第 49 卷 第 2 期
2022 年 2 月

湖南大学学报（自然科学版）
Journal of Hunan University (Natural Sciences)

Vol. 49 No. 2
February 2022

Open Access Article https://doi.org/10.55463/issn.1674-2974.49.2.24

Kinetics of Pulp and Paper Wastewater Treatment by High Sludge Retention Time Activated Sludge Process

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Abstract: Paper and pulp industrial processes lead to the discharge of wastewater that contains high pollutants concentrations into the environment, which subsequently contaminate freshwater. Thus, it necessitates a sustainable treatment approach. This study focused on the start-up of the bench-scale activated sludge system fed with pulp and paper wastewater to verify the influence of HRT, wastewater concentration, and sugarcane bagasse on COD and ammonia removal efficiencies during the treatment process. An activated sludge process was operated at a flow rate of 5 L/day, while the reactor kept running at 72 h, 48 h, and 24 h HRT, respectively. Wastewater concentrations were set at 1039, 3158, 5248 mgCOD//L and 13.74, 40.37, 67.04 mgNH_4^+-N/L corresponding to 10, 50 and 100% respectively. Findings revealed high removal efficiencies up to 98.11% and 92.67% for COD and ammonia, respectively. After treatment, effluent concentrations for both parameters have satisfactorily met the Standard "A" standard limits for industrial discharge at 48 hours HRT. Therefore, further testing is not required. The First order and Modified Stover-Kincannon models evaluated substrate removal rates. In the Modified Stover–Kincannon model, high correlation coefficients R^2 of 0.9999 and 0.9998 were obtained for COD and ammonia, respectively. Unfortunately, the activated sludge process in the bioreactor could not be described by the first-order kinetic model. The modified Stover-Kincannon model proved to best suit the experimental data.

Keywords: activated sludge process, hydraulic retention time, nutrients, organic matter, pulp and paper wastewater.

高污泥停留时间活性污泥法处理制浆造纸废水的动力学

摘要：造纸和纸浆工业过程导致将含有高浓度污染物的废水排放到环境中，从而污染淡水。因此，它需要一种可持续的治疗方法。本研究的重点是启动以纸浆和造纸废水为原料的小型活性污泥系统，以验证保留时间长、废水浓度和甘蔗渣对处理过程中化学需氧量和氨去除效率的影响。活性污泥法以5L/天的流速运行，而反应器分别在72小时、48小时和24小时保留时间长下运行。废水浓度设置为1039、3158、5248毫克化学需氧量//大号和13.74、40.37、67.04毫克NH_4^+-

Received: November 23, 2021 / Revised: December 07, 2021 / Accepted: January 06, 2022 / Published: February 28, 2022

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N/L，分别对应10、50和100%。结果显示，化学需氧量和氨的去除效率分别高达98.11%和92.67%。经过处理后，这两个参数的出水浓度都令人满意地满足了48小时保留时间长下工业排放的标准"A"标准限制。因此，不需要进一步的测试。一阶和改进的斯托弗-金坎农模型评估了基板去除率。在改进的斯托弗-金坎农模型中，化学需氧量和氨的相关系数决定系数分别为0.9999和0.9998。不幸的是，生物反应器中的活性污泥过程不能用一级动力学模型来描述。修改后的斯托弗-金坎农模型证明最适合实验数据。

关键词：活性污泥法、水力停留时间、营养物质、有机物、纸浆和造纸废水。

1. Introduction

Water is extensively used during the production process in the pulp and paper mills [1, 2]. It depends upon the raw material used in industrial processes. About 250–300 m^3 is required per ton of paper produced. This wastewater contains several contaminants [3]. The pulp wastewater is produced from multiple pulping processes, including mechanical, chemical, and kraft pulping. Each method has a different process method and produces different types of effluent. These processes produce wastewater with a high concentration of resin acid, chemical oxygen demand, sulfur compounds, total suspended solids, phenolics, biochemical oxygen demand, chlorinated lignin, terpenes, ammonia, and unsaturated fatty acids [4, 5]. The color of the pulp wastewater was stated that it is brown due to the high lignin content in the water. The paper and pulp mills produce large amounts of sludge and wastewater. Fig. 1 highlights the wastewater sources in the industry alongside their potential environmental impacts.

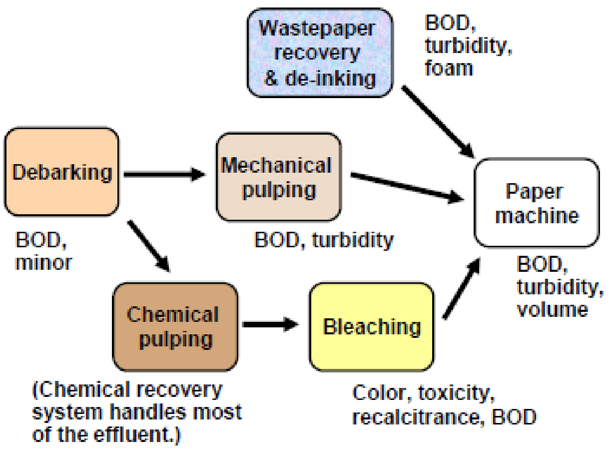


Fig. 1 Pulping processes that generate wastewater

Photocatalytic degradation, ion exchange, coagulation-flocculation, flotation, precipitation, ultrafiltration, electrochemical processes, pyrolyzed petrified sediment, membrane processes, adsorption [6, 7], nanofiltration, reverse osmosis, electrolysis, irradiation, and oxidation processes are examples of the common ways that pulping process generates effluent [8-15]. However, many of them have difficulty adhering to stringent environmental regulations.

Biological reactors are naturally sensitive to the environment, according to literature, because they require frequent and consistent monitoring, maintenance, and testing [16]. Numerous trials of biological wastewater treatment systems have produced less sludge [17, 18]. That includes sequencing batch reactors, pure culture of decolorizer, aerated lagoons, activated sludge, and oxidation ponds [19, 20]. Recently, Limits on industrial effluents have been a major concern for water quality management plans to protect the environment. For effective control, appropriate effluent restrictions are needed [21, 22]. The proper enforcement of discharge standards that also give the necessary means for legal action is critical to the success of a quality management plan.

Environmental researchers have recently been paying close attention to the sustainable use of activated sludge processes for wastewater treatment [23]. The present study focused on the start-up of the bench-scale activated sludge system fed with real industrial pulp and paper wastewater to verify the influence of HRT, wastewater concentration, and sugarcane bagasse during the treatment of paper and pulp wastewater. According to the authors' exhaustive literature search, this has never been studied before. Furthermore, the study observed the COD and ammonia removal efficiencies.

2. Materials and Methods

2.1. Materials

All chemicals utilized during the study were of the highest purity [24]. Sugarcane bagasse biomass was obtained from a local night market in the neighboring area of Seri Iskandar, Tronoh, Perak, Malaysia. The bagasse was washed with clean water to remove contaminants [25]. After drying, a mechanical granulator was used to reduce the size of the sample to

2.5–5.0 mm. The sugarcane bagasse was later grounded to 75 m powder [26]. The paper and pulp wastewater utilized was acquired from a company located in Gurun, Kedah, Malaysia. A sufficient amount of the wastewater sample was successfully gathered and stored in the cold room for preservation. The raw wastewater sample (see Fig. 2) obtained will be characterized before being used in the activated sludge system.

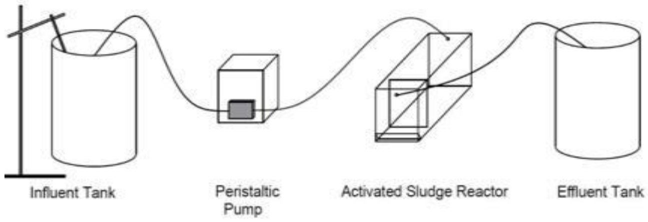
Fig. 2 Raw paper and pulp wastewater

2.2. Bioreactor and the Experimental Process

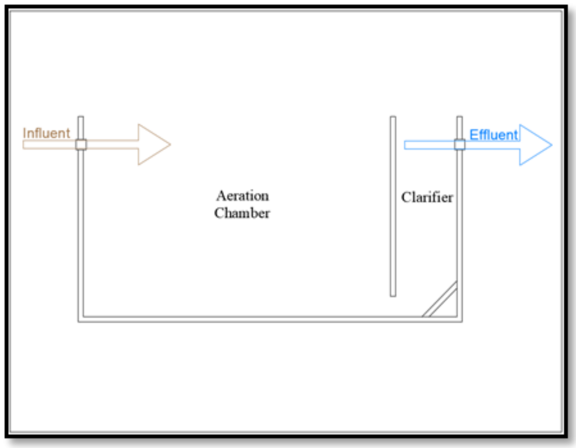
The wastewater sample is conducted using the activated sludge system set up in the laboratory. It is a slightly modified replica of the conventional wastewater treatment plants. Aerated influent and effluent tanks are connected to an aeration tank in the test design.

Peristaltic pump silicone tubing links the reactors to a 20-liter influent tank. It is critical to remove any physical things that could cause servicing or operational issues in the treatment and related systems [27]. The main bioreactor components and diagram of bioreactor components are shown in Fig. 3a and b. The bioreactor was fed with biomass obtained from a sewage treatment plant [2].

Table 1 highlights the utilized biomass characteristics. After acclimatization, the reactor was continuously operated and fed with paper and pulp wastewater [28]. The sludge age was kept under control by recycling and discarding sludge daily. The sludge age was kept under control by recycling and discarding sludge daily. The pH of the inflowing wastewater was held at the neutral value of 7.



(a)



(b)

Fig. 3 Pictorial view of (a) main bioreactor components, (b) diagram of bioreactor components

At a certain time each day, influent and effluent samples were collected, and the operation of the bioreactors was monitored regularly by measuring the COD, ammonia, MLSS, and MLVSS values. Thus, the reactor's performance efficiencies (%) were computed using equation 1 below:

$$E = \frac{M_o - M_e}{M_o} \times 100 \quad (1)$$

where E is removal efficiency (%), M_o and M_e are initial and final COD and ammonia concentrations, respectively in (mg/L) [29].

2.3. Procedure for COD and Ammonia Measurement

Generally, the organic strength of wastewater and contamination of natural waters are measured using COD [30]. It is the oxygen concentration necessary to oxidize organic compounds to CO_2 and H_2O in an acidic atmosphere using silver nitrate as a catalyst.

Table 1 Biomass characteristics

Parameter	Concentration
pH	7.7
Settleability (ml/L)	238.2
SVI (mL/g)	115.1
MLSS (mg/L)	3240
MLVSS (mg/L)	2416.3

The main merit of this test over the BOD test is that it takes only 3 hours to complete. It is possible to build a link between BOD and COD in order to assess the value of BOD quickly. The following expression can be used to find a common relationship between these two factors: BOD_5/COD for municipal wastewater = 0.5.

Start by homogenizing 500mL of water sample and using distilled water as a control. COD digestion's apex. The reagent vial was withdrawn and held at a 45° angle. A pipette was used to extract 2mL of wastewater sample into a vial, and the vial top was then tightened. The vial was placed over a sink and held in place by the cap. For combining the materials, it was gently flipped many times. It was an exothermic process when the heat was created. For two hours at 150°C, the vial was put in a preheated COD reactor. Lastly, a UV-VIS Spectrophotometer is used to assess the vial in three replicates.

Ammonia content was measured. The procedure for measuring Ammonia concentration includes diluting the wastewater sample with a dilution factor of 1:10. 25 mL of diluted sample was inserted in a measuring cylinder and mixed with three drops of mineral stabilizer, followed by three drops of polyvinyl alcohol. Then, the sample was mixed with 1 ml of Nessler reagent for one minute. Three cuvettes were filled with the mixed sample, while the blank cuvette was filled with distilled water. The program used to determine Nitrogen level is program 380 N, Ammonia, Ness in the spectrophotometer. The spectrophotometer was zeroed using the blank sample. Then, the cuvette sample mix was inserted into a spectrophotometer to read, and values were recorded.

2.4. Kinetic Modeling

Mathematical models are employed in the basic science of biological processes to test ideas, evaluate the significance of correlations among variables, direct experimental design, and decide the experiment's outcomes. These models are most often used to track and predict the treatment plant's efficiency and fine-tune the plant's setup and the effects of pilot scale-up experiments. To assess the effectiveness of the design of biological reactors, simpler and recognized models with fewer parameters are presently used, which are easier to regulate and crucial for industrial applications. The effectiveness of the reactor was predicted using the relevant, familiar bio-kinetic models. These include first and second pseudo-order models, Modified Stover–Kincannon model, etc. They will be utilized to evaluate the solute uptake rate and validate the rate-limiting phases in the biosorption process.

2.4.1. First-Order Kinetic Model

Assuming first-order kinetics prevails in steady-state conditions, the rate of change in substrate concentration in the activated sludge system can be expressed by Eq. (2) as follows:

$$\frac{W_{in}-W_{eff}}{t} = k_1 W_{eff} \quad (2)$$

By plotting $(W_{in}-W_{eff})/t$ versus W_{eff}, the value of k_1 (first-order contaminant removal rate constant) can be determined from the slope of the line.

2.4.2 Modified Stover–Kincannon Kinetic Model

Kincannon and Stover created this rotating biological contactor model based on the assumption that substrate utilization rate is proportional to organic loading rate. The updated Stover–Kincannon model takes the following broad form:

$$\frac{dS}{dt} = \frac{Q}{V_r}\left(W_{in} - W_{eff}\right) = \frac{U_{mxm}\left(\frac{QW_{in}}{V_r}\right)}{K_v + \left(\frac{QW_{in}}{V_r}\right)} \quad (3)$$

Eq. (3) will be linearized to produce Eq. (4):

$$\left(\frac{dS}{dt}\right)^{-1} = \frac{V_r}{Q(W_{in}-W_{eff})} = \frac{K_v}{U_{mxm}}\left(\frac{V_r}{QW_{in}}\right) + \frac{1}{U_{mxm}} \quad (4)$$

3. Results and Discussion

3.1. Paper and Pulp Wastewater Characteristics

The wastewater characteristics are highlighted in Table 2. The COD concentration indicates oxidizable organic materials present in the wastewater samples. That reflects the possibility of using the biosorption process with a high potential to degrade low, medium, and high-strength wastewater concentrations.

Table 2 Characteristics of the paper and pulp wastewater

Parameter	Concentration
pH	6.55-7.9
Temperature (°C)	25.1-27.4
COD (mg/L)	1039 - 5248
Ammonia (mg/L)	13.74 – 67.04

3.2. COD and ammonia removal

3.2.1. COD

The COD test is critical for determining the efficacy of the biological treatment process in the treatment plant [27]. COD intake in the reactor was consistent during the study, with minor changes when an entire load of paper and pulp wastewater was added and monitoring began. As expected, when the wastewater content was increased to around 100%, sludge generation surged dramatically. It consequentially increased the COD concentration from 3158 to 5248 mg/L. At high COD concentrations, the MLSS concentration rises. On the average and maximum, MLSS concentration in the reactor was 4094 mg/L. Inside the aeration basin, there was a direct link between influent wastewater concentration and MLSS concentration. As a result, this number may be raised depending on the wastewater properties and operating conditions. With hydraulic retention durations of 48 hours, about 98 percent of COD was removed. The COD removal was inconsistent throughout the samplings. It was observed that in the treatment system adopted for this study, the COD concentrations changed with variations in HRT values (24-72 h) once the reactor had been started and the treatment circumstances had been fine-tuned. During the 72-hour HRT, the COD reading was the least. All COD values were much below the Standard B maximum allowed limit of 200 mg/L after treatment. The findings show that air injection can speed up the biological treatment process, having the largest impact on COD removal from wastewater. According to the authors [28], the prolonged aeration reactor reduced COD concentrations more effectively. The elimination pattern of the studied parameters was linear, and it was inferred that as the strength of the wastewater increased, the system's treatment capabilities dropped. The device, however, was capable of treating larger CODs ≥ 5000 mg/L. The findings revealed that the system's influent COD concentration showed a favorable association with the MLSS and SVI.

3.2.2. Ammonia

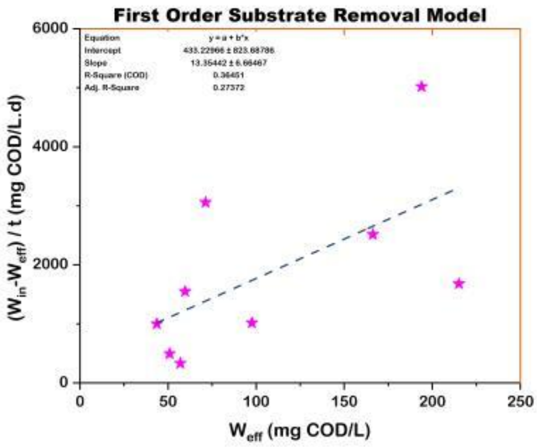
This study recorded the daily values of influent and effluent ammonia, and results were used for kinetic modeling. A generally consistent trend is shown, except for the 100% wastewater content, which was unusually high due to the arrival of some loads, which was bound to occur someday. These high values are ignored when computing typical values and assessing microbial population features. Ammonia was ~92% removed from the system due to aeration of the wastewater with a 2.94 mg/L effluent concentration. These results are consistent with the reductions recorded. It should also be highlighted that incorporating activated carbon into the reactor resulted in a significant reduction in effluent ammonia content by combining the benefits of biodegradation and adsorption. The prevalence of nitrifying bacteria, which consume ammonia and produce nitrate through an oxidation process, predicted the removal of nitrogen molecules through biological processes. Raising the retention duration in an extended aeration system allows for greater nitrogen compound utilization than activated sludge. The fact that the redesigned activated sludge system is successfully operating, as evidenced by the experimental parameters, has been further reinforced by ammonia and COD reductions that are well within the normal range. Because prolonged aeration reactors have a longer detention duration, they are better at removing COD and ammonia than activated sludge systems [28-30].

3.3. Biokinetic Models for Paper and Pulp Wastewater Treatment

When several researchers used different substrates and bioreactors, they obtained varied values of kinetic constants. The variation in mean biokinetic constants across studies suggests that reactor configuration and operational (aerobic and anaerobic) conditions, substrate concentration, substrate type, and microbial consortia presence can all significantly impact kinetic coefficient values. Numerous scholars have also developed the link between influent/initial concentration and effluent/equilibrium concentration. A few well-established kinetic models were studied to study the reactor efficiency in COD and ammonia removal.

3.3.1. First-Order Substrate Removal Model

The first-order bio-kinetic model coefficients k_1 and R^2 were calculated from the slope of the line at steady-state by plotting Eq (2). The plot yielded a straight line, as depicted in Fig. 4. COD removal constants were 13.354 and 0.3645 for k_1 and R^2, respectively, whereas ammonia removal constants were 5.9185 and 0.4691, respectively. The weak R^2 value implies that the first-order model cannot accurately forecast COD and ammonia removal rates in the bioreactor under evaluation.



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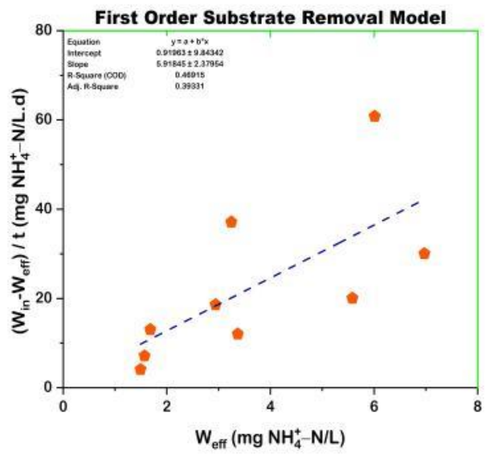
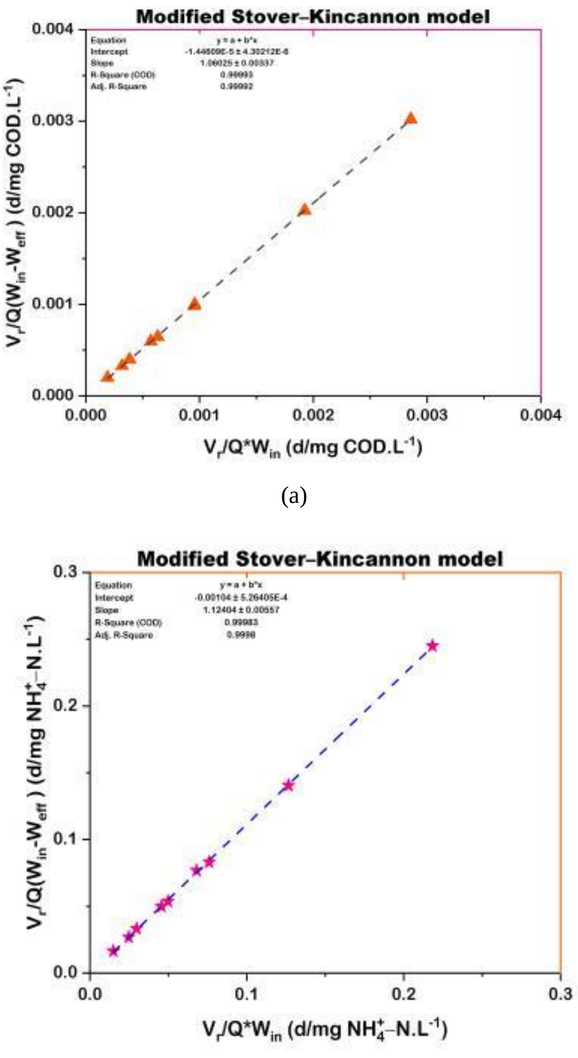


(b)

Fig. 4 The first-order kinetic model plot (a) COD (b) ammonia

3.3.2. Modified Stover–Kincannon Model

In this study, equations 3 and 4 plotting Fig. 5 to determine the biokinetic coefficients. From the line drawn on the graph, the K_v and U_{mxm} values for COD were 42.17 g/COD/L.d and 48.3 g/COD/L.d respectively, while that of the corresponding ammonia removal was 71.02 g/L/d and 77.58 g/L/d, respectively. The high K_v value suggests that the evaluated reactor effectively managed industrial wastewaters. The Stover–Kincannon model has the advantage of being able to predict COD and ammonia reduction and treatment efficacy under any loading state. It is also worth noting that the Stover–Kincannon model ignores COD and ammonia diffusion, hydraulic dynamics, and other factors.



(a)

(b)

Fig. 5 Stover–Kincannon model plot (a) COD (b) ammonia

4. Conclusion

The biosorption of COD and ammonia from paper and pulp effluent utilizing an activated sludge system with extended aeration was explored in this study. Adding activated carbon medium to the system's performance has also been investigated. Wastewater concentrations were set at 10, 50, and 100%. The efficiency was measured by examining the samples' influent and effluent quality. The effectiveness of the treatment process was determined by the amount of COD and ammonia removed. Findings revealed high removal efficiencies up to 98.11% and 92.67% for COD and ammonia, respectively. After treatment, effluent concentrations for both parameters have satisfactorily met the Standard "A" standard limits for industrial discharge after 48 hours HRT. Therefore, further testing is not required. The First order and Modified Stover-Kincannon models were used to evaluate substrate removal rates. In the Modified Stover–Kincannon model, high correlation coefficients R^2 of 0.9999 and 0.9998 were obtained for COD and ammonia, respectively. The modified Stover-Kincannon model proved to best suit the experimental data. Therefore, the model can be used to design the evaluated system and predict the bioreactor behavior.

4.1. Acknowledgment

The authors would like to acknowledge the support received by Universiti Teknologi PETRONAS, Malaysia, under the Graduate Assistantship (GA) scheme.

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