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Treatment of Fish Processing Wastewater using the Multi Soil Layering Method

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Abstract

The wastewater generated by fish-processing industries in Lamongan Regency contains TSS, BOD_5 and COD concentrations that exceed the quality standards specified in East Java Provincial Regulation No. 72/2013. The media in the Multi Soil Layering method consist of a mixture of soil and charcoal, along with a permeable layer. This research aims to analyze the effectiveness of MSL method with variations in soil type, Hydraulic Loading Rate, and sampling time. The results indicate that red soil is the most effective in removing TSS 89,26%, while andosol soil is more efficient in reducing BOD_5 42,86% and COD 68,33%. The physical, chemical, and mineral properties of each soil type significantly influence pollutant removal performance. Pollutant removal at an HLR of 0,2 m^3/m^2.hour was more optimal compared to 0,4 m^3/m^2.hour, as the lower HLR allows longer contact time between the wastewater and the media in the reactor. Sampling at the 7-hour mark yields optimal efficiency, indicating that the media had not yet reached saturation.

Keywords: *fish processing wastewater, multi soil layering, soil type, hydraulic loading rate, sampling time*

Abstrak

Limbah cair yang dihasilkan oleh industri pengolahan ikan di Kabupaten Lamongan mengandung konsentrasi TSS, BOD_5, dan COD yang melebihi baku mutu dalam Peraturan Gubernur Jawa Timur No. 72 Tahun 2013. Media pada metode *Multi Soil Layering* (MSL) terdiri atas campuran tanah dan arang, serta lapisan permeabel. Penelitian ini bertujuan untuk menganalisis efektivitas metode MSL dengan variasi jenis tanah, *Hydraulic Loading Rate* (HLR), dan waktu pengambilan sampel. Hasil penelitian menunjukkan bahwa tanah merah paling efektif dalam menurunkan TSS sebesar 89,26%, sedangkan tanah andosol lebih efisien dalam menurunkan BOD_5 sebesar 42,86% dan COD sebesar 68,33%. Sifat fisik, kimia, dan mineral dari masing-masing jenis tanah berpengaruh terhadap kinerja penyisihan beban pencemar. Penyisihan beban pencemar pada HLR 0,2 m^3/m^2.jam lebih optimal dibandingkan 0,4 m^3/m^2.jam karena HLR yang lebih rendah memberikan waktu kontak yang lebih lama antara air limbah dan media di dalam reaktor. Pengambilan sampel pada jam ke-7 menghasilkan efisiensi yang optimal, yang menunjukkan bahwa media belum mencapai kondisi jenuh.

Kata Kunci: *limbah cair pengolahan ikan, multi soil layering, jenis tanah, hydraulic loading rate, waktu sampling*

1. Introduction

Lamongan Regency is one of the regions that serves as a center for the marketing and processing of fishery products [1]. The development of the fish processing industry, both large-scale and household-based, has contributed to local economic growth. However, the increase in production has also led to a rise in waste volume, particularly wastewater generated from fish processing activities [2]. This wastewater contains pollutant loads that have the potential to contaminate water bodies if not properly treated.

Since early 2025, the results of wastewater quality tests from the fish processing industry in Lamongan have shown that BOD_5, COD, and TSS levels exceed the standards set by Governor Regulation No. 72/2013 [3]. One potential technology that can be applied is the Multi Soil Layering (MSL) method. This method is a wastewater treatment system based on filtration and adsorption processes using layers of soil, gravel, and additional media such as zeolite and perlite. The advantages of MSL include cost efficiency, land-use flexibility, and ease of implementation, even at the household scale [4].

The effectiveness of the MSL method is influenced by the type of soil used as the main medium. Soil functions not only as a filtration medium but also as a habitat for microorganisms that support the biodegradation process [4]. The physical and chemical characteristics of soil, such as porosity, organic matter content, and mineral composition, affect its ability to reduce pollutants. Previous studies have shown that andosol soil is effective in reducing BOD_5 and COD due to its high adsorption capacity and organic content that supports microbial activity [5]. In addition to andosol, red soil (laterite) and water treatment plant sludge (IPAM sludge) also have potential as filtration media [6]. The efficiency of the MSL system is also influenced by operational parameters such as Hydraulic Loading Rate (HLR) and retention time [7].

The increasing activities of the fish processing industry have resulted in a higher volume of wastewater. Therefore, an efficient, low-cost, and environmentally friendly treatment method is required. The Multi Soil Layering method is a physical treatment approach based on layered filtration, using soil as the main medium along with supporting materials. The type of soil significantly affects system performance, as physical properties such as porosity, adsorption capacity, and organic content determine the ability to reduce pollutants. Operational factors such as Hydraulic Loading Rate and sampling time also play an important role [8]. To test these hypotheses, research on Multi Soil Layering as a physical method for treating fish processing wastewater is necessary in order to prevent pollution of aquatic ecosystems.

2. Material and Methods

This study used a reactor system consisting of a wastewater storage tank, shown in **Figure 1** (no.1), which was equipped with a pump, as shown in number 2. The pump functioned to supply wastewater into the MSL reactor presented in number 3, which had dimensions of 30 × 20 × 70 cm. In addition, an outlet tap was installed at the upper part of the reactor as a sampling point to evaluate the wastewater treated using the MSL method. The media used in this study consisted of a permeable layer made of gravel and zeolite, as well as soil mixture blocks composed of a mixture of soil and charcoal.

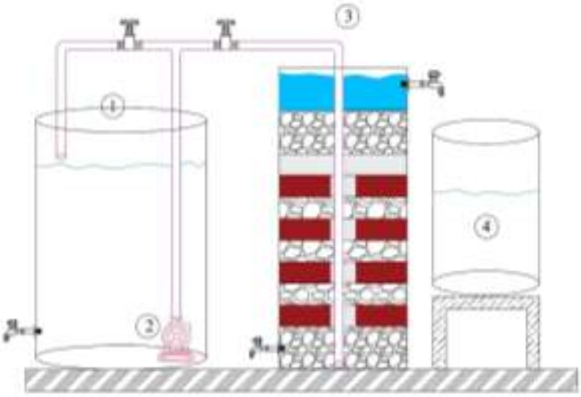

Figure 1. Multi Soil Layering reactor

The independent variables in this study were threefold: soil type, Hydraulic Loading Rate (HLR), and sampling time. The soil types used were andosol soil, water treatment plant sludge (IPAM sludge), and red soil. The HLR variations applied were 0.2 and 0.4 $m^3/m^2 \cdot hour$. The sampling times were 0, 3, and 7 hours. The parameters observed were Total Suspended Solids (TSS), Biochemical Oxygen Demand (BOD_5), and Chemical Oxygen Demand (COD), as these parameters exceeded the wastewater quality standards for the fish processing industry.

3. Results and Discussion

Wastewater from the fish processing industry contains high levels of organic matter and pollutants. This waste has been utilized for products such as fish meal, fish oil, animal feed, and organic fertilizer [9]. However, it has the potential to harm the environment if not properly managed. The presence of organic compounds and dissolved nutrients can contaminate drainage systems, rivers, and other water bodies.For example, wastewater from fish processing in Lamongan Regency, which is the object of this study, contains TSS, BOD_5, and COD parameters that exceed the quality standards stipulated in Governor Regulation No. 72 of 2013.

Table 1. Initial Wastewater Test Results

Parameter	Standard (mg/L)	Test Results in Wastewater (mg/L)	Remarks
TSS	30	121,6	Not Compliment
BOD_5	100	156,5	Not Compliment
COD	150	320,0	Not Compliment

Results of Soil Mineralogical Content Analysis

Fishery wastewater contains various chemical components, including positively and negatively charged ions derived from fish biological activities as well as dissolved inorganic compounds [10]. To treat this wastewater, this study employed the multi soil layering method using media consisting of gravel, zeolite, and soil mixture blocks. SMB is a medium in the multi soil layering system composed of a mixture of soil and charcoal. It is known that soil can treat wastewater, one mechanism being cation and anion exchange [11]. As the wastewater passes through the soil media, the ions in the wastewater begin to interact with the minerals present in the soil.

X-ray diffraction (XRD) is a technique used to identify the type and structure of minerals through the diffraction patterns produced [12]. In this study, XRD analysis was used to determine the mineral composition of the soils forming the soil mixture blocks. The identification of minerals helps to understand their role in pollutant reduction processes. This information also indicates the relationship between the physicochemical properties of the soil, such as ion exchange capacity, adsorption capacity, and interactions with organic compounds. Therefore, XRD analysis can help elucidate the mechanisms of pollutant removal in the MSL method. The following are the XRD test results for each type of soil:

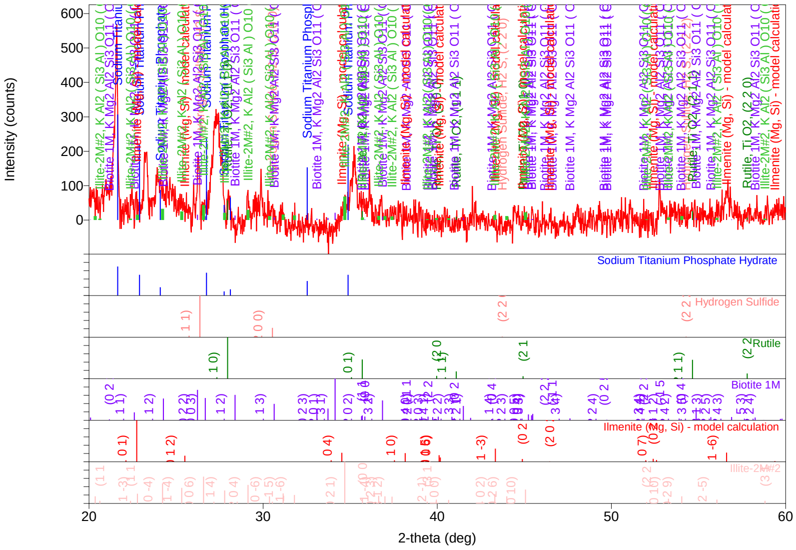

Figure 2. XRD Test Results of Andosol Soil

The graph between angles of 20° and 60° shows diffraction peaks, where at an angle of 21° the highest intensity peak is observed at 339 cps, indicating the presence of the mineral sodium titanium phosphate hydrate ($Na_2TiPO_4 \cdot 3H_2O$). In addition, similar mineral peaks are also detected at angles of 23° (88 cps), 27° (199 cps), and 35° (83 cps). Sodium titanium phosphate appears as the dominant peak, indicating that this mineral is the main component in the sample. The characterization of sodium titanium phosphate has been tested for its ability to bind various elements in simulated waste, including heavy metals and fission products. XRD and SEM-EDX results show that sodium titanium phosphate is capable of retaining up to 15% by weight of waste within its crystal structure, with a homogeneous distribution of waste elements. Leaching resistance tests demonstrate that sodium titanium phosphate has good chemical stability, preventing hazardous elements from being easily released into the environment [13].

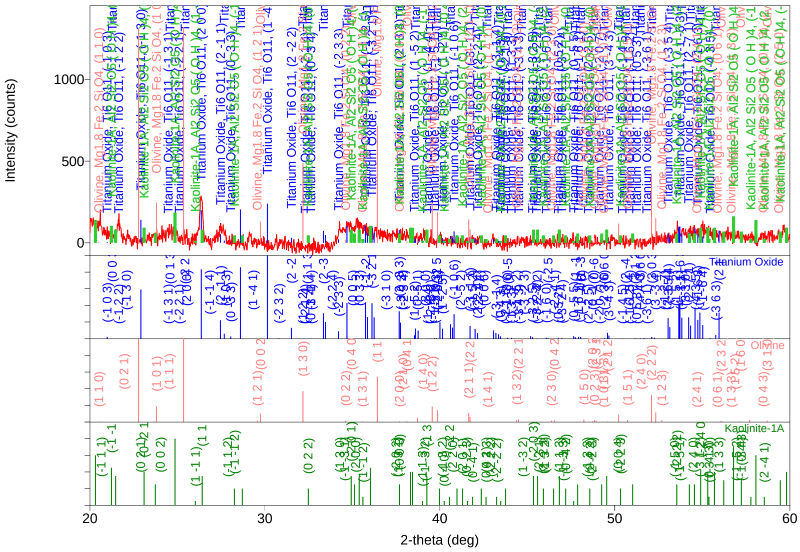

Figure 3. XRD Test Results of Sludge IPAM

The XRD peak list graph of IPAM sludge shows five diffraction peaks, indicating the presence of $Ti_6O_{11}Mg_{1.8}$, Ti_6O_{11}, and $Ti_6O_{11}Al_2Si_2$ compounds. Thus, it can be inferred that magnesium, aluminum, and silicon elements are likely incorporated into the crystal lattice of titanium oxide (Ti_6O_{11}). The presence of titanium oxide (Ti_6O_{11}) is observed at angles of 24°, 26°, 27°, 35°, and 54°. A study on the synthesis and characterization of titanium oxide–clay-based composites reported that this material was tested as both an adsorbent and a photocatalyst in a single-stage process. Adsorption and photocatalysis tests demonstrated that the material effectively removes pollutants, both through surface adsorption and photodegradation under UV radiation[14]. In addition, the presence of aluminum (Al) in the sludge, which is alkaline in nature, also contributes to the neutralization of wastewater [6].

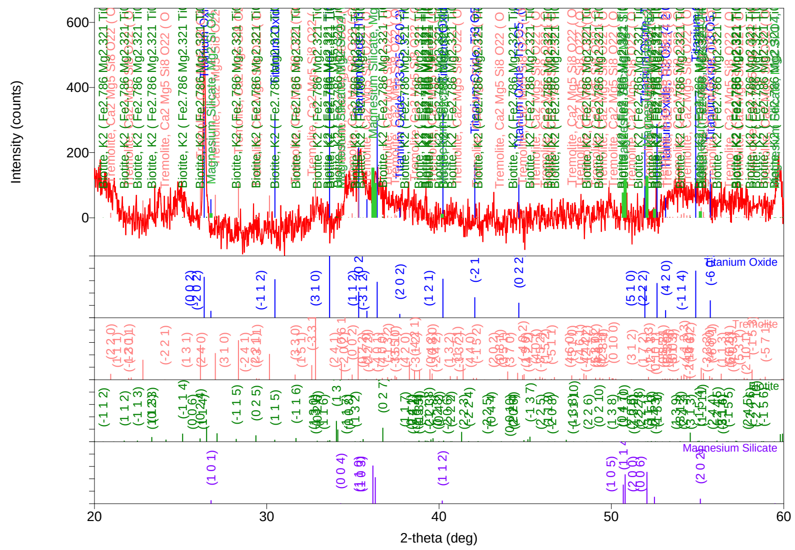

Figure 4. XRD Test Results of Red Soil (Laterite)

The XRD analysis of red soil shows that the main identified mineral is titanium with the chemical formula $Ti_3O_5Ca_2Mg_5$. The primary diffraction peaks appear at 2-theta angles of 26.37°, 35.37°, and 54.28°, indicating the dominant presence of this compound in the sample. The $Ti_3O_5Ca_2Mg_5$ mineral identified in the red soil sample has a chemical composition and crystal structure different from that of IPAM sludge. Analysis using XRD, FTIR, AFM, and SEM on titanium oxide–clay composites reveals the presence of anatase and rutile phases uniformly distributed on the soil surface. These phases have a large surface area and carry a negative charge under alkaline pH conditions, making them effective for adsorbing metal ions through interactions with hydroxyl groups on their surface [14]. In addition, the presence of magnesium contributes to increasing the surface area and the number of active sites in the material, thereby enhancing its pollutant adsorption capacity [15].

Analysis of the Effects of Soil Type, HLR, and Sampling Time on the Removal of TSS, BOD_5, and COD

The TSS, BOD_5, and COD parameters in fish processing wastewater show concentrations that exceed the quality standards based on the Regulation of the Governor of East Java Number 72 of 2013. High levels of TSS, BOD_5, and COD can disrupt the balance of aquatic ecosystems. Therefore, controlling TSS, BOD_5, and COD is an important aspect of wastewater treatment. The results present the percentage removal achieved by the multi soil layering method with variations in soil type, hydraulic loading rate (HLR), and sampling time on the reduction of TSS, BOD_5, and COD.

Table 2. Percentage Removal Results of TSS, BOD_5, and COD Parameters

Soil Type	HLR (m^3/m^2.day)	Sampling Time (hours)	TSS Removal (%)	BOD_5 Removal (%)	COD Removal (%)
Control	0,2	0	48,76	15,38	31,25
		3	55,00	20,00	32,50
		7	62,63	20,00	35,00
	0,4	0	42,57	13,85	25,00
		3	44,78	13,85	27,50
		7	51,50	16,67	28,75
Andosol Soil	0,2	0	76,96	36,00	58,40
		3	80,52	41,33	61,67
		7	87,34	42,86	68,33
	0,4	0	70,04	30,67	51,67
		3	73,66	31,43	55,00
		7	80,70	36,00	60,00
Sludge IPAM	0,2	0	76,86	30,67	56,25
		3	80,50	31,43	60,94
		7	86,67	37,14	66,41
	0,4	0	72,27	25,33	48,44
		3	74,15	30,67	51,56
		7	79,32	31,43	57,81
Red Soil (Laterite)	0,2	0	81,27	33,33	54,29
		3	83,56	33,33	58,57
		7	89,26	34,12	63,57
	0,4	0	72,09	28,89	48,57
		3	75,67	29,41	51,43
		7	81,25	29,41	54,07

In the multi soil layering method, gravel functions as a separator for large-sized particles, a supporting medium, and a base layer in the filtration reactor [16]. In this layer, a physical process in the form of filtration occurs, where suspended particles in the wastewater are retained within the gaps between gravel grains. In the TSS removal mechanism, the media in the reactor are arranged by placing larger particles in the lower layer and smaller particles in the upper layer. The same principle is applied in this study by positioning gravel at the base layer and zeolite in the subsequent layer. This combination enhances the effectiveness of the filtration process, as zeolite is capable of trapping fine particles within its pores, thereby reducing the load on the subsequent media layers [17].

Figure 5. Soil Mixture Block

SMB is a composite medium of soil and charcoal that functions as a filtration layer in the MSL system. In this study, physically activated wood charcoal was used, and SEM analysis showed the presence of pores that serve to adsorb pollutant loads in wastewater [18]. The reduction in TSS levels is also influenced by the ability of sawdust, straw, and coconut shell charcoal to filter fine particles [19]. [20] further stated that suspended particles can be removed through physical filtration processes occurring on the surface of the MSL layers. In this study, the control variation consisting of gravel and zeolite media achieved a TSS removal efficiency of 42,57–62,63%. Meanwhile, variations with the addition of SMB along with gravel and zeolite showed better performance, with removal efficiencies reaching 70,04–89,26%. These results indicate that SMB plays an active role in enhancing the filtration process in the MSL system.

Figure 6. Comparison of Wastewater Before and After Treatment with Andosol Soil Type

The SMB variation using red soil was newly applied for TSS reduction. In a previous study [21], this medium was only used to reduce phosphorus with an efficiency of 23%. However, the SMB variation with red soil produced the highest TSS reduction, reaching 89,26%. This is attributed to the fine and dense texture of red soil, which forms small and uniform pores [22]. Such conditions allow suspended particles to be more effectively retained within the media layers. XRD analysis results indicate the presence of titanium oxide minerals in the red soil; however, this content does not significantly influence TSS reduction. This is because mineral content plays a more prominent role in ion exchange processes occurring with dissolved particles [23]. Therefore, TSS removal primarily occurs through physical filtration mechanisms.

The highest reduction of BOD_5 and COD was observed in andosol soil, with removal efficiencies of 42,86% and 68,33%, respectively. This performance is influenced by the physical properties of andosol, which has a large and abundant pore structure, providing a suitable habitat for soil microorganisms [4]. Microorganisms growing within these pores form a biofilm layer [20]. Microbial activity within the biofilm enables the adsorption and degradation of organic matter in the wastewater. This process supports biological adsorption, which contributes to the reduction of BOD_5 and COD levels.

In the study by [24], BOD_5 levels were reduced by up to 99% at an HLR of 250 L/m²·day. At HLRs of 500, 1000, and 1500 L/m²·day, the efficiencies reached 98%, 96%, and 86%, respectively. The present study shows a similar pattern, where the best performance was obtained using SMB composed of andosol soil and physically activated wood charcoal through high-temperature combustion. At an HLR of 0,2 m³/m²·hour, BOD_5 reduction ranged from 36% to 42,86%, while at an HLR of 0,4 m³/m²·hour, the reduction ranged from 30,67% to 36%. These results indicate that SMB made from andosol soil and charcoal can reduce BOD_5 through adsorption within the pores of the andosol soil and wood charcoal. Additionally, it can be observed that higher HLR values lead to lower pollutant removal efficiency, and vice versa. This is because lower HLR results in longer contact time, enhancing the treatment process.

Zeolite and gravel used as media in the MSL method are part of the aerobic zone. Pollutants in the wastewater adhere to the surface of zeolite and gravel, resulting in an adsorption process. This occurs because zeolite has active hydroxyl groups on its surface, such as (≡Si–OH) and (≡Al–OH), which play an important role in adsorption [4]. These hydroxyl groups form silanol and aluminol surfaces with large surface areas, allowing all active sites to be easily accessible [25]. Zeolite also contains cations such as Na^+, K^+, Mg^{2+}, Sr^{2+}, and Ba^{2+} [26], which can be exchanged with other cations. In this study, the wastewater was found to contain NH_4^+ and Cl^- ions based on previous tests. The NH_4^+ ions in the wastewater can be exchanged with cations present in the zeolite. With this capability, zeolite acts as an effective ion exchanger for ammonium in wastewater. In a study by [27], non-activated zeolite was able to adsorb NH_4^+ up to 90,26%.

Soil is a natural resource capable of treating both solid and liquid waste through a combination of physical, biological, and biochemical processes. These processes include adsorption, desorption, precipitation, solubilization, cation and anion exchange, and microbial decomposition [11]. In line with this, soil can also perform ion exchange through its mineral content. XRD results show that andosol soil contains sodium titanium phosphate hydrate, while IPAM sludge and red soil are dominated by titanium oxide. Andosol soil has been proven to be more effective in reducing BOD_5 and COD compared to other soil types. The characterization of sodium titanium phosphate in andosol indicates that this material can bind various elements in simulated waste and is capable of retaining up to 15% by weight of waste within its crystal structure [13]. In addition, the chemical properties of andosol soil exhibit high cation and anion exchange capacity [5].

XRD analysis indicates that andosol soil contains positive ions, particularly Na^+. Meanwhile, fish processing wastewater is known to contain NH_4^+ and Cl^- ions. Minerals in andosol soil can exchange cations with NH_4^+ ions present in the wastewater. The general cation exchange reaction is as follows [28]:

$$M\text{-}(Na^+) + NH_4^+ \rightleftharpoons M\text{-}(NH_4^+) + Na^+ \qquad \text{.....(1)}$$

The specific reaction occurring between minerals in andosol soil and fish processing wastewater in this study is:

$$Na_2Ti(PO_4)_2{\cdot}3H_2O + 2NH_4^+ \rightleftharpoons (NH_4)_2Ti(PO_4)_2{\cdot}3H_2O + 2Na^+ \qquad \text{.....(2)}$$

These cations are adsorbed and then exchanged with other cations. This substitution process is known as cation exchange [25].

Ammonium ions (NH_4^+) in wastewater can undergo nitrification, a process that converts NH_4^+ into NO_2^- and subsequently into NO_3^- [29]. All stages of this process require oxygen. When NH_4^+ ions in the wastewater are exchanged with Na^+ ions in the soil, the remaining NH_4^+ concentration in the wastewater decreases. The reduction of NH_4^+ leads to a lower oxygen demand for the nitrification process. This condition indirectly reduces BOD_5 and COD values, as both parameters represent the oxygen demand required to oxidize organic compounds in wastewater. Therefore, the removal of NH_4^+ through ion exchange can reduce the organic load requiring oxidation, thereby indirectly contributing to the decrease in BOD_5 and COD.

4. Conclusion

Differences in pollutant removal effectiveness were influenced by the physical and chemical properties of the soil. Among the soil variations, red soil achieved the highest TSS reduction efficiency (89.26%) due to its fine texture and small pores that effectively retained suspended particles. Meanwhile, andosol soil was the most effective in reducing BOD_5 (42.86%) and COD (68.33%) because its larger pore structure enhanced filtration, adsorption, and microbial biofilm growth. An HLR of 0.2 $m^3/m^2{\cdot}$hour provided better pollutant removal than 0.4 $m^3/m^2{\cdot}$hour, as the lower flow rate increased wastewater contact time with the MSL media. The 7th-hour sampling also showed the highest reduction efficiency, indicating that the media had not yet reached saturation and remained effective for longer operation.

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