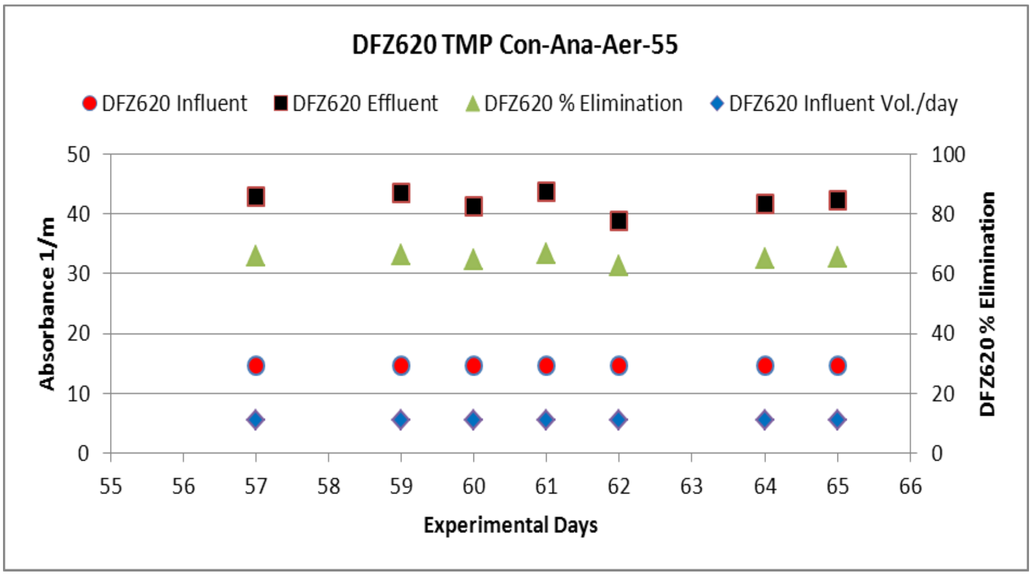

Figure 9: DFZ620 TMP-Con-Ana-Aer-55

TABLE 3: MLVSS/MLSS AND SRT RESULTS

PARAMETERS	***UNITS***	***TMP-AN55***
SRT	**Day**	**10 - 50**
MLVSS/MLSS	**%**	**69 - 75**

Sludge age or solid retention time (SRT) is highly influential in the performance of continuous biological treatment process. It is best to maintain throughout the experiment sludge of 5g/ml in the aeration tank. This is confirmed with daily measurements with the MLSS and MLVSS methodology and readjustment to maintain that optimum level. Precaution must be taken to make sure the sludge is uniformly aerated with dissolved oxygen to promote adequate sludge retention time. Other daily operations include mixing the sludge in the aeration tanks, checking of pH and removal of excess sludge from the SST. Sludge retention time is the average residence time of microorganism in the system. SRT is used preliminary design selection of reactor volume, sludge production volume and aeration requirement. Inversely temperature dependent, low SRT is an indicator of low inefficient elimination of pollutant, while other extremes are indication of death and decay of microorganism population in the activated sludge process within a period of 10-50 days.

The importance of a longer sludge retention time is its' influence on the food to microorganism ratio (F/M). A higher F/M ratio yields higher elimination rates. The targeted F/M ratio throughout this experiment has been 0.3 g. SS.D. As shown in Figure 10, F/M ratio was much lower than the target range. This was influenced by the MLSS/MLVSS range of 69-75%. The optimum range of MLSS/MLVSS range is over 80%. This ensures that there is enough viable microorganisms in the aeration tank to consume the food, thereby lowering the pollution and increasing the elimination rate of the parameters. The highest F/M ratio attained was 0.086, far lower than the expected target. This can be attributed to the thermophiles microorganism in the pretreated raw wastewater. Therefore, under mesophilic treatment conditions, mesophilic microorganism was unable to fully acclimate and build a sustainable stationary phase after the log phase was achieved in the aeration tank.

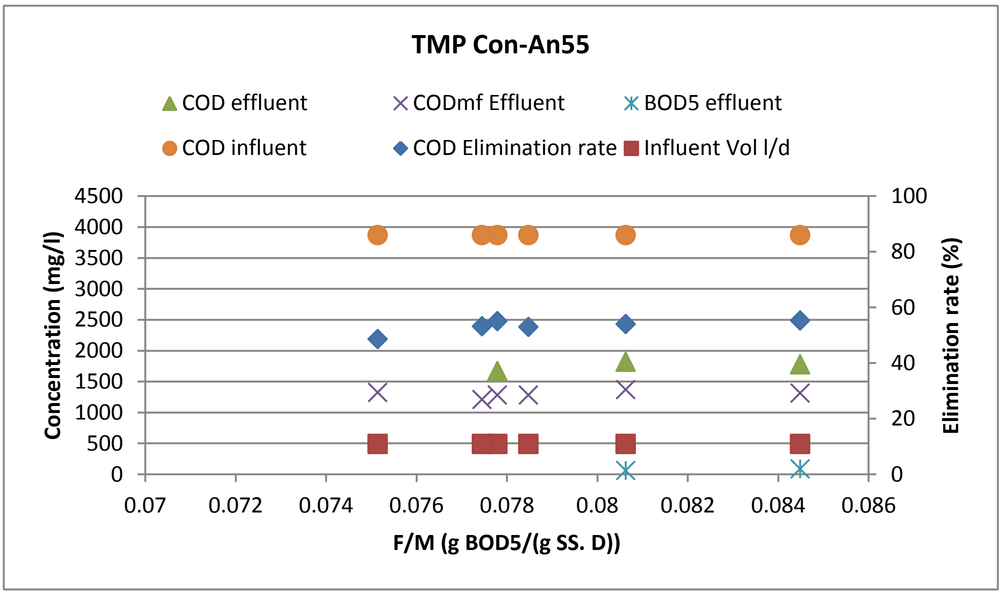

Figure 10: F/M ratio TMP-Con-Ana-Aer-55

The F/M ratio results were lower than 0.3g.SS.D. This is an indicator of lower biodegradability of COD_5 and BSB_5 while influent feed targeting which is lower than usual played an influential role in the low F/M ratio ranging from 0.075 to 0.085 g. SS.D for each respective experimental day as shown in Figure 10.

V. CONCLUSION

Membrane technologies in combination with activated sludge treatment are efficient in the treatment of wastewater treatment from the pulp and paper industry. Membrane filtration was very efficacious in the removal of higher molecules from highly concentrated permeate which allows for easy degradation of effluent parameters. Most parameters experienced high removal rate from influent wastewater (Table 2) and biological oxygen demand in 5 day (BOD_5) was highly biodegraded during this phase of the experiment.

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