Organic and nutrient removal from pulp and paper industry wastewater by extended aeration activated sludge system

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Abstract. Pulp and paper industries are critical to a country's economic growth. The type of raw material used and the pulping process determine the quality and quantity of wastewater generated. However, the generated wastewater with a dark colour comprises a high concentration of suspended solids, organic content, chemical oxygen demand (COD), volatile organic compounds, and a variety of other impurities. Therefore, in this study, a bench scale activated sludge treatment system was set up using a reactor consisting of an aeration tank with 5000 mg/L initial biomass and a clarifier chamber for the biomass to settle. The reactor was run few weeks with real domestic wastewater as the influent for 3 weeks to acclimatize the sludge inside the reactor. The reactor was then fed with the influent mixture of 20% industrial pulp wastewater and 80% domestic wastewater. Organic and nutrient parameter concentrations are tested from the influent and effluent sample throughout the study duration and recorded for data analysis. The removal of COD and TSS are at 83% and 90% respectively while the averaged BOD value of the treated wastewater is at 74.6%. The conclusion of this project is that the bench scale EAAS is able to treat BOD and TSS according to standard. However, a modification may be required to increase the efficiency of removing COD to meet the requirement standards. This modification could be either by using a biocarrier or an activated carbon to further enhance the treatment efficiency even at higher wastewater concentration.

1. Introduction

Paper industry is developing with about 400 million tonnes of paper produced every year from 5000 pulp and paper mills throughout the world [1]. Pulping is a process where soil, dirt and bark are removed from the wood raw material and converts into smaller bits. This process produced a lot of wastewater due to high amount of water is used to remove organic compound from the wood [2]. The pulp and paper industries are under rising economic and environmental restrictions due to international competition and increasingly severe environmental legislations. Between 2012 and 2021, the global pulp and paper waste and wastewater treatment market is projected to grow by 60% [3]. The pulp-and-paper company consumes a lot of fresh water and produces a lot of wastewater at different phases of the pulping and papermaking process. Tannins, lignins, resins, and chlorine compounds are the most common organic and inorganic contaminants found in this wastewater [4]. Water is produced in the pulp and paper manufacturing throughout a variety of processes, including wood debarking or chipping, pulp manufacturing and...
bleaching, paper production, and fibre recycling. All these processes require a lot of fresh water and generate a lot of effluent [5-17].

Pulp wastewaters are produced from the multiple pulping processes. There are 3 type of pulping processes which are mechanical, chemical and kraft pulping. Pulp wastewater that contain COD level of 1000 mg/L are consider low strength wastewater, 5000 mg/L are in the range of medium strength and 10,000 mg/L are designated as high strength [18]. As the pulp and paper industries rapidly expand around the world, a lot of them strive to meet the government's stringent environmental regulations. A lot of option can be chosen to treat the wastewater produced from the pulping process [2]. Therefore, industrial effluents limit for effluents such as pulp and paper wastewater is a priority issue of water quality management scheme for the protection of the environment. Their effective control depends upon appropriate effluent limitations. The correct application and enforcement of discharge standards, which also provide the required tools for legal actions, are crucial to the implementation of a quality management plan. The Malaysian Environmental Quality Act (EQA) of 1974 established two criteria for industrial effluent discharge: Standard A for discharge upstream of any raw water intake and Standard B for discharge downstream of any raw water intake. The Industrial Effluent Discharge Limits established in the Environmental Quality Regulations 2009 are shown in Table 1.

| Parameter                  | Standard  A | Standard  B |
|---------------------------|------------|------------|
| pH                        | 6.0-9.0    | 5.5-9.0    |
| BOD (mg/L)                | 20         | 50         |
| COD (mg/L)                | 120        | 200        |
| Suspended Solids (mg/L)   | 50         | 100        |
| Oil and Grease (mg/L)     | 1.0        | 10         |
| Ammoniacal Nitrogen (mg/L)| 10         | 20         |
| Temperature (°C)          | 40         | 40         |

Treatment system capable of COD, color and conductivity value reduction in pulp and paper wastewater seem to be of great importance in order to recycle the final effluent [19]. Owing to pulp and paper wastewater's inhibitory and recalcitrant behavior, it is usually not appropriate to apply traditional biological wastewater treatment as most of the substances with large molecular mass are immune to biodegradation [20, 21]. However, a review by [22] concluded that biological treatment methods commonly utilized, in treating industrial effluents with the sole objective of removing fully or partially organics compound into acceptable limit when discharged are effective, reliable, eco-friendly, energy saving, financially appealing dye degradation techniques with minimum chemicals usage requirement. According to [23], color substances in pulp wastewater is a sort of refractory organic material, that can either be solubilized or utilized by microorganisms in traditional biological treatment systems as sources of energy and carbon. In biological treatment systems, a broad variety of microorganisms, particularly bacteria, algae, yeast and fungi, are capable of de-staining and degrading many types of contaminants [24] in pulp and paper wastewater with bacteria and fungi as the most intensively researched [20].

Strategies other than the conventional ones employed for pulp and paper wastewater treatment for possible reuse categorized into physicochemical, biological and combined systems have been reviewed in recent times [25]. The typical biological wastewater treatment methods such as activated sludge process, pure culture of decolorizer, oxidation ponds, aerated lagoons and sequencing batch reactors produces less sludge [26]. Recently, the usage of extended aeration activated sludge system (EAAS) in wastewater
treatment, has continuously receive attention from scientists. Therefore, the objective of the present study is to verify the feasibility of treating pulp and paper industry wastewater using bench scale (EAAS) for organic and nutrients removal to achieve the requirement standard.

2. Materials and methods

2.1. Materials and experimental plan
All materials and chemicals were of analytical quality and were utilized exactly as they were obtained [27]. Figure 1 below describes the experimental flow process adopted.

2.2. Pulp and paper wastewater collection
The pulp wastewater (Figure 2) used in this study is collected from a local company producing biodegradable food packaging by utilizing rice straws and husks located in Gurun, Kedah. Five tanks of 20 L of pulp wastewater were successfully acquired and stored in cold room located in Environmental Research Laboratory (ERL) [28]. The wastewater collected will be tested for characterization before being used in activated sludge system.

![Figure 1. Methodology Flowchart.](image)
![Figure 2. Raw pulp and paper wastewater.](image)

Experimental procedure
In this experiment, a bench scale Extended Aeration Activated Sludge system was set up to treat pulp wastewater from the industry. A total of 100 L of pulp wastewater was collected from the industry where the wastewater will be characterized and treated using a bench scale activated sludge system. The samples were monitored for Ammonia, Nitrate, Phosphorus, COD, TSS, MLSS and MLVSS with a frequency of three times per week. Finally, the data were collected and analyzed. Flow chart of the experiment is visualized in Figure 2. Influent and effluent sample from the bench scale system will be taken to be analysed for organic parameter such as BOD, COD, TSS, MLSS and MLVSS. The influent samples with parameter concentrations shown in Table 2 also will be taken to test for stated parameter with a variation of organic loading to the system. The data will then be compared to environmental discharge standard set by Malaysia Department of Environment to see whether the effluent is in within standard or not.
Table 2. Wastewater characteristics

| Parameters  | Concentrations (mg/L) |
|------------|-----------------------|
|            | Domestic wastewater   | Pulp and paper wastewater |
| COD        | 500                   | 1195                     |
| BOD$_5$@20°C | 250                | 1566                     |
| TSS        | 300                   | 660                      |
| NH$_3$-N   | 2                     | 42                       |
| NO$_3$-     | 1                     | 5                        |
| PO$_4^{3-}$ | 7                    | 65                       |

From the characterization process conducted, the industrial wastewater has a low Nitrate concentration of 5.0 mg/L. However, the concentration of Ammonia in the wastewater is high, which is around 42 mg/L and has exceeded Standard B industrial effluent limits of 20 mg/L by the Malaysian Environmental Quality. It is also found that concentration of Phosphorus in the wastewater is very high, which can reach a maximum concentration of 65 mg/L.

**Reactor start-up and acclimatization**

Two fabricated biological reactors (reactors A and B), each consisting of aeration tank and clarifier section were installed in the laboratory for continuous flow. The design of the reactors was adopted from the concept and design approach of Ahmad et al [29]. Venus Distributor Sdn Bhd, based in Malaysia, built the reactors. Acrylic glass with a thickness of 5 mm was used to make the reactors. A considerable number of tube diffusers were fitted to provide thorough mixing and aeration [30]. Cole Parmer Tygon tubing (Cat 06429-24) was used to link the reactors to the feed tank (20 L). Figure 3 depicts the design and dimensions of the reactors.

Reactor A was acting as the control with raw domestic water and reactor B was used for the co-digestion of pulp and paper wastewater with domestic wastewater. The domestic wastewater used and seeded biomass in both reactors were fetch up from Universiti Teknologi PETRONAS wastewater treatment (STP)). The biomass seeded into the reactors was collected from the return sludge pipeline of the treatment plant. The activated sludge system was allowed to acclimatize with the domestic wastewater and pulp wastewater. Authors used (1 L) biomass and (9 L) wastewater inside both reactors.

The reactor will be operated at extended aeration to achieve nitrification. Domestic wastewater sample will be mixed with pulp wastewater. After it is properly mixed, the wastewater will be pumped into the system until steady state are reached at a flowrate of 2.6 litres/day (detention time = 24 hours). The moment acclimatization is achieved, the organic loading to reactor will be increased gradually until the effluent limits is violated. From this study, optimum organic loading will be determined. Maintenance need to be done to all part of the process from influent tank until effluent tank on every sampling day after sampling testing have been done. Influent tank needs to be clean thoroughly and to ensure no residue left from previous influent mix. Tubing connecting from influent tank feeding the influent mix into the reactor needs to be check for blockage which can occur due to suspended solid clumping caused by low flow rate of influent being fed into the reactor. Inside of the reactor, splashes of sludge occur due to burst of oxygen from oxygen tube causing some of the sludge to be present on the inside surface of the reactor cover. The sludge must be cleaned of from the reactor cover using brushes or sprayed with the same wastewater back into the reactor. The inner part of the reactor needs to be brush weekly to prevent the growth of algae.
which may affect the sample testing results. Excessive sludge also needs to be removed based on MLSS level on that sampling day to maintain the MLSS range of 4000 mg/L as prescribed in Table 3.

Table 3. Typical design parameters of activated sludge system.

| Parameter   | Values |
|-------------|--------|
| SRT, d      | 20-40  |
| F/M Ratio   | 0.04-01|
| MLSS, mg/L  | 4000-6000|

For this research, a bench scale model was used to treat the wastewater sample receive from a company producing biodegradable packaging from rice straws and husks. The reactor dimension is shown in Figure 3. The volume of sludge used was determined by selecting appropriate sludge age ranging between 15 to 25 days. The desired period of steady-state was preferred when the proportion of the standard deviations is based on the average efficiency of the TSS, COD, NH₃, NO₃, and Total phosphorus, MLSS and MLVSS and SVI was less than 10% In this continuous flow study, domestic and pulp industry wastewater were fed to both reactor every two days (48 hours) until steady state. The design allows influent to flow into both reactors continuously. A peristaltic pump will be used to feed the influent to both reactors at a flow rate of 10 L/d. Sampling will be taken from the influent and effluent of both reactors every two. The tests will be carried out in accordance with APHA Method 4500-NO₃: Standard Methods for the Examination of Water and Wastewater, 15th Edition and Standard Methods for the Examination of Water and Wastewater, 4500-NH₃ B & C.

3. RESULTS AND DISCUSSION
In this chapter, results of the experiment will be discussed thoroughly for every aspect of the study, explaining the behaviour of the graph and factor affecting the data obtained. All graph shown will be divided into two (2) phases which are phase 1 (acclimatization phase) and phase 2 (treatment of pulp and paper wastewater). In phase 1, the value of the graph will be slowly stable to reach the optimum acclimatization of the reactor. Phase 2 started when the reactor was guaranteed it is fully acclimatized and ready be fed with industrial wastewater sample at 20% loading rate.

3.1. Acclimatization phase of sludge
The Performances of EAAS are evaluated using two reactors simultaneously- namely Reactor A and Reactor B, for approximately 18 days. The effluent parameters of TSS, COD, ammonia, nitrate, total phosphorus, MLSS and MLVSS were all measured. At the acclimatization phase, sludge was able to start to stabilize at sampling day 4.

3.1.1. TSS. The concentration of TSS throughout this study is presented in Figure 3 (a) above. As expected, during phase 1, the influent TSS value was below 200 mg/L due to the domestic wastewater characteristic. Value for phase 2 was higher due to the high concentration of TSS in pulp wastewater. The effluent produced from the reactor at sampling days 1 to day 9 shows the value of TSS less than 20 mg/L which can be consider very low. The effluent produced from the reactor reduced the TSS content to below 100 mg/L. From sampling day 10 to day 18, the results produced consistently show the TSS value below
50 mg/L. On sampling day 19 to day 24, the value starts to float around 30-95 mg/L. The average removal percentage of TSS is 90%.

3.1.2. COD. The concentration of COD throughout this study is presented in Figure 3 (b) above. The difference of COD value can be clearly seen between phases 1 and 2, which illustrate the difference of domestic, and pulp and paper wastewater respectively. From sampling day 1 to 9, the reactor had a minimum COD of 500 mg/L. The mixture ratio is split into 80% of domestic wastewater and 20% of the pulp wastewater with the total volume of 20 L. After being fed into the reactor, the effluent produced show COD value that are slightly below 200 mg/L for sampling day 10 to 14. This value slowly increases over sampling day from around 200 mg/L at sampling day 15 to over 300 mg/L at sampling day 18. This is the result of slightly higher COD content in the second sample tank. For sampling day 19 to day 24, the reactor has stabilized and effluent COD value floats around 250-350 mg/L. The average percentage of COD removal is calculated 83% are obtained.

3.1.3. Ammonia. The concentration of ammonia throughout this study is presented in Figure 3 (c) above. The influent Ammonia concentration drops from 6.5 mg/L to 2.6 mg/L in sampling days 1 to 4. In sampling day 5 to 7, ammonia concentration increases to 6.5 mg/L which can be the result of different domestic wastewater fed into the reactor and decreases back to 4.3 mg/L on sampling day 9. From day 10 onwards, a mix of 20% pulp wastewater, 80% domestic wastewater results in sharp increase in influent Ammonia concentration by 200%, from 4.2 mg/L to 13 mg/ on day 10. The reading of Ammonia concentration throughout treating the pulp wastewater averages at 13.8 mg/L for influent and 5.2 mg/L for the effluent. The percentage of removals of ammonia was calculated to be 62.3%. The result shows that the effluent reading of ammonia concentration passes the acceptable conditions for discharge of industrial effluent limits for standard A which does not exceed 10 mg/L.

3.1.4. Nitrate. The concentration of nitrate throughout the study is presented in Figure 3 (d). During sampling days 1 to 9, it was observed that the reading of influent nitrate is relatively low with an average 1.6 mg/L with a peak reading of 3.4 mg/L on day 7. It was found that effluent concentration is high with an average reading of 14.6 mg/L. Starting from sampling day 10 onwards, the reactor was feed with 20% pulp wastewater and 80% domestic. Nitrate influent concentration increases from 1.2 mg/L to a maximum of 12.5 mg/L on day 14. From day 19 onwards, influent nitrate concentration varies between 5-10 mg/L with an average reading of 2 mg/L. While the effluent experienced a major reduction in Nitrate concentration of 89% decreases from 17.4 mg/L to 1.9 mg/L.

3.1.5. Total Phosphorus. The concentration of total phosphorus throughout this study is presented in Figure 3 (e). For sampling day 1 until sampling day 9, the reactor was fed with 20 L of domestic wastewater for acclimatization of reactor. It was observed that Phosphorus concentration in influent averages at 15.9 mg/L while the effluent averages at 6.9 mg/L. On sampling day 10, it was seen that the influent Phosphorus concentration experienced a significant increase of 20% from 15.95 mg/L to 48.1 mg/L. From sampling day 15 onwards, the sample of pulp wastewaster was taken from tank two which results in increase of effluent phosphorus reading with a peak of 40.5 mg/L on day 21 with an average reading of Phosphorus concentration of 30.5 mg/L. The average percentage of removals of Phosphorus was calculated to be 34%.
4. Conclusion

The primary objectives of this research are to investigate the performance of activated sludge in organic and nutrient removal during the co-treatment of pulp and paper wastewater in a bench-scale EAAS system. The performance of the system is determined by evaluating the removal efficiency of phosphorus, nitrate, ammoniacal-nitrogen, COD, and TSS. For Ammonia, average of 13.8 mg/L influent concentration was reduced to an average of 5.2 mg/L effluent concentration with percentage of removals of 62.3%. While for Phosphorus, average of 46.2 mg/L of influent concentration was reduced to an average of 30.5
mg/L effluent concentration with percentage of removals of 34%. The results shows that Ammonia has successfully meet the standard limits of Standard A with limit of 10 mg/L when 20% of pulp wastewater was added into the influent mix. Consequently, COD and TSS removal efficiencies were 83% and 90% respectively. However, most of effluent COD value does not meet the requirement standard and thus further testing need to be done. The average effluent BOD was tested to be 4.54 mg/L. Further studies can also be done with varying organic loading rate, flow rate and detention time to find the optimum parameters in treating the industrial wastewater.

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