

Abstract





Use of Activated Sludge Process in Molasses Industry Wastewater Treatment

Pekmez, Endüstrisi Atık Sularının Arıtılmasında Aktif Camur Prosesinin

Kullanımı

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Özet

Water is production of molasses and it takes an kaynaktır ve üretim esnasında gerçekleştirilen important role in cleaning, cooling and heating temizlik, soğutma ve ısıtma gibi çok çeşitli process. Therefore, the process of molasess islemlerde önemli rol oynar. Bu nedenle, leads to release large amount of wastewater pekmez üretim aşamasında çok fazla su containing organic components. Due to the kullanır ve bunun sonucu olarak, yüksek fact that this wastewater mainly consists of miktarda organik madde içeren atık su açığa organic components, the wastewater needs cikar. Pekmez üretim atık sularının kimyasal high level of biological oxygen (BOD) and iceriği chemical oxygen (COD). Thus, wastewater from the molasses process can ve kimyasal oksijen (KOİ) ihtiyacına sahiptir. causes of environmentally serious problems. Bu nedenle, bu atik su ciddi cevresel sorunlara To prevent these environmental problems, neden olmaktadır. Pekmez endüstrisi atık biological treatment systems are currently sularında yüksek organik madde içeriği used in the molasses industry. Active sludge nedeniyle biyolojik arıtma yöntemiyle etkili process is one of the most common processes aritma sağlanabilmektedir. used in the industry. Carbon, phosphate and prosesi biyolojik arıtma yöntemleri içerisinde nitrogen can be removed from the wastewater en fazla kullanılan yöntemdir. Aktif çamur by active sludge treatment process. Also, prosesi ile atık suda bulunan karbon, azot ve active sludge treatment systems are very fosfat biyolojik olarak uzaklaştırılmaktadır. effective, cheaper, and eco-friendly. This Arıtma sürecinde farklı görevleri olan study aims to show real-time analytical data mikroorganizma topluluğu olarak bilinen aktif from the wastewater from molasses industry camur prosesi cevre dostu, ekonomik ve etkili by using active sludge treatment system bir arıtma yöntemidir. Bu çalışmada pekmez (According to the governmental regulations on endüstrisi atık sularının aktif çamur prosesi ile wastewater treatment). In the molasses (atiksu aritma industry, COD was 2500-5500 mg/L, BOD aritilmasi sağlanmıştır.

an important component in Su, pekmez üretim prosesi için önemli bir genellikle organik maddelerden the oluştuğundan yüksek biyolojik oksijen (BOİ) Aktif camur tebliğine uygun olarak) Pekmez üretim was 800-1800 mg/L, TSS was 1.10-1.40 g/L, endüstrisi atık suyunun giriş değerleri KOİ conductivity was 1800-2600 µS/cm and pH 2500-5500 mg/L, BOİ 800-1800 mg/L, AKM was 3.5-5.5. After the active sludge treatment 1.10-1.40 g/L, iletkenlik 1800-2600 µS/cm ve process, COD and BOD were 30-70 mg/L and pH 3.5-5.5 arasında değişmektedir. Aktif 8-24 mg/L, respectively. Conductivity was çamur prosesi sonrası KOİ ve BOİ değerleri 1100-1200 µS/cm. Also, total nitrogen, nitrite, sırasıyla 30-70 mg/L ve 8-24 mg/L arasına, pH nitrate and total phosphate were analysed to 7.6-8.0 ve iletkenlik ise 1100-1200 µS/cm observe the low level of them. After the getirilerek teblige uygun halde getirilmiştir. treatment process, environmental pollution Ayrıca arıtılmış suyun toplam azot, nitrit, was reduced to minimum levels.

nitrat, toplam fosfat gibi değerleri de analiz edilerek uygunluğu kontrol edilmiştir. Bu sayede yüksek kirliliğe sahip olan atık su arıtılarak çevre kirliliği önlenmiştir.

Keywords: Molasses, Active sludge,	Anahtar kelimeler: Pekmez, Aktif çamur,
Wastewater	Atık su

Abbreviations: COD, chemical oxygen demand; BOD, biological oxygen demand; TSS, total suspended solids; TDS, dissolved solids; MBR, Membrane bioreactor

1. INTRODUCTION

1.1. The Importance of Water

Although more than %70 of the earth is covered by water, only % 0.5 of all water can be used as drinkable water. Because of this limited water supply and increasing human population, water became more important issue all over the world (Aladag, 2011; Sarkar et al., 2006). Unfortunately, water scarcity and drought are now a very important problem not only in dry zone, also in wetland areas (Al-Isawi et al., 2016). Within last 50 years, the amount of water used in industry increased by 3 times, and we consume more water than the amount which is essential for sustainable environment (Katip, 2018).

Accelerating industrial development and urbanisation has negative effect on the seas and freshwater bodies. These industrial activities cause of serious environmental problems. Over time, more industrial activities, and cities force the environment negatively. Lakes, rivers, and coasts are exposed to pollutants due to bigger cities and industry. Sometimes, these pollutants can cause of destroying of the water supplies irreversibly (Chan et al., 2009; NG Wun, 2006).

1.2. Wastewater

Until 19th century, the relationship between water pollution and diseases could not be determined. In 1854, Dr. John Snow could detect that epidemic cholera was happened because well water which was contaminated with sewage was used as drinking water (Aladag, 2011). After this determination, treatment of water became more important issue.

Last century, demand to drinking and utility water increased by ten times. Correspondingly, pollution level of water increased. Therefore, the pollution criteria (BOD, TSS, oil, nitrogen etc.) of domestic and industrial wastewater should be investigated (CIrIk & Eskikaya, 2018).

Industrial wastewaters can be classified based on the industry field and raw materials that were used in the process. Some industrial wastewaters can contain too-much organic components, high amount of inorganic or toxic components, or easily decompose to biological matters. Which means that suspended solid (TSS), biological oxygen (BOD) and chemical oxygen (COD) can be in a wide range from 1 mg/L to 1000 mg/L (NG Wun, 2006).

1.3. Content of Molasses

Molasses is a food containing high amount of carbohydrate, mineral and organic acids. It is made of fresh or dried fruits such as grape, mulberry, fig, locust bean, apple, watermelon, sugar beet etc. each kind of molasses was called as the fruit that was used in the process. Fresh or dried grape is treated with calcium carbonate and then some enzyme. The final product is highly viscose product (Çümen, 2021). Also, molasses contains large amount of carbohydrade like glucose and fructose.

1.4. Active Sludge Process

Physical, chemical and bacteriological features of water can be regained partly or completely with water treatment systems. Water treatment systems involve all processes of physical, chemical, and biological operations to cleanse wastewater before wastewater releases to receiving environment (Chan et al., 2009; Stainer, 1976). Water treatment stations are generally succession of activities involving tanks and pools where wastewater is purified (Anlı & Şanlı, 2019).

Activated sludge process is the most used method among biological treatment methods (Martins et al., 2004). Activated sludge process, which was developed in England as a result of the studies carried out by Arden and Lockett in 1913-1914, is a turning point in biological wastewater treatment (Metcalf & Eddy, 1991). Microorganisms in wastewater benefit from

organic materials in wastewater that have biological value for structure change and energy gain. Thus, the organic matter taken into the microorganism is removed from the environment as activated sludge. This removed sludge is considered as activated sludge since it shows biological activity (Akdemir, 2011). Activated sludge method, which aims to transform wastewater into activated biological flocs, is a process equivalent to naturally self-treatment of polluted water under aerobic conditions (Berkun, 2006).

In activated sludge systems, organic matter resides in the pool where the aerobic bacterial culture is suspended. In the pool, the stoichiometric transformation of the bacterial culture takes place as in reactions (1) and (2) below. In case of oxidation and synthesis;

COHNS +
$$O_2$$
 + Nutrient \longrightarrow CO₂ + NH₃ + C₅H₇NO₂ + other products (1)

In the reaction equation, COHNS represents the organic matter in the wastewater. When the amount of substrate is insufficient, some of the cell's storage materials are oxidized by its own mass. This internal respiration is expressed by equation (2).

$$C_5H_7NO_2 + 5O_2 \xrightarrow{\text{Bacterium}} 5CO_2 + 2H_2O + NH_3 + Energy$$
 (2)

Equations (1) and (2) describe the complete oxidation of organic matter (Akdemir, 2011; Metcalf & Eddy, 1991).

In wastewater treatment with activated sludge process; decomposition, degradation or oxidation of carbonaceous wastes, degradation or oxidation of nitrogenous wastes, removal of fine solids and removal of heavy metals take place and these processes are given in Table 1 (Gerardi, 2003).

Table 1. Processes in the activated sludge system

Aim	Oxidation and removal equation		
Biochemical Oxygen Demand (cBOD) oxidation involving carbon	$cBOD_{(protein)}+O_2 \longrightarrow C_5H_7O_2N+CO_2+H_2O+NH_4^++SO_4^{2-}+HPO_4^{2-}$		
Nitrogen-containing Biochemical Oxygen Demand (cBOD) oxidation	$nBOD_{(ammonium ions)} + O_2 \longrightarrow C_5H_7O_2N + NO_3^- + H_2O$		
Fine solids removal	Colloids, split growth, insoluble substances		
Removal of heavy metals	Al, Cr, Cu, Cd, Fe, Pb, Hg, Ni, Zn		

Basic components of an activated sludge system; aeration tank and settling basin or clarifier (Figure 1). Activated sludge; mixed liquid containing the source of microorganisms such as bacteria, fungi, protozoa and high-form organisms includes biological flocs containing

biodegradable and non-degradable suspended, colloidal and dissolved organic and inorganic substances (Akdemir, 2011; Spellman, 2000; Wang et al., 2009).



Figure 1. Semas Food basic activated sludge process diagram

In this study, it was aimed that the wastewater profile of Semas Gida, which produces molasses, was formed and the wastewater of the molasses industry was treated in accordance with the activated sludge process and the wastewater treatment communiqué. For this purpose, 30 days pH, conductivity, dissolved oxygen, total solids (TS), suspended solids (TSS), dissolved solids (TDS), chemical oxygen demand (COD) of molasses production wastewater, activated sludge pool and MBR filter outlet. , biological oxygen demand (BOD), total kjendahl nitrogen, nitrate, nitrite and total phosphate values were tested for compliance with the communiqué. The effluent water features that should be in the wastewater treatment pool are as in Table 2.

Analysis	Results
рН	6-9
SS (mg/L)	<70
COD mg/L	<150
BODmg/L	<50

2. MATERIALS and METHODS

2.1. Sampling

In this study, samples were taken from the points indicated in Figure 1 in the wastewater facility, at intervals of 4 hours, for 30 days, 5000 mL per day from each sample. Then, these samples

were mixed and homogenized and the following tests were carried out. Table 3 shows the daily sampling time and amount. Samples were taken according to Table 3 for 30 days.

	Table 3.	The	times	of t	aking	daily	water	sampl	es
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	1 ^{stv} sample	2 nd sample	3 rd sample	4 th sample	5 th sample
Time	08:00	12:00	16:00	20:00	24:00
Quantity	1000 mL	1000 mL	1000 mL	1000 mL	1000 mL

2.2. Microscope Analysis

Samples taken from the wastewater facility were examined daily with A. Krüss Optronic brand microscope with 10X, 40X and 100X magnifications. In microscope analysis; vitality in wastewater and MBR outlet, vitality in activated sludge and morphological structure analysis were performed.

2.3. pH Determination

The pH analyzes of the samples taken from the facility were made with Ohaus Starter2100 brand pH meter and Ohaus ST310 brand probe.

2.4. Conductivity Determination

Conductivity analyzes of the samples taken from the facility were made with Emaf EM77G brand conductivity meter and Mettler toledo 950-K19/120 brand probe.

2.5. Dissolved Oxygen Determination

Dissolved oxygen of the samples taken from the facility was made with Hanna HI2004 brand oxygen meter.

2.6. Determination of Total Solids (TS), Suspended Solids (TSS) and Dissolved Solids (TDS)

A well-mixed 250 mL sample is evaporated in a constant weight capsule and dried in an Ankyralab brand oven until a constant weight is obtained at 103-105°C, then cooled in a desiccator and weighed. The weight gain in the empty capsule represents the total solids content.

After keeping it in an Ankyralab oven at 105 °C for 1 hour and cooling it in a desiccator, the vacuum filtered sample from filter paper (0.45 µm membrane for clean water, glass fiber for

wastewater) is carefully taken on an aluminum or stainless steel tray by using forceps. It is dried in an oven at -105°C for 1 hour. It is cooled in a desiccator and weighed. The weight of the filtrate residue refers to the suspended solids. The filter residue obtained for the suspended solids (105°C) is in a capsule brought to a constant weight at 550±50°C in an Elimko 70 brand muffle furnace at 550±50°C and in a muffle furnace at 550±50°C. It is burned for 20 minutes. It is cooled in a desiccator and weighed. The solid remaining after the weight loss caused by the combustion of volatile solids; is a volatile suspended solid (Baird, 2017).

2.7. Chemical Oxygen Demand Determination (COD)

Chemical oxygen demand (COD) is one of the important parameters used to determine the organic pollution level of domestic and industrial wastewater. Chemical oxygen demand (COD) is the amount of oxygen required for the chemical oxidation of oxidizable substances in water (Dedkov et al., 2000).

Organic compounds

$$C_aH_bO_c$$
 + $Cr_2O_7^{2-}$ + H⁺ Catalyst
 $C_aH_bO_c$ Cr³⁺ + CO₂ + H₂O

5 mL of the sample is taken into the reaction flask and 2.5 mL of potassium dichromate solution (80 g of mercury sulfate is dissolved in 800 mL of water. 100 mL of 1.84 g/mL sulfuric acid is added and 11,77 gr K₂Cr₂O₇, dried at 105 °C, is added and cooled and made up to 1000 mL) is added and mixed. 7.5 mL of silver sulfate/sulfuric acid solution (10 g silver sulfate is dissolved in 35 mL of water, and 965 mL of 1.84 g/mL sulfuric acid is added) is added to the refrigeration system. The reaction is continued for 2 hours at 150 °C. The cooled samples are tested with a Shimadzu 1201V brand spectrophotometer at 600 nm. Potassium hydrogen phthalate was used as a standard (1000 mg/L COD sodium phthalate; 0.8 g sodium phthalate dissolved in 1000 mL water).

2.8. Determination of Biological Oxygen Demand (BOD)

The BOD is defined as that mass of oxygen that is consumed by chemicals in the course of n days to oxidatively degrade the organic substances present in 1 Lof water at 20 °C. The dissolved oxygen oxidizes manganese(II) to manganese(III). In acidic solution, this reacts with Titriplex® II to form a red complex that is determined photometrically (modified Winkler method). The BODn is calculated as the difference between the oxygen concentrations determined immediately after sampling and that after n days of incubation of a water sample to which allyl thiourea has been added to inhibit nitrification. The method is analogous to EPA 405.1 (BOD Cell Test 100687).

Check the pH of the sample, specified range: pH 6-8. If required, add dilute sodium hydroxide solution or sulfuric acid drop by drop to adjust the pH. Fill oxygen reaction boots each with pretreated sample and 2 glass bads to overflowing. Close bubble-free with the slanted ground-glass stoppers. Fill oxygen reaction boots each with inoculated nutrient-salt solution and 2 glass bads to overflowing. Close bubble-free with the slanted ground-glass stoppers. Use one bottle of pretreated sample on done of inoculated nutrient-salt solution fort the measurement of the initial oxygen concentration. Incubate one bottle of pretreated sample a done of inoculated nutrient-salt solution closed in a thermostatic incubation cabinet at 20°C for 5 days. After incubation, use one bottle of pretreated sample a done of inoculated nutrient-salt solution for the measurement of the final oxygen concentration. Add 5 drops of BOD-1K and then 10 drops of BOD-2K, close bubble-free, and mix for approx. Add in one minute 10 drops of BOD-3K, reclose and mix. Place the cell into the cell compartment. Align the mark on the cell with that on the spectrophotometer 500 nm.

Calculation:

BOD of measurement sample: Result 1-Result 2 (measurement sample) = A in mg/L

BOD of blank1 - Resut 2 (blank) = B in mg/L

BOD of original sample in $mg/L = (A-B)^*$ dilution factor

2.9. Determination of Total Kjeldahl Nitrogen

The 100 mL sample is taken into the Kjeldahl flask containing the boiling stone. 50 mL of digestion solution (67 g of K₂SO₄ is dissolved in 400 mL of distilled water and 67 mL of 1.84 g/mL H₂SO₄ is added to it and completed to 500 mL by adding 3.65 g of CuSO₄). It is boiled until 15-20 mL remains in the solution and cooled. 250 mL of distilled water is added. Distillation is carried out with 50 mL of neutralization solution (250 g NaOH and 12.5 g Na₂S₂O₃.5H₂O are taken, completed to 500 mL and dissolved in distilled water) (200-250°C). 50 mL of H₃BO₃ solution (10 g of H₃BO₃ is dissolved in ammonia-free freshly distilled water and made up to 500 mL. 5 mL of mixed indicator is added) into a 500 mL flask. When pH is 3.4-3.5 mixing stopped. The distillate is collected until a minimum of 200 mL has accumulated (green color). Titrate with 0.02N H₂SO₄ until the initial pH value is determined. Glutamic acid, 1 g/L equivalent to 100 mg N, was used as a standard. (Mixed indicator; 1 g Methylene blue + 1 g Methylene Red + 100 mL ethyl alcohol)

Calculation:

$$TKN = \frac{(A-B) \times N \times 14000}{V} = \frac{(A-B) \times 280}{V}$$

N=0.02N

V= sample volume A=Titrant amount B= Witness titrant

2.10. Nitrate Determination (with 2,6 – dimethylphenol spectrophotometric method)

In the presence of sulfuric acid and phosphoric acid, nitrate reacts with 2,6-dimethylphenol to form 4-nitro-2,6-dimethylphenol. The reaction time is about 5 minutes. The absorbance of the reaction product is measured spectrometrically at a wavelength of 324 nm and the nitrate concentration in the analysis sample is read from the calibration curve. 35 mL of sulfuric acid-phosphoric acid mixture (250 mL of sulfuric acid + 500 mL of ortho-phosphoric acid is carefully mixed and 0.020 g of amidosulfonic acid is added to a 100 mL flask). 5 mL of wastewater sample and 5 mL of 2,6-dimethylphenol solution (0.6 g of 2,6-dimethylphenol is dissolved in 500 mL of glacial acetic acid) are added and left for 30 minutes. Samples are tested with a Shimadzu 1201V brand spectrophotometer at a wavelength of 324 nm. In this method, potassium nitrate solution in the range of 1-50 mg/L was used as a standard (Huifa et al., 2003).

2.11. Nitrite Determination

Nitrite (NO₂⁻) is determined by the principle of reacting with sulfanilamide and N-(1 naphthyl)ethylenediamine dihydrochloride under acidic conditions to form a reddish-purple azo dye. The resulting product is measured spectrophotometrically at a wavelength of 543 nm and the concentration of nitrite in the sample is determined. 50 mL of sample, passed through a 0.45 μ m filter, is taken into a 100 mL flask and 2 mL of color developing reagent is added and mixed. After waiting for 20 minutes, the samples are tested with a Shimadzu 1201V brand spectrophotometer at 543 nm wavelength. In this method, NaNO₂ solutions containing 0.05-0.5 μ g N were used as standard (Nagaraja et al., 2001).

2.12. Determination of Total Phosphorus with Stannous Chloride

One of the phosphorus determination methods is the colorimetric method in which Tin (II) Chloride is used as the reducing agent. In this method, phosphate ions form ammonium molybdate and ammonium phospho molybdate. The molybdenum blue complex is formed as a result of the reduction of this compound with stannous chloride. Since the color of this complex is proportional to the phosphate concentration, the orthophosphate concentration is determined colorimetrically by measuring the color intensity (Arnold et al., 1992).

4 mL Molybdate reagent (25 g (NH₄)₆Mo₇O₂₄·4H₂O is dissolved in 175 mL distilled water to 100 mL of wastewater or effluent sample, carefully added to 266 mL concentrated H₂SO₄ 400 mL distilled water in a separate container, cooled, molybdate solution is added. and diluted to 1 L) and 0.5 mL (10 drops) of stannous chloride reagent (2.5 g of fresh SnCl₂·2H₂O dissolved in 100 mL of glycerine. Heated in a water bath and dissolved by mixing with a glass baguette). The ammonium phosphomolybdate complex is reduced, revealing the molybdenum blue color depending on the phosphorus concentration. After the reagents are added, the following reaction takes place:

 $(NH_4)_3PO_4.12MoO_3 + Sn^{2+}$ molybdenum blue + Sn⁴⁺

After 10 minutes, the developed color is measured in the spectrophotometer at 690 nm and the result is compared with the calibration curve and the phosphorus content is found. As a standard, a solution of KH₂PO₄ in the range of 0.05-1.2 mg/L is used (219.5 mg of anhydrous potassium dihydrogen phosphate is dissolved in pure water and made up to 1 L. 1 mL of this solution is $50 \ \mu g \ PO_4$ -P).

Calculation:

$$P = \frac{mg \ P \ \times \ 1000}{mL \ sample}$$

3. RESULTS and DISCUSSION

As a result of the tests, the average of the 30-day chemical and biological results of the molasses industry wastewater and the average of the results of the MBR effluent after the activated sludge process are given in Table 4.

Analysis	Unit	Wastewater	Sludge Pool	MBR output
pH	-	4.54±0.63	7.28±0,09	7.71±0,13
Conductivity	μS/cm	2263.01±221.54	1535.10±40.02	1213.03±21.71
Dissolved oxygen	mg/L O ₂	0.4±0.27	5.23±0.21	5.88±0.10
TSS (Total suspended solids)	g/L	3.68±0.35	6.14±0.48	n.d.
SS (Suspended solids)	g/L	1.21±0.09	5.92±0.48	n.d.
TDS (Total dissolved solids)	g/L	2.44±0.34	0.21±005	n.d.
COD (Chemical oxygen demand)	mg/L O ₂	3889.03±944.80	121.03±26.56	52.53±10.27
BOD (Biological oxygen demand)	mg/L O ₂	1297.22±324.36	41.74±8.87	17.75±3.68
Total Kjeldahl Nitrogen	mg/L N	119.85±46.17	not tested	1.01±0.21
Nitrate	mg/L NO ₃ -	82.66±27.21	not tested	0.33±0.07
Nitrite	mg/L NO ₂ -	5.84±1.39	not tested	n.d.
Total Phosphate	mg/L PO ₄ -P	55.34±8.42	not tested	1.01±0.25

Table 4. Average results of all analysis over 30 days

3.1. Microscope Analysis

Samples taken from the wastewater facility were examined daily with A.Krüss Optronic brand microscope at 10X, 40X, 100X and 200X magnifications. In microscope analysis; a high rate of bacterial and yeast viability was observed in the wastewater, but the species were not determined. No vitality was observed in the MBR output. In the activated sludge, the vitality and morphological structure analyzes were made daily and the species specified in Table 5 were determined.

Туре	Genus	Туре	Genus
	Pseudomonas spp.		Ciliates
Bacterium	Arthobacter spp.	- Drotozoa	Flagellates
	Bacillus spp.		Rhizopoda (Amoeba)
	Cytophaga spp.	_	Rotifers
	Zooglea spp.		Geotrichum
	Acinetobacter spp.	_	Penicillium
	Nitrosomonas spp.	 Fungi	Cephalosporium
	Nitrobacter spp.	_	Cladosporium
	Sphaerotilus spp.	_	Alternaria

Table 5. Types of microorganisms determined in activated sludge

3.2. pH, Conductivity and Dissolved Oxygen Analysis

The results of the inlet, activated sludge pool and effluent samples taken from the wastewater pool, showing the 30-day graphs of pH, conductivity and dissolved oxygen, are given in Figure 2-4.



Figure 2. 30-day pH graph of wastewater, activated sludge pool and MBR effluent

The pH of the wastewater inlet for 30 days was 4.54 ± 0.63 , while the pH of the effluent was 7.71 ± 0.13 .



Figure 3. 30-day conductivity graph of wastewater, activated sludge pool and MBR effluent

In addition, the conductivity of the wastewater inlet was 2263 ± 221.54 µS/cm, while the conductivity of the effluent was 1213 ± 21.71 µS/cm.



Figure 4. 30-day DO graph of wastewater, activated sludge pool and MBR effluent

Finally, the dissolved oxygen of the wastewater inlet was 0.4 ± 0.27 mg/L O₂, while the dissolved oxygen of the effluent was 5.88 ± 0.10 mg/L O₂.

3.3. Total solids (TS), Suspended Solids (TSS) and Dissolved Solids (TDS) Analysis

The results showing the 30-day graphs of the total solid, suspended solids and dissolved solids amounts of the inlet, activated sludge pool and effluent samples taken from the wastewater pool

are given in Figure 5. As a result of the analyzes, the TSS of the wastewater pool inlet samples was 1.21 ± 0.09 , while no solid matter was detected in the effluent.



Figure 5. 30-day solids graph of wastewater and activated sludge pool

3.4. Chemical Oxygen Demand Determination (COD)

The results showing the 30-day graphs of the chemical oxygen demand analyzes of the inlet, activated sludge pool and effluent samples taken from the wastewater pool are given in figures 7 and 8. Results were calculated against the potassium hydrogen phthalate standard (Standard graph figure 6). The standard graphic equation gives the equivalent COD value of potassium hydrogen phthalate at different concentrations. It is seen that there is a linear change between these two variables. As a result of the analyzes made, the mean COD value of the wastewater inlet was 3889.03 ± 944.80 mg/L O₂, while the MBR effluent was 52.53 ± 10.27 mg/L O₂.



Figure 6. Potassium hydrogen phthalate standard graph



Figure 7. 30-day COD graph of wastewater



Figure 8. 30-day COD graph of activated sludge and MBR effluent

3.5. Determination of Biological Oxygen Demand (BOD)

The results showing the 30-day graphs of the biological oxygen demand analyzes of the inlet, activated sludge pool and effluent samples taken from the wastewater pool are given in figures 9 and 10. As a result of the analyzes, it was seen that the average BOD value of the wastewater inlet was 1297.22 ± 324.36 mg/L O₂, while the MBR effluent was 17.75 ± 3.68 mg/L O₂.



Figure 9. 30-day BOD graph of wastewater



Figure 10. 30-day BOD graph of activated sludge and MBR effluent

3.6. Determination of Total Kjeldahl Nitrogen

The results showing the 30-day graphs of the total nitrogen analyzes of the inlet and outlet water samples taken from the wastewater pool are given in Figures 11 and 12. As a result of the analyzes, it was seen that the total nitrogen of the wastewater inlet was 119.85 ± 46.17 mg/L N on average, while the MBR outlet water was 1.01 ± 0.21 mg/L N.



Figure 11. Graph of 30-day total Kjeldahl Nitrogen of wastewater



Figure 12. 30-day total Kjeldahl Nitrogen graph of MBR effluent

3.7. Determination of Nitrate

The results showing the 30-day graphs of nitrate analyzes of the inlet and outlet water samples taken from the wastewater pool are given in figures 14 and 15. In addition, the potassium nitrate standard graph used to calculate the results is given in Figure 13. The standard graphic equation gives the equivalent nitrate value of potassium nitrate at different concentrations. It is seen that there is a linear change between these two variables. As a result of the analysis, it was seen that the nitrate of the wastewater inlet was $82.66\pm27.21 \text{ mg/L NO}_3^-$ on average, while the MBR outlet water was $0.33\pm0.07 \text{ mg/L NO}_3^-$.



Figure 13. Potassium nitrate standard graph



Figure 14. 30-day nitrate graph of wastewater



Figure 15. 30-day nitrate graph of MBR effluent

3.8. Nitrite Determination

The results showing the 30-day graphs of the nitrate analyzes of the inlet and outlet water samples taken from the wastewater pool are given in Figure 17. In addition, the sodium nitrite standard graph used to calculate the results is given in Figure 16. The standard graphic equation gives the equivalent nitrite value of sodium nitrite at different concentrations. It is seen that there is a linear change between these two variables. As a result of the analysis, it was observed that the nitrate of the wastewater inlet was 5.84 ± 1.39 mg/L NO₂⁻ on average, while nitrite could not be detected in the MBR outlet water.



Figure 16. Sodium nitrite standard graph



Figure 17. Wastewater inlet 30-day nitrite graph

3.9. Total Phosphate Determination

The results showing the 30-day graphs of the total phosphate analyzes of the inlet and outlet water samples taken from the wastewater pool are given in Figures 19 and 20. In addition, the potassium dihydrogen phosphate standard graph used to calculate the results is given in Figure 18. The standard graphic equation gives the equivalent total phosphate value of potassium dihydrogen phosphate at different concentrations. It is seen that there is a linear change between these two variables. As a result of the analysis, it was seen that the nitrate of the wastewater inlet was 55.34 ± 8.42 mg/L PO₄-P on average, while the MBR outlet water was 1.01 ± 0.25 mg/L PO₄-P.



Figure 18. Potassium dihydrogen phosphate standard plot



Figure 19. 30-day total phosphate graph of wastewater



Figure 20. 30-day total phosphate graph of MBR output

4. CONCLUSION

In this study, the wastewater of the molasses industry was characterized and treated with activated sludge process. Firstly, wastewater properties were determined by taking wastewater samples from the inlet water for 30 days. Characteristics of wastewater vary between COD 2500-5500 mg/L, BOD 800-1800 mg/L, TSS 1.10-1.40 g/L, conductivity 1800-2600 μ S/cm and pH 3.5-5.5. Then, the characteristics of the effluent after the activated sludge process were determined. After the activated sludge process, the COD and BOD values were determined

between 30-70 mg/L and 8-24 mg/L, pH 7.6-8.0 and conductivity 1100-1200 μ S/cm, respectively. In addition, the properties and TSS of the activated sludge were measured for 30 days. In this way, the amount of suspended solids (TSS) in activated sludge pools was kept in the range of 4-9 g/L. In cases where the density of activated sludge is over 9 g/L, sludge swelling was observed in the pools and it was determined that the microorganisms in the sludge died. It was observed that adequate treatment could not be performed in pools with 4 g/L activated sludge density. In addition, values such as total nitrogen, nitrite, nitrate, and total phosphate of wastewater and treated water were determined and it was determined that they were in compliance with the communiqué. As a result, the discharge of highly polluted wastewater has become possible by using this activated sludge process.

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