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*RESEARCH ARTICLE*

# ANALYSIS OF THE COMBINED ADSORPTION-MICROFILTRATION (AMFIL) PROCESS FOR WWTP EFFLUENT TO PROCESS WATER

**Sri Rulianah and Prayitno**

1. Chemical Engineering Department, State Polytechnic of Malang, Indonesia.

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## *Abstract*

The seaweed processing industry produces Alkali Treated Cotton Chips (ATCC) requires large amounts of process water used for the washing and soaking processes, where process water is generally obtained from groundwater. On the other hand, wastewater from the production process after being treated in Wastewater Treatment Plant (WWTP) and meeting quality standards is discharged into the environment. The effluent of WWTP in large volumes (150 - 200 $M^3$ per day) contains TDS, TSS, turbidity, pH, odor, and organic matter that are relatively small, so it has the potential to be reused as process water. The aims of the research are to investigate the removal of pollutants from the combined adsorption and membrane microfiltration (AMFIL) process on a pilot plant scale from the effluent of WWTP in the seaweed processing industry to process water. The real effluent from the WWTP flows into an adsorption column containing granular activated carbon with varying weights and sizes, then the effluent from the adsorption column flows into a membrane microfiltration with varying weights and sizes membrane. Parameters measured in this study were turbidity, TDS, total hardness, $Fe^{+2}$, $Mn^{+2}$, and pH. Using an adsorption capacity of 0.352 $mg.g^{-1}$ and the size of the membrane is 5µm can obtain the total removal pollutants of the AMFIL processasturbidity, TDS, total hardness, $Fe^{+2}$, $Mn^{+2}$, and pH were 86.67%, 94.00%,95.83%,96.36%,93 .33%, and (6.5 - 7.5), respectively.



## Introduction:-

In the seaweed processing industry, producing ATCC (Alkali Treated Cotton Chips) requires a large amount of processed water, whereas for a production capacity of 2tons of ATCC per day requires process water of ± 150 $M^3 \cdot day^{-1}$. Process water is used for the raw material washing process and the seaweed soaking process. Process water is obtained from groundwater and continuous withdrawal of large amounts of groundwater can result in a decrease in environmental quality, in the form of subsidence, landslides, and an imbalance in the distribution of groundwater usage. In addition, excessive groundwater extraction can result in high production costs.Effluent from the WWTP process has a large volume and meets quality standards and is discharged directly into the aquatic environment (river). The characteristics of WWTP effluent are odorless, color (15 - 25 Pt-Co), Turbidity (8 - 14

**Corresponding Author:-**Prayitno
**Address:-**Chemical Engineering Department, State Polytechnic of Malang, Indonesia.

NTU), pH (6 - 7), TSS (20 - 30 mg·$L^{-1}$), TDS (4,000 - 4,500 mg·$L^{-1}$), Total hardness (700 - 900 mg·$L^{-1}$), BOD (30 - 40 mg·$L^{-1}$), and COD (60 - 80 mg·$L^{-1}$). By considering the potential and characteristics of WWTP effluent and problems in obtaining process water, the WWTP effluent has the potential to be reused as process water through a combination of adsorption (GAC) and microfiltration (MF) processes which is known as the AMFIL process.

Granular activated carbon (GAC) fixed bed adsorption technology was applied to remove carbamates carbaryl, methomyl, and carbofuran from a public water supply. To minimize the effect of clogging and to evaluate adsorbent saturation for carbamates, microfiltration (MF) was previously used to adsorb, and the backwash procedure of the GAC bed was carried out. The MF of the water previously adsorbed in the GAC fixed bedcan removal of 100 % of the carbamate's pesticides with an initial concentration of 25 µg.$L^{-1}$ during the first 48 h of operation(Alves et al., 2020).The combination of adsorption and microfiltration processes has the ability to remove contaminants in water such as hardness, nitrate, and heavy metals(Bakalár et al., 2019).The combination of adsorption and deep-bed filtration proved to be a robust and economic process combination for the removal of micropollutants with economic advantages over membrane filtration(L Pillay & Jacobs, 2007).PAC/Alum)/MF achieved 75% to complete removal of total microcontaminants with 4-18 mg. $L^{-1}$ of a mesoporous PAC and 2 h contact time, with a reliable particle separation (turbidity < 0.03 NTU) and low aluminum residuals(Campinas et al., 2021).

In wastewater treatment, membrane technology is called a major technology in the field of water treatment in the 21st century. With the development of membrane technology and the development of other emerging technologies in combination, microfiltration membrane technology is widely used in the treatment of various types of wastewaters such as radioactive wastewater and heavy metal wastewater(Abdel-Fatah, 2018; Wang et al., 2020).The physicochemical characterization of this sample before and after treatment showed a remarkable increase in removal rate from 1 hour to 3 hours of filtration; 54.8% after 1 hour and 73.28% after 3 hours for COD (73.28%) after 3 hours for BOD5 (26.48%) after 3 hours of filtration for conductivity and a total elimination rate of turbidity that was achieved 99% after 3 hours of treatment(El Khalfaouy et al., 2017).

The combination of coagulation and adsorption processes for fulvic acid uptake shows that the effectiveness of fulvic acid uptake is quite effective compared to the coagulation process. This system provides sufficient contact time for PAC to adsorb organics compared to the system with in-line adsorbent addition. More than 85% of FA was removed from water containing 8 mg·$L^{-1}$ of FA.The removal efficiency also increased with the increase in mixing intensity and mixing time. The permeate flux slightly improved when a membrane of pore size 0.22 µmwas used with shorter hydraulic residence time and lower PAC concentration so from this study it is evident that the combined membrane process has a potential application for organic dye removal(Jirankova et al., 2010).

The study on the effect of powder-activated carbon (PAC) adsorption on microfiltration (MF) membrane performance are showed that PAC pretreatment offered high organic matter removal rates for both dissolved organic carbon (DOC) and ultraviolet absorbance at 254 nm (UV254) during 10–200 mg. $L^{-1}$ PAC dosage. The removalefficiencies of organic matter by MF membrane filtration decreased with the increase of organic matter removal rate by PAC adsorption. PAC mainly removed organic matter of about 3 kDa molecular weight (MW). MF membrane maintained more than 5 kDa MW organic matter on the membrane after PAC adsorption. The results of membrane filtrationindicated that PAC pretreatment slightly promoted membrane flux, regardless of PAC dosage. It seems that the organic matter fouling membrane was concentrated in more than3 kDa MW. PAC removed markedly less than 3 kDa MW organic matter and had less effect on more than 3 kDa organic matter. Thus, PAC cannot reduce membrane fouling Löwenberg, 2016).The AF2B/GAC process was able to reduce contaminants such as TSS, BOD, COD, NH3N and Chlorine by 98 %, 99 %, 97.3 %, 97.8 %, and 100 % respectively. Furthermore, in the CNTs process, all pollutants are not detected until they meet drinking water quality standards(Prayitno et al., 2022).

## Materials and Methods:-

### Materials and equipment preparation:-

The feedwater used in this study is effluent from WWTP in the seaweed processing industry, where the feedwater meets quality standards. Feed water contains odorless, color (15 - 25 Pt-Co), Turbidity (8 - 14 NTU), pH (6 - 7), TSS (20 - 30 mg·$L^{-1}$), TDS (4,000 - 4,500 mg·$L^{-1}$), Total hardness (700 - 900 mg·$L^{-1}$), BOD (30 - 40 mg·$L^{-1}$), and COD (60 - 80 mg·$L^{-1}$). Furthermore, feedwater is fed into the influent column of 2,000 L to have flowed into the treatment system as fed in the AMFIL process which operates continuously as Fig. 1.The equipment used in this study includes an adsorptioncolumn, microfiltration column, pump, and storagecolumn. The adsorption column was made from PVC material with a diameter of 25 cm, a height of 150 cm, and at the top of the column, there is a distributor for feedwater.The adsorption columncontaining granular activated carbon (GAC) of 20 - 25 kg thatGAC from YUJIA production had specifications: total ash content max 4%, moisture content max 5%, apparent density 0.4 -0.5 $g \cdot cm^{-3}$, mean particle diameter 4 - 8 mesh, iodine number 900 $mg \cdot g^{-1}$. While the microfiltration membrane used cellulose membrane is type UF 280 with specifications: PVC Alloy; Shell Material: U-PVC; Size: 8 x 40 inch; Filtration Method: Inside - Out; Flow Rate: 1500 - 2500 $L \cdot h^{-1}$; Area: 28 $m^2$; In/Out: 2″; pore diameter of 1 – 10 µm.

**Operational procedure:-**

Real wastewater discharged from the WWTP in the seaweed processing industry was used as feed water in this study, where 2,000 L of feed water was collected in a storage column, and sampling was carried out to analyze the characteristics of the feed water.Furthermore, the feed water is flowed by a pump into an adsorption columnthat already contains 20 kg of granular activated carbon and membrane size 5 µm. The effluent of the adsorption column is collected in a temporary storage column which is then sampled to analyze the effluent characteristics. The effluent from the adsorption column is pumped using a jet pump of 1-10 bar into the microfiltration column where the 2-membrane tube is arranged in parallel.The effluent from the membrane tube (filtrate), hereinafter known as process water, is sampled, and analyzed for its characteristics. The experiment was repeated using the weight of the adsorbent 25 kg, and the number of different membrane tubesas 10 µm.

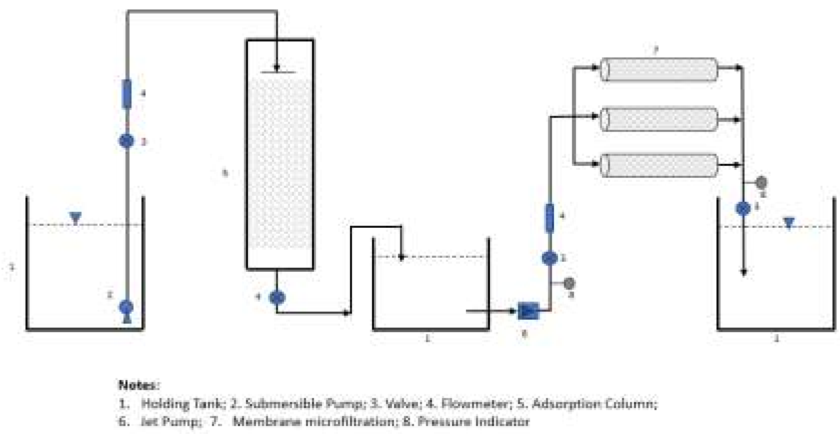



**Figure 1. The Process Flow of AMFIL System**

**Analysis Method:-**

The samples were analyzed by using APHA standard procedures to investigate the pH, Ferro ($Fe^{+2}$), Mangan ($Mn^{+2}$), Turbidity, TDS, and Total Hardness. The pH was measured using a Fisher Scientific type pH meter (Model XCL25) previously calibrated with pH 6,7, and 8 standard solutions. $Fe^{+2}$ and $Mn^{+2}$were measured using UV-Vis's spectrophotometry(Shimadzu UV-1280). $Fe^{+2}$ analysis used the phenanthroline method, while $Mn^{+2}$ analysis used the persulfate method. $Fe^{+2}$ testing can be done using the phenanthroline complexing compound and analyzed at a wavelength of 480 nm – 560 nm. By making a standard curve, the concentration of $Fe^{+2}$ in the sample can be determined.While determination of manganese in samples was carried out by the persulfate method at a wavelength of 400 - 700 nmaccording to the APHA standard method, with a calibration curve including standards of 0.5, 1.0, 1.5, 2.0 and 2.5 $mg \cdot L^{-1}$.

The determination of Total DissolvedSolids (TDS), according to the analytical method (MA115-S.S. 1.2), was carried out by filtering a sample portion (normally 100 ml) through a previously dried and weighed Whatman 934 AH glass microfiber filter (pore size 0.5µm) under vacuum. When the filtration was completed, the residue was dried at 103-105 C overnight or at least 1hour. The weight of dissolved solids is obtained by calculating the difference of the filter weights before and after drying. To determine pH using an electrical conductivity method in the form of measuring instrument pH Meter EZ-9910. The quality standard used as a reference is based on the Decree of the Minister of Health of the Republic of Indonesia No.32 of 2017.Furthermore, to determine the adsorption capacity of total dissolved solids in the adsorption column, it is estimated to use equation 1.

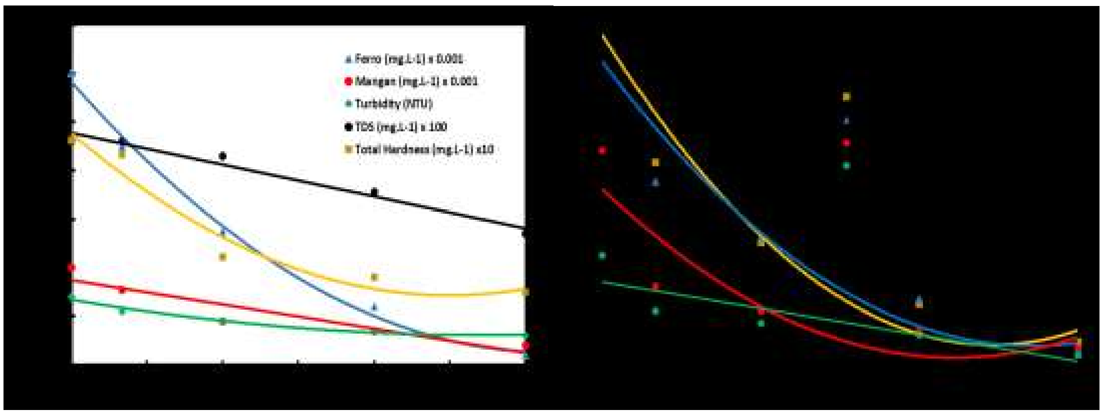
$q_t = (C_o - C_t)(V/m)$…………… (1)

**Which:**
$q_t$ is the adsorption capacity in $mg \cdot g^{-1}$.
$C_o$ is the TDS initial concentration in $mg \cdot L^{-1}$
$C_t$ is the TDS concentration in $mg \cdot L^{-1}$attime.
V is the solution volume in liter
M is the adsorbent mass in gram

## Results and Discussion:-

### The effect of adsorbent weight:-

In the AMFIL process, especially in the adsorption column containing granular activated carbon of 5 µm, using an adsorbent weight of 20 kg and 25 kg and different feed characteristics, experimental results were obtained as shown in Figure 2.




(a) (b)

**Figure 2. The performance of the adsorption column at different adsorbent weights**
**a. Weight 20 kgb. Weight 25 kg**

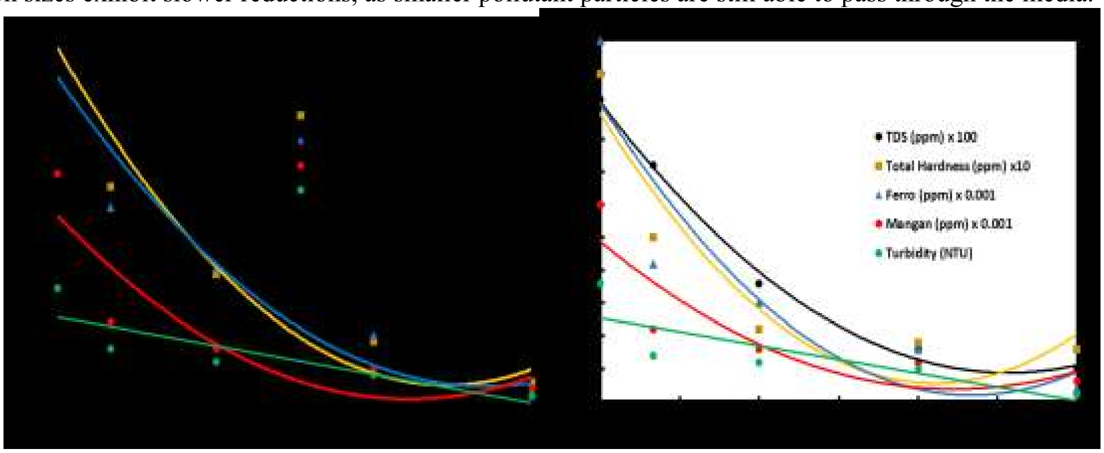
Figure 2(a) indicates that as the adsorption time increases, the concentration of each pollutant decreases, with iron and total hardness showing particularly substantial reductions. Similarly, Figure 2(b) demonstrates that longer adsorption durations lead to greater decreases in pollutant concentrations in the water, with all parameters significantly declining except for turbidity. Additionally, the most pronounced reduction in concentration occurs within the first 60 minutes of the adsorption process. Thus, the mass of the adsorbent significantly affects its adsorption capacity, as increasing the amount of adsorbent provides a larger surface area for interaction between the adsorbent and the adsorbate.The adsorbent surface is largely occupied, resulting in a diminished capacity of the adsorbent to capture additional adsorbate.The comparison of theadsorption isotherms of raw and milled PACs showed that, due to the grinding of PAC particles,additional inner pores structures became available for NOM adsorption. Results of this study pointout that structural properties of PAC dramatically influence the efficiency of combined PAC-UF,which needs to be considered during PAC selection and process design(Schulz et al., 2017).

Therefore, Figure 2 illustrates that increasing the mass of adsorbent in the adsorption process leads to greater reductions in pollutant concentrations. In particular, using 25 kg of adsorbent in the continuous flow system achieves removal efficiencies of 94.0% for TDS, 95.83% for total hardness, 96.36% for iron, 93.33% for manganese, and 86.67% for turbidity.Other studies have shown the adsorption capacity of activated carbon in continuous processes, either partially or in combination with other processes.The processes of activated carbon/ultrafiltration or microfiltration could be interesting alternatives for processing waters containing small amounts of pharmaceuticals(Abdi Bogoreh&Charcosset, 2017).The adsorption capacity of activated carbon in this process is strongly affected by several factors, including the concentration of adsorbate in the feed, the particle size of the adsorbent, the feed flow rate, and the properties of the adsorbent itself (Hoffman, 2008; Maimoun et al., 2020; Rashid et al., 2021). Therefore, further studies are required to determine the optimal performance of activated carbon in continuous adsorption systems, particularly for treating WWTP effluent from the seaweed processing industry into reusable process water.

**The effect of the size of the microfiltration membrane:-**

**Experiments using an adsorbent weight of 25 kg and microfiltration membrane sizes of 5 and 10 µm resulted the following experimental data as figure 3:-**

The figure 2 indicates that filtration size plays a crucial role in reducing pollutant concentrations.Overall, all curves display a downward trend, showing that pollutant levels decrease as filtration time or processing continues. However, the effectiveness of this reduction varies depending on the filtration size used.Finer filtration sizes generally result in greater pollutant removal. This is because they offer a larger surface area for contact and are more capable of trapping smaller particles, allowing more contaminants to be retained or adsorbed. In contrast, coarser filtration sizes exhibit slower reductions, as smaller pollutant particles are still able to pass through the media.




(a) (b)

**Figure 3. Performance of microfiltration membranes at different membrane sizes**

**a. Size of membrane 5 µm b. Size of membrane 10 µm**

In addition, a sharp decline in concentration is observed during the initial stage for all filtration sizes. This suggests that the filtration or adsorption process is most effective at the beginning, when the available active surface area is still abundant. As time progresses, the rate of reduction slows down and approaches a steady state, indicating that the filtration media is becoming saturated.In summary, the graph demonstrates that smaller filtration sizes lead to higher efficiency in pollutant removal, particularly in the early stages of the process before saturation occurs.In the AMFIL process, the use of 5 µm microfiltration membranes is more effective in reducing the concentration of pollutants (iron, manganese, dissolved solids, and total hardness), while turbidity remains relatively stagnant. This occurs because smaller membrane sizes are more efficient at filtering particles or pollutants under 1–5 µm, which are categorized as dissolved particles. In contrast, particles larger than 1–5 µm, known as suspended particles (turbidity), are filtered less effectively. Figure 3(a) illustrates that within 60 minutes of operation, the adsorption process is capable of removing pollutants of94.0% for TDS, 95.83% for total hardness, 96.36% for iron, 93.33% for manganese, and 86.67% for turbidity. Several earlier studies have demonstrated that factors such as membrane size, membrane type, feed flow rate, and pressure differential influence the effectiveness of filtration.

The removalefficiencies of organic matter by MF membrane filtration decreased with the increase oforganic matter removal rate by PAC adsorption(Song et al., 2015). Fixed bed adsorption of GAC was efficient during the first 108 h of FBAC - GAC operation, withremovals close to 90% for carbaryl, methomyl and carbofuran. However, before saturation (96 h), thefilling of the GAC fixed bed occurred, and the backwash procedure was essential for the continuedoperation of the adsorption column as well as the identification of the saturation of the carbamates in108 h (methomyl), 120 h (carbofuran) and 144 h (carbaryl)(Alves et al., 2020b).The color rejectionof nanofiltration was almost complete and permeate color was always lower than 10 Pt–Co. Similarly, quite high rejections were observed forCOD (80–100%). Permeate conductivity was between 1.98 and 2.67 mS/cm (65% conductivity rejection).Wastewaterfluxes were between 31 and37 L/m2/h at 5.07 bars corresponding to around 45% flux declines compared to clean water fluxes(Sahinkaya et al., 2008).

In addition, experimental results of the AMFIL process using adsorbent weights of 20 kg and 25 kg and microfiltration membrane sizes of 5 µm and 10 µm show that pollutant removal efficiency varies across different process conditions, as illustrated in Figure 4. The highest pollutants removal efficiency was achieved during adsorption with an adsorbent weight of 26 kg and a membrane size of 5 µm, whereas the lowest efficiency occurred with an adsorbent weight of 20 kg and a membrane size of 10 µm.

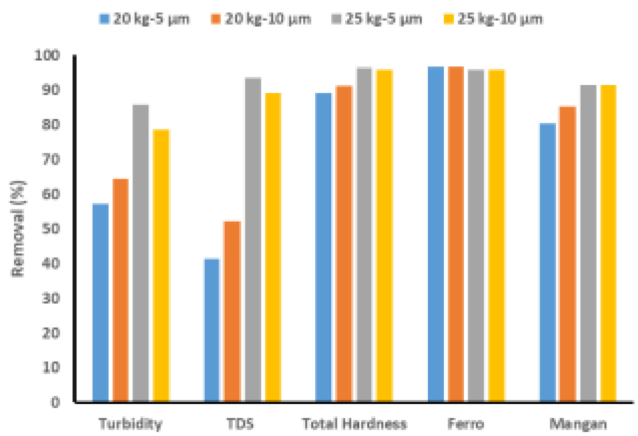



**Figure 4. The performance of the AMFIL process**

The removal efficiency also increased with the increase ofmixing intensity and mixing time. The permeate flux slightly improved when a membrane of pore size 0.22 µmwas used with shorter hydraulic residence time and lower PAC concentration(Ngo et al., 2000; Xing et al., 2008).The adsorption capacity represents the amount of adsorbate removal per unit mass of adsorbent. In Figure 5, the adsorption capacity in the AMFIL process increases over time. This indicates that the longer the adsorption duration, the greater the amount of adsorbate captured by the adsorbent.

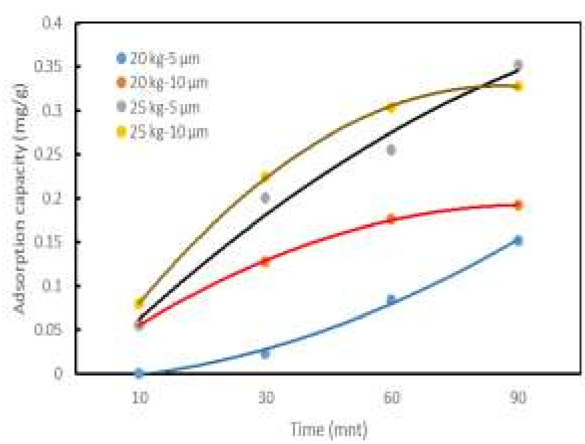



**Figure 5.The adsorption capacity in continuous flow AMFIL process**

After 60 minutes, most of the adsorbent surface reaches saturation. However, in the AMFIL process using 25 kg of adsorbent and a microfiltration membrane with a pore size of 5 µm, the maximum adsorption capacity achieved is 0.352 mg·g$^{-1}$.Conversely, the lowest adsorption capacity was observed under operating conditions with an adsorbent weight of 20 kg and a membrane size of 5 µm, reaching 0.15 mg·g$^{-1}$.

## Conclusion:-

The AMFIL process with an adsorption capacity of 0.352 mg.g$^{-1}$ and the size of a microfiltration membrane is 5µm are capable of processing effluent from WWTP into process water according to standard quality with the total removal pollutants such as turbidity, TDS, Fe, Mn, and pH are 86.67, 94.0, 95.83, 96.36, 93.33%, and (6.5 – 7.5), respectively.

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