ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

# INVESTIGATION OF TREATMENT PERFORMANCES AND ENERGY RECOVERIES FROM A REAL TEXTILE WASTEWATER IN CONVENTIONAL AND HIGH-RATE MBR PROCESSES

**Ph.D. THESIS**

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**JANUARY 2024**

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**ISTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ**

# KONVANSİYONEL VE YÜKSEK YÜKLÜ MBR PROSESLERİNDE GERÇEK BİR TEKSTİL ATIKSUYU ARITIM PERFORMANSININ VE ENERJİ GERİ KAZANIMININ ARAŞTIRILMASI

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**OCAK 2024**

Tülay YILMAZ, a Ph.D. student of İTU Graduate School student ID 501192805 successfully defended the thesis/dissertation entitled "INVESTIGATION OF TREATMENT PERFORMANCES AND ENERGY RECOVERIES FROM A REAL TEXTILE WASTEWATER IN CONVENTIONAL AND HIGH-RATE MBR PROCESSES", which she prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

| | | |
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***To my family,***

## FOREWORD

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January 2024

Tülay YILMAZ
(Environmental Engineer)

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## ABBREVIATIONS

| | |
|---|---|
| **ADMI** | : American Dye Manufacturers Institute |
| **ANOVA** | : One-way analysis of variance |
| **AOB** | : Ammonium oxidizing bacteria |
| **AOTR** | : Actual oxygen transfer rate |
| **CAS** | : Conventional activated sludge |
| **CO** | : Consumed oxygen concentration |
| **COD** | : Chemical oxygen demand |
| **C/N** | : Carbon to nitrogen ratio |
| **CST** | : Capillary suction time |
| **CSTR** | : Continuously stirred tank reactor |
| **DO** | : Dissolved oxygen |
| **EPS** | : Extracellular polymeric substances |
| **FA** | : Free ammonia |
| **FBR** | : Fluidized bed reactor |
| **FNA** | : Free nitrous acid |
| **F/M** | : Food to microorganism |
| **FTIR** | : Fourier-transform infrared spectroscopy |
| **FS** | : Flat sheet |
| **GPC** | : Gel Permeation Chromatography |
| **HF** | : Hallow fiber or fibre |
| **HRT** | : Hydraulic retention time |
| **HRAS** | : High-rate activated sludge |
| **HR-MBR** | : High-rate membrane bioreactor |
| **LB-EPS** | : Loosely bound extracellular polymeric substances |
| **LMH** | : Litres per square metre hour |
| **MBR** | : Membrane biorector |
| **MF** | : Microfiltration |
| **MLSS** | : Mixed liquor suspended solids |
| **MLVSS** | : Mixed liquor volatile suspended solids |
| **MMBR** | : Moving bed biofilm reactor |

**MT** : Multi-tube
**NF** : Nanofiltration
**NOB** : Nitrite oxidizing bacteria
**$N_{ox}$** : Oxidizable ammonia concentration
**$NO_{3,e}$** : Nitrate nitrogen concentration
**$OD_{theory}$** : Theoretical oxygen requirement
**ODfield** : Field oxygen requirement
**ORP** : Oxidation-reduction potential
**PAC** : Powdered activated carbon
**PE** : Polyethylene
**pH** : Potential of hydrogen
**PP** : Polypropylene
**PVDF** : Polyvinylidene fluoride
**PSD** : Particle size distribution
**$P_{x,bio}$** : Biological sludge wasted daily
**RO** : Reverse osmosis
**$R_S$** : Dynamic oxygen uptake rate
**$R_{Sp}$** : Specific dynamic oxygen uptake rate
**SBR** : Sequencing batch reactor
**$S_E$** : Effluent COD concentration
**SEM** : Scanning electron microscopy
**SEM-EDS** : SEM coupled with energy dispersive spectroscopy
**SF** : Supernatant filterability
**SMP** : Soluble microbial products
**$S_o$** : Influent COD concentration
**$S_{RB}$** : Residual soluble bulk COD
**SRT** : Sludge retention time
**SS** : Suspended solid
**SRF** : Specific resistance to filtration
**STR** : Stirred tank bioreactor
**SOTR** : Standard oxygen transfer rate
**TB-EPS** : Tightly-bound extracellular polymeric substances
**TIN** : Total inorganic nitrogen
**TKN** : Total Kjeldahl nitrogen
**TMP** : Transmembrane pressure

| | |
|---|---|
| **TN** | **:** Total Nitrogen |
| **TP** | **:** Total Phosphorus |
| **TS** | **:** Total solid |
| **TSS** | **:** Total suspended solid |
| **UF** | **:** Ultrafiltration membrane |
| **VSS** | **:** Volatile suspended solid |
| **UASB** | **:** Upflow anaerobic fixed bed |
| $\mathbf{Y_{obs}}$ | **:** Observed sludge yield |

## SYMBOLS

| | |
|---|---|
| **$^{o}C$** | : Celsius |
| **cm** | : Centimeter |
| **d** | : Day |
| **$DO_{S,20}$** | : Oxygen saturation concentration |
| **E** | : Oxygen transfer efficiency of diffuser |
| **h** | : Hour |
| **g** | : Gram |
| **kg** | : Kilogram |
| **kWh** | : Kilowatt hours |
| **L** | : Liter |
| **mg** | : Miligrams |
| **min** | : Minute |
| **mL** | : Milliliter |
| **$m^2$** | : Square metre |
| **$m^3$** | : Cubic meter |
| **Pt-Co** | : Platinum-cobalt |
| **s** | : Second |
| **T** | : Temperature |
| **Q** | : Wastewater flow rate |
| **$\alpha$** | : Oxygen transfer correction factor |
| **$\beta$** | : Wastewater correction factor for oxygen solubility |
| **$\Theta$** | : Temperature correction factor |

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## INVESTIGATION OF TREATMENT PERFORMANCES AND ENERGY RECOVERIES FROM A REAL TEXTILE WASTEWATER IN CONVENTIONAL AND HIGH-RATE MBR PROCESSES

### SUMMARY

Textile industry wastewater, which is among the most water-consuming sectors worldwide, is extremely hazardous for receiving water bodies due to its toxic and complex structure. Many studies have been conducted involving physical, chemical, biological and combined processes for the textile wastewater treatment, but none of them alone is sufficient to meet discharge standards, and each process requires higher investment and operating costs. Additionally, energy-neutral plants should be expanded to ensure circular economy during the textile wastewater treatment, and the treated wastewater should be appropriate to be used for further reuse processes. Aerobic membrane bioreactors (MBRs) have been widely preferred in the textile wastewater treatment due to their ease of operation, lower volume requirement, and providing higher quality effluent compared to traditional biological methods. However, aerobic MBRs require higher energy input to both supply the oxygen requirement of microorganisms and minimize membrane fouling. In the treatment of textile industry wastewater, a single aerobic process is not sufficient to remove organic matters, azo and reactive dyes (anaerobic/aerobic combined system are needed) and nitrogen-based pollutants (nitrification and denitrification processes are needed). In addition, processes selected should be less-energy consuming due to high volume of textile industry wastewater. Considering that water resources are decreasing globally today, it is not enough to treat textile industry wastewater and their recycling is also extremely important. Therefore, sustainable and practical treatment strategies should be developed for textile industry.

This thesis aims to investigate the treatment potential of two different biological processes with different advantages for real textile wastewater. First of all, the treatment performance of the real textile wastewater and the recovery of organic matter that can be preferred as a raw material source for biomethane generation were investigated in an aerobic high-rate MBR including a hollow fiber (HF) ultrafiltration membrane (UF) with a pore size of 0.04 μm. In the high-rate MBR process, it was aimed to recover organic matters rather than oxidation at short (0.5-5 days) sludge retention times (SRTs) and hydraulic retention times (HRTs). Additionally, the effects of SRT/HRT ratios on membrane filtration performance, sludge characterization, sludge production, and the energy requirements for aeration were examined, and all parameters were compared with a conventional long SRT aerobic MBR system operated in parallel. In another MBR process equipped with an HF-UF membrane, the feasibility of intermittent aeration strategy for simultaneous nitrification and denitrification to remove nitrogen-based pollutants and for energy minimization were investigated. Additionally, the effects of different aeration patterns on membrane filtration performance, sludge characterization, sludge production and energy requirements for aeration, and specific removal rates were studied. Within the scope of this thesis, all studies were carried out in four stages.

In the first stage, textile wastewater treatment performances of conventional MBRs were investigated at SRTs of 30, 20 and 10 d. During this stage, two identical MBR systems were run, one at SRT for 30 days (MBR-1), and the other at SRT for 20 and 10 days (MBR-2), respectively. The average total chemical oxygen demand (COD), color and total dissolved nitrogen concentrations of textile wastewater used in the study averaged 927±277 mg/L, 910±287 Pt–Co and 39±10 mg/L, respectively. While COD removal performance was above 90% in all SRTs, color removal reached its lowest value at SRT of 10 d and did not show any correlation with SRT. In both MBRs, transmembrane pressure (TMP) was below 10 mbar throughout the study and no membrane fouling was observed. Supernatant filterability (SF) and specific filtration resistance (SRF) values increased at 10 d of SRT. A decrease in viscosity values was also observed due to the decrease in suspended solids (SS) concentration as SRT decreased. While SMP concentrations were similar at all tested SRT values, an increase in EPS concentration was observed at 10 d SRT. Additionally, reducing SRT resulted in an increase in waste sludge generation and observed biomass yield ($Y_{obs}$), and a decrease in the energy requirement for aeration. According to gel permeation chromatography (GPC) results, as SRT decreased, organic compounds with low molecular weight had higher signals.

In the second stage, aim was to investigate textile wastewater treatment performance and organic matter recovery efficiency at short SRTs, and a laboratory-scale high-rate aerobic MBR was run at SRTs of 0.5 – 5 d and HRTs of 1.2 – 24 h, corresponding to predetermined different SRT/HRT ratios of 5, 10 and 20. The average total COD, color, and soluble nitrogen concentrations in the wastewater were 834±143 mg/L and 1037±407 Pt-Co and 51±11 mg/L, respectively. While COD removal performances ranged between 86 and 92% at SRT of 5, 3, and 2 d (in all SRT/HRT ratios), it decreased to 82 and 77% at SRT 1 and 0.5 d (at SRT/HRT ratio of 10), respectively. There was no correlation between decolourization performance and SRT or HRT as it varied between 26% and 70%. The nitrification performance in the system stopped completely at SRTs ≤ 2 d. In particular, when SRT decreased from 5 days to 1 day, the amount of sludge produced and $Y_{obs}$ values increased. The SRT/HRT ratio played an important role in the energy requirement for aeration. In addition, reducing the SRT in the system resulted in higher SRF values, lower SF values, and rapid membrane fouling. SMP in the supernatant increased especially at SRTs ≤ 2 d. The total EPS concentration increased as SRT decreased, but the it decreased as the SRT/HRT ratio increased at each SRT value. No significant change occurred in the molecular weight distributions of the organic substances in the supernatant and filtrate at SRT of 3, 2, and 1 d. Throughout the study, in the cake layer deposited on membrane, Al, Si, and Fe were detected below 2%.

In the third stage, the aim was to investigate optimum operating conditions for the simultaneous nitrification and denitrification processes to remove organic matter, color and nitrogen-based pollutants in the real textile wastewater using an MBR with an intermittent aeration strategy. The system was first operated at different dissolved oxygen values (DO of 6 and 3 mg/L) and then aeration-on/off durations varying between 2 min/2 min and 90 min/360 min. The average total COD, color, and TN concentrations of the wastewater were measured as 793±173 mg/L, 1171±458 Pt–Co and 65±15 mg/L, respectively. COD removal performance ranged from 84 to 91%. In all tested conditions, color removal performance was highly variable and independent of operating conditions, ranging from 40 to 68%. While ≥89% nitrification performance was achieved in the MBR at a minimum aeration-on durations of 30 min, the highest denitrification efficiency was achieved in the cycle with an aeration-off durations of 360 min. With the intermittent aeration process, higher TN removal, less sludge production and less energy requirement for aeration were achieved. However, membrane fouling profiles occurred more quickly at the aeration-off durations of 60 min and longer. Additionally, while SS, VSS, SF and viscosity values decreased under intermittent aeration conditions, SRF values increased. Although SMP concentrations decreased with intermittent aeration, EPS concentrations were quite similar. While no change was observed in the molecular weights of the supernatant and filtrate samples of the MBR, the average particle sizes in the supernatant increased as the aeration-off time increased. Finally, according to SEM-EDS results, inorganic substances such as Ca, Mg, Si, and Na were detected in the cake-deposited membrane surfaces.

In the fourth stage, the impacts of different aeration patterns on the specific ammonium oxidation, denitritation and denitrification rates were investigated in batch assays. The batch experiments were conducted using the sludge taken from the MBR operated at various DO concentrations and aeration on/off times. While the specific ammonium oxidation rate was determined by batch tests and respirometric studies, specific denitritation and denitrification rates were determined by parallel batch reactors containing different nitrite and nitrate concentrations. The highest specific ammonium oxidation, denitritation, and denitrification rates were obtained as 5.4, 3.8, and 5.3 mg N/(g VSS.h), respectively, at the aeration-on/off durations of 90/360 min. Specific ammonium oxidation rates increased by 1.8 and 2.1 times in the last period (the aeration-on/off time of 90/360 min), compared to continuous aeration conditions with DO of 6 and 3 mg/L, respectively.

# KONVANSİYONEL VE YÜKSEK YÜKLÜ MBR PROSESLERİNDE GERÇEK BİR TEKSTİL ATIKSUYU ARITIM PERFORMANSININ VE ENERJİ GERİ KAZANIMININ ARAŞTIRILMASI

## ÖZET


Dünya çapında en çok su tüketen sektörlerden biri olan tekstil endüstrisinin atıksuları toksik ve karmaşık yapısı nedeniyle alıcı su kütleleri için ciddi tehlike oluşturmaktadır. Tekstil atık sularının arıtımı için pek çok fiziksel, kimyasal ve biyolojik proses araştırılmıştır, ancak bunların hiçbiri tek başına deşarj standartlarını karşılamak için yeterli bulunmamaktadır ve uygulanması gereken her proses yüksek yatırım ve işletme maliyeti gerektirmektedir. Ayrıca tekstil atıksularının arıtımı sırasında döngüsel ekonomiyi uygulayabilmek ve yüksek enerji gereksinimleri, büyük miktarlarda atık çamur üretimi ve sera gazı emisyonları gibi çıktıları en aza indirmek için enerji nötr arıtma tesislerinin dünya çapında yaygınlaştırılması gerekmektedir. Aerobik membran biyoreaktörler (MBR'ler), işletme kolaylığı, daha az alan ihtiyacı ve geleneksel biyolojik yöntemlere göre kıyasla daha yüksek kalitede çıkış suyu sağlaması gibi sebeplerden dolayı tekstil atıksularının arıtımında günümüzde yaygın olarak tercih edilmektedir. Ancak aerobik MBR'ler, hem mikroorganizmaların oksijen ihtiyacını karşılamak hem de membran kirlenmesini en aza indirmek için yüksek miktarlarda enerjiye ihtiyaç duyarlar. Ayrıca tekstil atıksularının ana kirleticileri olan azo ve reaktif boyaların (anaerobik/aerobik kombine sisteme ihtiyaç vardır) ve nitrojen bazlı kirleticilerin (aerobik nitrifikasyon ve anoksik denitrifikasyon işlemlerine ihtiyaç vardır) uzaklaştırılması için aerobik işlem tek başına yeterli değildir. Ayrıca, seçilen proseslerin, tekstil endüstrisi atıksuyunun yüksek hacmi nedeniyle düşük enerji ihtiyacına sahip olması gerekmektedir. Günümüzde su kaynaklarının küresel olarak azaldığı göz önüne alındığında, tekstil endüstrisi atıksularının sadece arıtılması yeterli olmadığı gibi geri kullanımı da son derece önemlidir. Bu nedenle tekstil endüstrisi atıksuları için sürdürülebilir ve pratik arıtma stratejileri geliştirilmelidir.

Bu tezin temel amacı; gerçek bir tekstil atıksuyunun arıtımı için iki farklı biyolojik prosesin arıtma performanslarını belirlemektir. İlk proseste, farklı düşük çamur yaşı ve hidrolik bekletme sürelerinde işletilen ve 0.04 µm gözenek çapına sahip hollow fiber (HF) ultrafiltrasyon (UF) membran içeren aerobik bir MBR'de gerçek tekstil atıksuyunun arıtım performansı ve biyometan üretimi için hammadde kaynağı olarak kullanılabilecek organik maddenin geri kazanımı araştırılmıştır. Ayrıca düşük çamur yaşı (SRT) ve hidrolik bekletme süreleri (HRT) ile farklı SRT/HRT oranlarının membran filtrasyon performansı, çamur karakterizasyonu, çamur üretimi ve havalandırma için gereken enerji gereksinimi gibi parametrelerin üzerindeki etkileri incelenmiş ve tüm parametreler paralel olarak işletilen geleneksel bir aerobik MBR sistemi ile karşılaştırılmıştır. İkinci biyolojik proseste ise 0.04 µm gözenek çapına sahip HF-UF membranı içeren MBR'de hem organik hem de azot kaynaklı kirleticilerin tekstil atık suyundan eş zamanlı olarak giderilmesi için aralıklı havalandırma stratejisinin uygulanabilirliğini ve farklı havalandırma modellerinin membran filtrasyon performansı, çamur karakterizasyonu, çamur üretimi, havalandırma için enerji gereksinimi ve spesifik giderim hızları üzerindeki etkileri

araştırılmıştır. Bu tez kapsamında yapılan tüm çalışmalar aşağıdaki verilen dört ana aşamadan oluşmaktadır.

İlk aşamada; konvansiyonel MBR sistemlerinin tekstil atıksuyu arıtım performanslarını belirlemek amacıyla laboratuvar ölçekli iki aerobik membran biyoreaktör kurulmuş ve aynı atıksu ile beslenen bu sistemlerden biri SRT 30 günde, diğeri sırasıyla SRT 20 ve 10 günde işletilmiştir. İlk aşamada, tesisten alınan ve sistemleri besleyen atıksuyun ortalama toplam kimyasal oksijen ihtiyacı (KOİ), renk ve çözünmüş toplam azot (TN) konsantrasyonları sırasıyla 927±277 mg/L, 910±287 Pt–Co ve 39±10 mg/L'dir. Test edilen tüm SRT değerlerinde KOİ giderim performansı %90'ın üzerinde olup, kayda değer bir farklılık gözlemlenmemiştir. Renk giderim performansı ise 30 günlük ve 20 günlük SRT değerlerinde işletilen dönemlerde sırasıyla %55 ve 60 iken, 10 günlük SRT döneminde %50 seviyelerine düşmüştür. Her iki MBR'de de transmembran basıncı (TMP) çalışma boyunca 10 mbar'ın altında kalmış ve herhangi bir membran tıkanması gözlenmemiştir. Süpernatant filtrelenebilirliği (SF) ve spesifik filtrasyon direnci (SRF) değerlerinde SRT'nin 10 güne düşürülmesiyle artış gözlenmiştir. Askıda katı madde (AKM) konsantrasyonunun SRT düştükçe azalmasına bağlı olarak viskozite değerlerinde de düşme gözlemlenmiştir. Test edilen tüm SRT değerlerinde çözünmüş mikrobiyal ürün (SMP) konsantrasyonu benzer iken hücre dışı polimerik madde (EPS) konsantrasyonu SRT'nin 30 günden 10 güne düşürülmesiyle artmıştır. Jel geçirgenlik kromatografisi (GPC) analizine göre, SRT azaldıkça süzüntüdeki organik maddelerin moleküler ağırlıkları geniş bir spektruma dağılmıştır ve düşük molekül ağırlıklı organik bileşikler daha yüksek sinyaller vermiştir. Son olarak, SRT azaldıkça atık çamur miktarı ve mikroorganizmaların gerçek dönüşüm oranı ($Y_{obs}$) artarken havalandırma için gerekli enerji ihtiyacı azalmıştır.

İkinci aşamada ise SRT/HRT oranları sırasıyla 5, 10 ve 20'de sabit tutularak düşük SRT (0,5 – 5 gün) ve HRT (1,2 – 24 saat) değerlerinde işletilen laboratuvar ölçekli yüksek yüklü aerobik MBR'de tekstil atıksuyu arıtım performansı ve organik madde geri kazanım oranının araştırılması amaçlanmıştır. Bu aşamada kullanılan atıksuyun ortalama toplam KOİ, renk ve TN konsantrasyonu sırasıyla 834±143 mg/L ve 1037±407 Pt-Co ve 51±11 mg/L'dir. KOİ giderim verimi SRT'nin 5, 3 ve 2 gün (tüm SRT/HRT oranlarında) olduğu periyotlarda %86-92 arasında değişirken, SRT'nin 1 ve 0,5 gün (SRT/HRT oranı 10) olduğu periyotlarda sırasıyla %82 ve 77 seviyelerine kadar düşmüştür. Test edilen SRT ve HRT değerleri ile renk giderim performansı arasında bir korelasyon gözlemlenmemiş olup, renk giderim verimi %26 ile 70 arasında değişmiştir. MBR'de nitrifikasyon prosesi 2 gün ve daha düşük SRT değerlerinde tamamen durmuştur. Özellikle, SRT'nin 5 günden 1 güne düşürülmesiyle atık çamur miktarı ve $Y_{obs}$ değerlerinde artış meydana gelirken, SRT/HRT oranı havalandırma için gereken enerji ihtiyacında önemli rol oynamıştır. Ayrıca sistemde SRT'nin düşürülmesi, daha yüksek SRF, daha düşük SF değerlerine ve hızla meydana gelen membran kirlenme profillerine yol açmıştır. Süpernatanttaki SMP konsantrasyonu özellikle $\leq$ 2 günlük SRT periyotlarında artarken, SRT düştükçe üretilen toplam EPS konsantrasyonu artmıştır, fakat her bir SRT değerinde SRT/HRT oranı arttıkça üretilen toplam EPS konsantrasyonu düşmüştür. MBR'den alınan süpernatant ve süzüntü numunelerindeki organik maddelerin moleküler ağırlıklarında 3, 2 ve 1 günlük SRT dönemlerinde (SRT/HRT oranı 10 iken) herhangi bir değişiklik gözlenmezken, membranın kek tabakasında %2'nin altında Al, Si ve Fe varlığı tespit edilmiştir.

Üçüncü aşamada ise aralıklı havalandırma stratejisinin uygulandığı bir membran biyoreaktörde (MBR) gerçek tekstil atıksuyundan organik madde, renk ve azot bazlı kirleticilerin eş zamanlı uzaklaştırılması için optimum işletme koşullarının belirlenmesi amaçlanmıştır. Bu amaçla sistem öncelikle iki farklı çözünmüş oksijen (ÇO) konsantrasyonunda ve daha sonra 2 dak/2 dak ile 90 dak/360 dak arasında değişen havalandırma-aç/kapa sürelerinde işletilmiştir. Bu aşama boyunca kullanılan atıksuyun ortalama toplam KOİ, renk ve TN konsantrasyonları sırasıyla 793±173 mg/L, 1171±458 Pt–Co ve 65±15 mg/L olarak ölçülmüştür. Test edilen tüm koşullarda, KOİ giderim performansı %84 ila 91% arasında değişirken, renk giderim performansı oldukça değişken olup işletim koşullarından bağımsız olarak %40 ile 68 arasında kalmıştır. MBR'de minimum 30 dakikalık havalandırma-aç süresiyle ≥%89 nitrifikasyon performansı elde edilirken, en yüksek denitrifikasyon verimi havalandırma-kapa süresinin 360 dakika olduğu döngüde elde edilmiştir. Aralıklı havalandırma prosesi ile MBR'de daha yüksek TN giderimi, daha az çamur üretimi ve havalandırma için daha az enerji gereksinimi sağlanmıştır. Fakat, aralıklı havalandırma prosesi ile birlikte özellikle havalandırma-kapa süresinin 60 dakika ve daha uzun olduğu periyotlarda membran kirlenmesi daha hızlı gerçekleşmiştir. Aralıklı havalandırma koşullarının sistemde başlamasıyla ayrıca askıda katı madde (AKM), uçucu askıda katı madde (UAKM), SF ve viskozite değerlerinde azalma gerçekleşirken, SRF değerleri artmıştır. Reaktör içinde SMP konsantrasyonlarının aralıklı havalandırma ile azalmasına rağmen EPS konsantrasyonlarında anlamlı bir değişiklik gözlemlenmemiştir. MBR'den alınan süpernatant ve süzüntü numunelerinde organik maddelerin moleküler ağırlıklarında herhangi bir değişiklik tespit edilemezken, havalandırma kapa süresi arttıkça süpernatanttaki ortalama partikül boyutlarında artış gözlemlenmiştir. Son olarak, membran kek tabakasında Ca, Mg, Si ve Na gibi inorganik maddeler tespit edilmiştir.

Son aşamada ise, önceki adımda farklı ÇO konsantrasyonları ve havalandırma aç/kapa sürelerinde işletilen MBR'den alınan çamur örnekleri ile değişen işletme koşullarının mikroorganizmaların spesifik amonyum oksidasyon, denitritasyon ve denitrifikasyon hızların üzerine etkisi araştırılmıştır. Spesifik amonyum oksidasyon hızı, farklı çalışma koşullarında MBR'den alınan çamur ve amonyum içeren sentetik atıksu ile kurulan kesikli reaktörler ve respirometrik çalışmalar ile belirlenirken, spesifik denitritasyon ve denitrifikasyon hızları farklı nitrit ve nitrat konsantrasyonları içeren paralel kesikli reaktörler ile araştırıldı. Testler sonucunda en yüksek spesifik amonyum oksidasyon, denitritasyon ve denitrifikasyon hızları sırasıyla 5,4, 3,8 ve 5,3 mg N/(g UAKM.sa) olarak havalandırma-açma/kapa süresinin 90/360 dk olduğu periyotta elde edilmiştir. Spesifik amonyum oksidasyon hızları 360 dk havalandırma-kapa süresinin uygulandığı son periyotta, ÇO'nun 6 ve 3 mg/L olduğu sürekli havalandırma koşullarına kıyasla sırasıyla 1,8 ve 2,1 kat artış göstermiştir.

# 1. INTRODUCTION

Wastewater treatment and reuse is considered an urgent issue due the severe consumption of fresh water reserves worldwide by human-induced activities. Several biological, physical, chemical, and hybrid wastewater treatment proceses have been investigated and applied using lab-, pilot- or full-scale installations. However, the selection of the most appropriate process is still a matter of debate, and its determination depends on the advantages and limitations in terms of treatment efficiency, applicability, environmental impact assessment, and cost (Crini and Lichtfouse, 2019).

The textile industry is one of the sectors that needs high volumes of fresh water due to production processes applied and the chemicals used (Holkar et al., 2016). Discharging textile industry wastewater effluent to the receiving environment without an appropriate treatment application causes irreversible damage to water bodies and aquatic life (Zaharia et al., 2009). However, textile wastewater treatment is very difficult due to its complexity and variability in composition (Spagni et al., 2010). Many physicochemical and biological treatment processes have been investigated for textile wastewater treatment, and generally, these processes alone do not provide sufficient treatment (Gosavi and Sharma, 2014).

Membrane bioreactors (MBRs) are a promising and innovative system for wastewater treatment in which a membrane is added to activated sludge systems without the need for any secondary or tertiary treatment processes (Melin et al., 2006). High biomass concentrations, which cannot be obtained due to settling in conventional activated sludge systems, can be easily provided with membrane rejection in MBRs (Sözen et al., 2018). Sludge retention time (SRT) is among the most important operating and design factors in determining MBR performance, energy requirement, membrane fouling profile and the amount of waste sludge produced in process. MBRs are generally operated at long SRTs due to their advantages, such as low food/microorganism (F/M) ratio, low amount of sludge production and efficient nitrification performance (Chen et al., 2012). However, high energy demands due to the excessive oxygen requirement of microorganisms is the main limitation of the aerobic MBRs operated at long SRTs (Martin et al., 2011). During the global expansion of the circular economy in wastewater treatment facilities, high energy needs, high waste sludge production and greenhouse gas emissions are considered as serious problems of the traditional treatment plants (Zhang and Liu, 2022). In MBRs, on the other hand, an interesting approach not only for reducing the energy requirement but also for energy recovery is operating the system at shorth SRTs. At low SRTs, organic matters in wastewater are extracted and converted into biomaterials, becoming a new raw material source for biomethane production (Ng and Hermanowicz, 2005a). Organic matter recovery in previous studies in the literature is achieved at low SRTs in domestic/municipal or synthetic wastewaters (Başaran et al., 2014; Ng and Hermanowicz, 2005a, 2005b; Orhon et al., 2016; Sözen et al., 2014). However, no study was found on textile wastewater in MBRs operated at short SRTs (< 5 days), generally known as high-rate MBRs. It is very essential to investigate the processes that can reduce the energy requirements and even provide energy recovery, especially for textile industry wastewater, which is a global sector that consumes high amounts of water and produces wastewater with toxic and mutagenic properties for the receiving environment.

Another worrisome pollutant in textile wastewater is nitrogen-based inorganic/organic substances, as well as organic contaminants, dyes and heavy metals. Especially, the nitrogen concentration of textile wastewater resulting from newly developed and preferred dyeing recipes and the technologies is quite higher and variable (Yılmaz and Sahinkaya, 2023). MBRs are also preferred for nitrogen removal simultaneously with organic matter and dye removal from textile wastewater. However, traditionally, a nitrification process in an aerobic condition and a denitrification process in an anoxic condition are needed for the biological removal of nitrogen-based contaminants (Sun et al., 2015). For this reason, conventional treatment plants are often designed to include anaerobic-anoxic-aerobic reactors (Zhao et al., 2009), which causes operational difficulties, backwashing regularly and higher energy requirements (Zhang et al., 2016). Many processes are being investigated to achieve simultaneous nitrification and denitrification. Intermittent aeration strategy allows simultaneous nitrification/denitrification to occur in only one reactor with aeration on/off cycles. This strategy also reduces aeration-related operating costs and energy requirements and is possible to retrofit into existing treatment systems (Dotro et al., 2011). The simultaneous removal of organic and nitrogen-based pollutants from domestic and some industrial wastewater by an intermittent aeration strategy has been investigated previously (Cheng and Liu, 2001; Lim et al., 2007; Sahinkaya et al., 2017; Zhao et al., 1999). However, simultaneous removal of organic matter, dye and nitrogen from textile wastewater by intermittent aeration are quite limited, especially with real textile wastewater. In addition, optimization of aeration on/off cycles is necessary to achieve successful textile wastewater treatment efficiency with decreased energy requirement.

## 1.1 Purpose of Thesis

The main purposes of the thesis are to examine (a) the suitability of high rate MBRs for organic matter or chemical oxygen demand (COD) recovery from real textile wastewater, and (b) nutrient removal and energy minimization with intermittent aeration process in a MBR treating real textile wastewater at conventional SRT and hydraulic retention time (HRT) values. Four different studies were carried out within the scope of this thesis, which are summurized as follows:

- Investigation of textile wastewater treatment and filtration performances of two lab-scale aerobic MBRs at different SRTs (30, 20 and 10 d) and at a fixed HRT of 1 d.
- Investigation of treatment and filtration performances of high rate-MBR treating real textile wastewater at different SRTs (5, 3, 2, 1 and 0.5 d) and HRTs (1.2 – 24 h) combinations, corresponding to three different SRT/HRT ratios of 5, 10 and 20.
- Investigating the impact of various intermittent aeration patterns on nutrient removal efficiency and filtration performances in a lab scale aerobic MBR at different dissolved oxygen (DO) concentrations (6 and 3 mg/L) and the aeration on/off durations (from 2 min/2 min to 90 min/360 min) for textile wastewater treatment.
- Determination of specific ammonium oxidation rate, denitritation, and denitrification rates in batch reactors using the biomass from the MBR operated under different DO and aeration on/off times.

## 1.2 Literature Review

### 1.2.1 Textile industry wastewater

The textile industry is a rapidly growing sector on a global scale, and is more prevalent in China, India, Pakistan, Brazil, Bangladesh, Malaysia, Sri Lanka and Vietnam due to their positive contribution to employment and economy (Kishor et al., 2021; Slama et al., 2021). In addition to its significant contribution to the economy, especially in developing countries, it is also known for its high freshwater consumption and wastewater production that threaten human health, the receiving environment and the aquatic ecosystem (Al-Mamun et al., 2019). Approximately 200-350 $m^3$ of wastewater is produced for each ton of final product in the textile industry, and it constitutes 2.1% of the total industrial water consumption (Nidheesh et al., 2022).

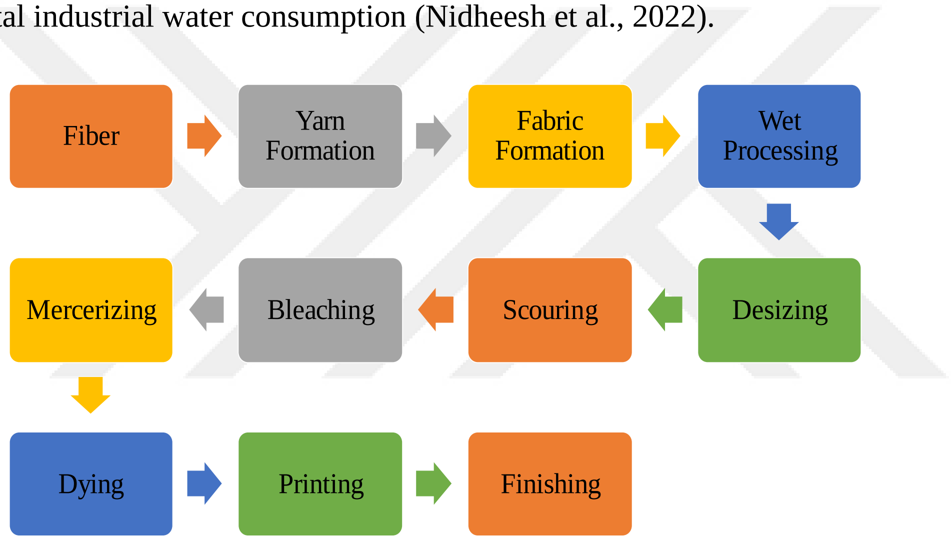


**Figure 1.1 :** General process flow diagram for textile factory (adopted and modified from Madhav et al., (2018)).

The textile industry aims to produce natural or synthetic fibres such as wool, cotton, acetate-cellulose ester, polyester, silk and casein, nylon, and acrylic (Keskin et al., 2021). Depending on fibre production, textile industries are divided into two categories, dry and wet processes (Figure 1.1). While solid wastes are generally occurred in the dry process, wastewater is mostly generated in the wet stages (Holkar et al., 2016). In the wet stages, many complex and different stages are applied during the conversion of raw materials into the final product, using high amounts of chemicals and fresh water (Kishor et al., 2021). Especially wastewater is produced during pre-treatment, dyeing, printing and finishing stages in wet processing (Wang et al., 2022). According to a study, approximately 72% of the water consumed in a textile company is used in the wet processing, and 70% and 13% of this consumption occurs in the washing and dyeing stages, respectively (Figure 1.2) (Panda et al., 2021).

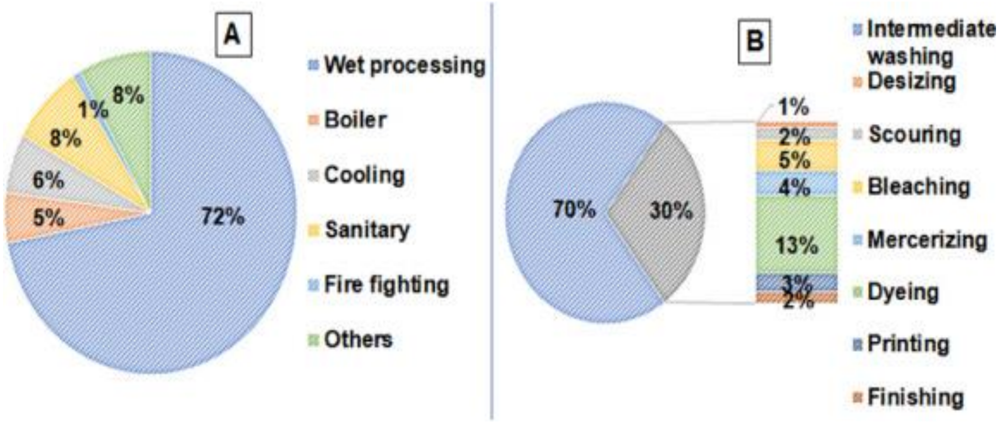


**Figure 1.2 :** Water consumption for various needs in the textile sector (A), and distribution of water consumption for wet stages (B) (Panda et al., 2021).

Textile wastewater is characterized by high levels of chemical oxygen demand (COD), total suspended solids (TSS), biochemical oxygen demand (BOD), color, turbidity, heavy metal, pH and temperature (Hossain et al., 2018; Tianzhi et al., 2021). Generally, the water consumption is closely related to the raw material and final product used (Correia et al., 1994), however characterization of the produced wastewater varies depending on many parameters such as raw materials, the stages used during production, chemicals, and machinery (Bisschops and Spanjers, 2003). Large volumes of water is needed for the cotton production while silk, wool and synthetic fibres need less water per product compared to cotton fibres (Ahsan et al., 2023). The major sources of the pollutants are the dyeing and finishing stages due to different chemicals and dyestuffs used (Al-Kdasi et al., 2004) such as toxic and hazardous chemicals, dyes, heavy metals, starch, detergents, chelating agents, surfactants, salt and suspended solids (Dhruv Patel and Bhatt, 2022; Nidheesh et al., 2022). Dyeing wastewater generally has high salt content, color and alkalinity, while finishing wastewater contains organic pollutants (Patel and Vashi, 2015). However, the chemicals and dyestuffs used do not completely turn into the final product and are mixed into the water stream (Al-Kdasi et al., 2004). Determining of the characterization of typical composite textile wastewater is quite difficult due to the differences in the applied processes between industries (Khandare et al., 2013; Yaseen and Scholz, 2019).

### 1.2.2 Treatment of textile industry wastewater

The main environmental issues related to textile industry are fresh water consumption, discharge and the proper treatment of wastewater (Yusuff and Sonibare, 2004). Textile wastewater should be discharged into receiving water bodies after appropriate wastewater treatment to prevent detimental environmental impacts (Ozturk and Cinperi, 2018).

Textile wastewater has a very complex structure (Sharma et al., 2007), and is being investigated to find an economical and efficient treatment with physicochemical, biological and combined treatment processes (Siddique et al., 2017). While membrane processes, adsorption and ion exchange are preferred as physical methods during the textile wastewater treatment, chemical methods are coagulation, electrocoagulation and oxidation (Paździor et al., 2019). However, these processes have very limiting disadvantages such as inadequacy of chemical coagulation for dissolved reactive dye removal, the higher cost and secondary waste problem in activated carbon adsorption, and the economic problems in ozonation, photocatalysis, fenton reagent and ultrasonic oxidation processes (Can et al., 2006). In addition, many of these methods require external chemicals addition (Alinsafi et al., 2005).

Biological treatment of textile wastewater is often recognized as challenging due to its high toxic nature and low biodegradable potential of the pollutants (Ledakowicz and Gonera, 1999). However, biological treatment methods are highly adaptable to a wide variety of wastewater compositions (Mkilima et al., 2021), and different studies have found biological processes successful for the textile wastewater treatment. It is very attractive with its advantages such as being more cost-effective, environmentally friendly, less sludge production, non-hazardous intermediate and end products, and less fresh water requirement over the physical/oxidation process (Holkar et al., 2016).

Biological processes are based on the principle of oxidation or reduction of organic or inorganic pollutants in wastewater by various microorganisms (Paździor et al., 2019). Aerobic treatment systems are highly effective to remove suspended solids, pathogens, COD, biochemical oxygen demand (BOD) and pollutants from the textile wastewater (Mullai et al., 2017). However, the removal of azo and reactive dyes, which are among the primary pollutants of textile wastewater, in oxidative catabolism is quite low due to their lack of electrons, and they are not suitable for traditional aerobic treatment systems due to their hydrophilic structure (Manu and Chaudhari, 2002). Azo dyes are degraded to aromatic amines by accepting four electrons in the azo bond in the presence of carbon sources as electron donor in the anaerobic process, and thus the color caused by azo dyes is removed from the wastewater. However, aromatic amines with carcinogenic and mutagenic properties cannot be removed from wastewater due to partial mineralization under anaerobic conditions (Şen and Demirer, 2003). Since traditional aerobic processes cannot break down most azo dyes in textile wastewater and aerobic microorganisms are needed to degrade aromatic amines formed by the dye breakdown in anaerobic processes, combined systems are the most effective way to treat textile wastewater (Işık and Sponza, 2008).

Textile wastewater has been biologically treated in many different aerobic, anaerobic and/or combined reactor configurations such as sequencing batch reactor (SBR), stirred tank bioreactor (STR), continuously stirred tank bioreactor (CSTR), upflow anaerobic fixed bed (UAFB), biological aerated filter (BAF), fluidized bed reactor (FBR) and MBR. Abu-Ghunmi and Jamrah (2006) reported 80-95% COD removal at a reaction time of 22-25 h and the $S_0/X_0$ (BOD/MLSS) ratio between 0.13 and 0.5 in an SBR. Andleeb et al. (2010) reported around 84.53% color, 66.50% BOD and 75.24% COD removals at 50 ppm of initial dye and HRT of 24 h in an STR. Kim et al. (2002) investigated performances of a combined system including fluidized biofilm process-chemical coagulation-electrochemical oxidation. While COD and color removals were 68.8 and 54.5% in the fluidized biofilm reactor with low SRT and MLSS values, they increased to 95.4 and 98.5% at the end of the combined system. Chang et al. (2002) reported 99% BOD, 92% COD, 74% suspended solid (SS) and 92% total nitrogen (TN) removals at hydraulic loading rate of 1.83 $m^3/m^2$-h in a pilot-scale BAF. Sandhya and Swaminathan (2006) achieved 84.80% COD and 90% color removal in two hybrid column UAFB reactors. Shoukat et al. (2019) obtained 99.5% COD, 99.3% TKN and 78.4% dye removal in a hybrid anaerobic SBR-aerobic SBR. Işık and Sponza (2008) reported 83-97% COD and 80-87% color removal at HRT of 3.3 days in a sequential anaerobic UASB/aerobic CSTR. Haroun and Idris (2009) obtained 98% soluble COD, 95% $BOD_5$ and 65% color removals in an FBR with the addition of 600 mg/L glucose. Although researches for full-scale wastewater treatment derived from different textile processes is still ongoing, studies covering MBR systems (discussed in the following title), which are generally preferred for the municipal and other industrial wastewater treatment, are quite limited and their full potential for textile wastewater has not yet been revealed (Jegatheesan et al., 2016).

### 1.2.3 Membrane bioreactors for textile industry wastewater treatment

MBRs are more preferred in domestic and industrial wastewater treatment for approaches such as reuse and recovery of wastewater, thanks to their excellent wastewater quality and limited footprint (Meng et al., 2009; Wang et al., 2014).

An MBR consists of a CAS combined with a membrane process to kept biomass within the bioreactor. Since the membrane pore size preferred in the MBRs is generally below 0.1 μm, they produce high quality treated and disinfected wastewater and provide high biomass concentration in the bioreactor (Santos et al., 2011). MBRs are applied in two ways: external/sideflow configuration and submerged/immersion configuration (Figure 1.3). In the external/sideflow, the membrane is outside the tank, and recirculation is applied. In the other configuration, membrane modules are placed inside the tank containing the mixed liquid. While the membrane driving force is provided by a high cross-flow in the external configuration, approaches such as pressurizing the tank or using negative pressure on the filtrate channel are preferred in the internal configuration (Lin et al., 2012). While external MBRs are preferred for wastewater having higher temperature, pH, organic content, toxicity and low filterability, submerged configuration has generally been preferred for the domestic wastewater treatment (Yang et al., 2006).

The MBRs are further categorized mainly based on material type, pore size, and module of the membrane. Two membrane materials are generally preferred as polymeric and ceramic, and modules are generally classified as multi-tube (MT), hollow fiber (HF) and flat sheet (FS) (Deowan et al., 2015). The membrane pore sizes are divided into four classes; ultrafiltration (UF) and microfiltration (MF), nanofiltration (NF) and reverse osmosis (RO) (Pearce, 2008). While UF and MF (0.01–0.1 μm pore size) are mostly suitable for the separation of treated wastewater and sludge and other solids (Melin et al., 2006), NF and RO are efficient for rejection of some pathogenics, organics and inorganics (Goswami et al., 2018). Considering the studies reported, MF and UF are widely applied during municipal/domestic/industrial wastewater treatment (Gander et al., 2000; Juang et al., 2013; Kitanou et al., 2018; Qin et al., 2007; Subtil et al., 2014) and NF/RO membranes are preferred in water recovery stages or removal of certain pollutants or inorganic compounds (Asik et al., 2021; Alturki et al., 2010; Gander et al., 2000; Kimura et al., 2003).

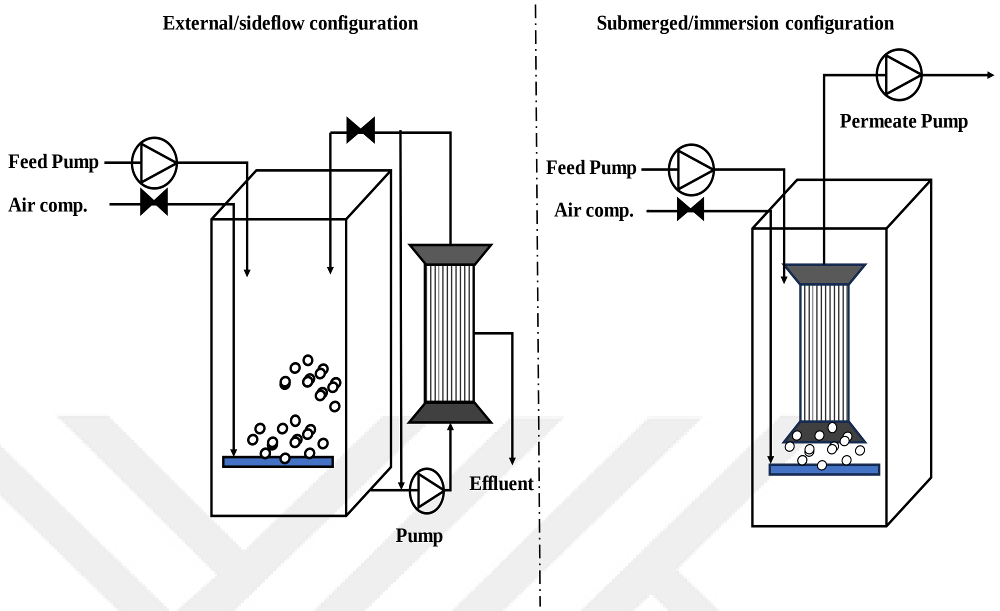


**Figure 1.3 :** Commonly used MBR configurations (adopted and modified from Lin et al., (2012)).

During the wastewater treatment, MBRs can be operated as aerobic, anoxic, anaerobic, or combined systems, and the selection of the process depends on considering many factors, including but not limited to the characterization of the wastewater, the operation condition and cost, and targeted discharge standards. However, generally all MBR configurations have attractive advantages such as no need for a settling tank, high removal efficiency due to higher biomass retention, minimization of sludge settling problems, lower reactor volume and low sludge production (Marrot et al., 2004).

Despite all their advantages, MBRs require higher energy costs compared to conventional treatment systems. The major important reason for the high cost of energy is the high SRT values applied in MBRs. MBRs are generally used to both minimize sludge at high SRT values and obtain high quality wastewater with low suspended solids concentration, without any sludge settling problems (Pollice et al., 2008). However, high concentrations of sludge are retained in the bioreactor at high SRT values, but this increases the amount of oxygen needed by microorganisms and aeration cost (Ittisupornrat et al., 2021). Other reasons for high energy costs is membrane fouling, which occurs when the membrane pores are blocked by suspended solids, dissolved substances and sludge flocs, and membrane fouling negatively affects membrane filtration performance and significantly reduces membrane lifespan (Iorhemen et al., 2016). The formation of membrane fouling profiles generally depends on some parameters such as feed and biomass characteristics, operating conditions, HRT, membrane type and especially SRT (Isma et al., 2014). However, conflicting findings have been reported on the effect of SRT on membrane fouling, then more detailed studies are needed (Jinsong et al., 2006).

MBR performance is influenced by a variety of factors such as wastewater characterization, bacterial activity, membrane type and configuration (Cinperi et al., 2019). Anaerobic MBR systems have lower energy needs since they do not require oxygen and also are very attractive with low sludge production and methane production. However, the fouling tendency of anaerobic MBRs is higher than aerobic MBRs. One of the main reasons for this is thought to be higher SMP production in anaerobic MBRS, which clogs membrane pores in MBR, and therefore anaerobic MBR is not generally preferred in the wastewater treatment (Jegatheesan et al., 2016), especially having high flow rate. However, lab- and pilot-scale research studies have been conducted on the textile wastewater treatment in anaerobic MBRs (Spagni et al., 2012; Yurtsever et al., 2021, 2020). Although the anaerobic process is generally preoffered for the wasteeater having high organic matter concentration, aerobic processes are better at removing soluble organic matter and have better treated wastewater quality compared to anaerobic system (Chan et al., 2009). Nowadays, aerobic MBR processes are becoming widespread in textile wastewater treatment due to their high COD removal efficiency of more than 90% (Samsami et al., 2020) and the treated wastewater is suitable to be fed for further reuse processes, such as reverse osmosis. The studies on textile wastewater treatment using aerobic MBRs are provided in Table 1.1, and it shows that most of the studies were conducted at high SRTs (>10 days), and no study was found on textile wastewater treatment with MBRs operated at short SRTs (< 5d), known as high-rate MBRs. This subject will be discussed in detail in the flowing chapter.

**Table 1.1 :** Studies conducted with aerobic MBRs for the textile wastewater treatment.

| Wastewater Source | Membrane Type | SRT (d) | Removal efficiency (%) | | | References |
|---|---|---|---|---|---|---|
| | | | COD | Color | N | |
| Mixed textile wastewater | Tubular ultrafiltration membrane | - | 97 | 70 | 70 (as $NH_3$-N) | (Badani et al., 2005) |
| Reconstituted reactive BB150 dye wastewater | External tubular cross-flow MF | - | 80–93 | 91–100 | - | (Khouni et al., 2020) |
| Synthetic textile wastewater | Ceramic flat sheet membrane module | 30 | 93.1 | 87.1 | - | (Sari Erkan et al., 2020) |
| Real textile wastewater | Crossflow UF flat sheet | - | 100 | 98 | - | (Friha et al., 2015) |
| Synthetic textile wastewater | UF-HF membrane | - | 92.33 | 91.36 | - | (Niren and Jigisha, 2011) |
| Textile mill wastewater | HF immersed membrane modules | infinite | 70 | 62 | 28 (as TN) | (Cinperi et al., 2019) |
| Concentrated mixed wet processes wastewater | Submerged HF membrane module | no sludge wastage (P1) and SRT of 25 d (P2) | >95 | >97 | 78 (as high as TN) | (Yigit et al., 2009) |
| Textile industry effluent | External UF membranes | 45 | 89–92% | 72-73% (with UF) | - | (Lorena et al., 2011) |
| Local laundry textile wastewater | Flat sheet UF membrane module | - | 90% | - | - | (Deowan et al., 2019) |
| Local textile industry wastewater | HF membrane module | - | 91 | 80 | 90 (as TN) | (Yang et al., 2020) |

MBRs operated at high SRT values increase nitrogen removal efficiency by keeping slow-growing bacteria such as nitrifiers in the system, but both aerobic and anoxic stages are required for a complete nitrogen removal through nitrification and denitrification processes (Holakoo et al., 2007). MBR systems including aerobic/anoxic/anaerobic combined systems have been examined in many research articles. For example, a sequential system consisting of an anaerobic, and anoxic reactor and then aerobic MBR was operated to treat textile auxiliary wastewater at different recycling rates. An average of 87% COD, 96% $NH_4^+$–N, and 55% TN removal efficiencies were obtained, and the organic matter and recycling rate showed a serious effect on the treatment performances (Sun et al., 2015). However, to both reduce operating costs and minimize operating difficulties, various strategies such as micro-aeration (Sun et al., 2018), intermittent aeration (Yoo et al., 1999), and the formation of aerobic and anoxic medium inside microbial biofilms and flocks (Chiu et al., 2007) for simultaneous nitrification and denitrification processes have been investigated. However, studies on complete nitrogen removal of textile wastewater in a single MBR system are quite limited and further research is required. This subject will be discussed in detail in the flowing chapter.

### 1.2.4 High-rate membrane bioreactors with low SRTs

SRT has a decisive effect on the removal and filtration performances of MBRs (Xia et al., 2012). MBRs are theoretically suitable for operation at infinite SRTs due to the advantages mentioned in the previous topics, but generally infinite SRT has many disadvantages such as excessively higher biomass concentration in the bioreactor, reduced aeration efficiency, biomass decay and sludge mineralization (Van den Broeck et al., 2012). Currently, MBRs are mainly operated at SRT values ranging from 25 to 3500 d, with sludge concentrations in the bioreactor varying from 10,000 to 50,000 mg/L (Ng and Hermanowicz, 2005a). Although long SRTs (low F/M ratio) provide high nitrification efficiency, low waste sludge generation, less sludge disposal problems and the associated operating costs, the removal performance is not linearly proportional to SRT and the concentrations of dead or inactive microorganisms in MBRs are quite higher (Chen et al., 2012). In addition to SRT, HRT is another operating parameter that determines the biomass concentration and filtration performance in the MBR, and the SRT/HRT ratio determines the biomass concentration in a biological system at the steady-state condition (Xu et al., 2015). The equation below is used to determine biomass concentration in a biorector. According to this equation, amount of the biomass in a reactor must be the same for the same SRT/HRT ratios, ignoring variations in other parameters (Ng and Hermanowicz, 2005a). In addition, by reducing SRT in a system operated at a constant HRT, the concentration of biomass in the system decreases.

$$\mathbf{X} = \mathrm{Y}(\mathrm{S_{in}} - \mathrm{S_{out}})\left(\frac{\mathrm{SRT}}{\mathrm{HRT}}\right)\left(\frac{1}{1 + \mathrm{K_d SRT}}\right) \tag{1.1}$$

$X$ = biomass concentration

$Y$ = yield coefficient

$S_{in}$ = influent substrate concentration

$S_{out}$ = effluent substrate concentration

$Q_x$ = SRT

$Q_h$ = HRT

$k_d$ is = the biomass decay rate

The activated sludge process with low SRTs (1-4 d) and HRTs (2-4 h) (Jimenez et al., 2015), also known as high rate activated sludge system (HRAS) aims to remove particulate and colloidal COD with minimum energy requirements through two mechanisms. In the first mechanism, the particulate and colloidal part of the wastewater remain in the sludge directly by bioflocculation instead of completely converting it into carbon dioxide (Faridizad et al., 2022), while soluble part of the organic matter is used in intercellular storage, microbial growth and carbon oxidation (Canals et al., 2023). While wastewater discharge standards are ensured by the partial soluble organic matter (or COD) removal from wastewater at low SRTs, the caloric value and COD concentration of the mixed liquid of the reactor becomes suitable for methane production in an anaerobic digester by the bioflocculation of the particle and hydrolysable organic matter (Başaran et al., 2014). The COD in the wastewater is mostly removed by oxidation at high SRTs, which results in generation of sludge with low methane production potential, but at low SRT values, higher portion of the COD in the watstewater is transferred to the sludge (Jimenez et al., 2015). The second mechanism is carbon harvesting, where organic matter is recovered through before being metabolized by microorganisms and is exposed to anaerobic digestion for methane production, but settling problems is the main limitation in carbon harvesting (Canals et al., 2023). Additionally, bioreactors designed with shorth SRTs require lower aerobic tank blower capacity due to lower MLSS concentration, which reduces the aeration requirements (Verrecht et al., 2010). However, it is very difficult to obtain both high organic matter recovery and high quality effluent in HRAS (Rocco et al., 2023). MBRs, on the other hand, allow direct reuse of high-quality effluent by rejecting many pollutants such as suspended solids (SS), bacteria and viruses (Sun et al., 2007), and due to this advantage, high-rate MBRs operating at SRTs of 5 to 2 d are being examined for the wastewater treatment (Rocco et al., 2023). Operating conditions and results of different biological systems operated under low SRT and HRT values reported are given in Table 1.2. Especially, high rate-MBRs have higher

COD removal and harvesting efficiencies compared to other conventional short SRT activated sludge process configurations.

**Table 1.2 :** Literature-reported operating conditions and results of processes operating at exceptionally low SRTs.

| Process | Wastewater type | SRT | HRT | COD removal (%) | Main findings | References |
|---|---|---|---|---|---|---|
| **HRAS** | Municipal wastewater | 0.35 d | 130 min | 49 | Higher total COD removal efficiency at low HRTs due to removal of particulate COD by flocculation and sedimentation. | (Guven et al., 2017) |
| | | 0.35 d | 95 min | 53 | | |
| | | 0.35 d | 60 min | 59 | | |
| **HRAS** | Municipal wastewater | 0.24 ± 0.07 d | 59.5 ± 0.5 min | 62 | Higher methane yield with the addition of food waste to wastewater. | (Guven et al., 2019) |
| | A mixture of municipal wastewater and food waste | 0.19 ± 0.18 d | 60.7 ± 1.9 min | 63 | | |
| **HR- aerobic system** | Effluent of a sewage biofilm reactor | 0.5-3 d | 0.5 h | 35.6-84.6 | The anaerobic degradability of sludge increased from 66% to over 80% by reducing SRT from 3 to 0.5 d | (Ge et al., 2017) |
| **High-rate SBR** | Abattoir wastewater | 2–3 d | 0.5–1 d | >80 | 70-80% of carbon removal with biomass assimilation and/or accumulation in the system. | (Ge et al., 2013) |
| **HR-contact-stabilization** | Chemically enhanced primary treatment effluent of a municipal wastewater | 1.1 d (total) | 33 min | 52–59 | Carbon redirection and recovery with low SRTs. | (Rahman et al., 2016) |
| **High loaded membrane bioreactor** | Synthetic municipal wastewater | 0.5, 1 and 2 d | 2.5 h | 93-94 | Higher removal efficiencies and membrane performances at the shorter SRT | (Faridizad et al., 2022) |
| **Super-fast MBR** | 200 mg/L peptone mixture | 0.5, 1 and 2 d | 8 h | ≥86 | Energy conversion between 54% and 77% | (Sözen et al., 2018) |
| | 250 mg/L settled sewage | 0.5, 1 and 2 d | 8 h | ≥82 | | |
| **Super-fast MBR** | 200 mg COD/L | 0.5, 1 and 2 d | 1 h | ≥91.5 | Faster process kinetics with low SRTs | (Başaran et al., 2014) |
| **MBR** | Synthetic wastewater | 0.25 to 5 d | 3 and 6 h | 97.3–98.4 | Higher yield and biomass decay coefficients in MBR | (Ng and Hermanowicz, 2005a) |
| **Completely mixed activated sludge** | | 0.25 to 5 d | 3 and 6 h | 77.5–93.8 | | |

High-rate MBRs also allow minimizing aeration problems, reducing energy requirements, and using waste biosolids and sludge for energy production without any sludge settling problems and escaping suspended solids in the effluent of the system

(Duan et al., 2009). Sözen et al. (2018) reported that energy conversion (based on particulate COD) of an MBR operated at SRT of 1 d was 1.4, 1.6 and 2.3 times higher than that of the HRAS, CAS and extended aeration process, respectively (Sözen et al., 2018). However, one of the most worrying and costly basic problem in MBRs is membrane fouling, and there are quite conflicting results regarding the contribution of long or short SRTs to membrane fouling. For example, it is generally reported that short SRTs cause low membrane filtration performance due to high EPS production (Silva et al., 2017). Although overall fouling resistance in submerged MBRs increases at higher SRT values (Lee et al., 2003), longer SRT is mainly considered to be favorable for membrane performance (Al-Halbouni et al., 2008; Le-Clech et al., 2006). Therefore, detailed studies are needed on the wastewater treatment and membrane filtration performance of MBRs operated at low SRT values. However, as seen in Table 1.2, no study has been found in the literature on the textile wastewater treatment in high rate MBRs.

### 1.2.5 Intermittently aerated membrane bioreactors

Domestic or industrial wastewater should be treated before being discharged to reduce eutrophication conditions in receiving environment (Nagaoka and Nemoto, 2005). Biological methods are generally preferred in removing carbon, nitrogen and phosphorus from wastewater. Especially small-scale wastewater treatment plants are not sufficient for nutrient removal and have limited nitrification/denitrification efficiency depending on operating conditions (Abegglen et al., 2008). Nitrogen removal in biological processes occurs in two different processes. In the first stage, the ammonium is oxidized first to nitrite and then to nitrate by autotrophic bacteria in the presence of oxygen. In the second stage, produced nitrate and/or nitrite is reduced to $N_2$ with organic matter in an anoxic condition (Sun et al., 2017). The traditional processes, especially anaerobic-anoxic-aerobic ($A_2O$), are mainly preferred in removing nitrogen based pollutants, but these processes have serious disadvantages such as their complex operation, high energy needs, low effluent quality, waste sludge problems and low phosphorus removal (Y.-K. Wang et al., 2015).

Alternative cycle processes (aerobic/anoxic phase in a single reactor using intermittent aeration) have been successfully implemented to upgrade existing treatment facilities (Capodici et al., 2016). The intermittent aeration process facilitates both ammonium oxidation and nitrate reduction by allowing aerobic and anoxic conditions in a single tank (Campo et al., 2017). In addition, intermittent aeration reduces the aeration requirements, which cover approximately 50% of the total energy consumption in treatment facility (Ratanatamskul and Kongwong, 2017), and provide sufficient carbon for subsequent denitrification process (Lim et al., 2012).

Intermittent aeration, which can also reduce sludge yield by 30% compared to traditional nitrification-denitrification, has been investigated in various system such as MBBR, AS and MBRs (Miao et al., 2022). Table 1.3 presents studies on the optimization of intermittent aeration for simultaneous nitrification/denitrification during different wastewater treatment, and studies on the industrial wastewater treatment with intermittent aeration are quite limited.

**Table 1.3 :** Literature-reported operating conditions and the results of processes employing intermittent aeration.

| Process | Wastewater type | Aeration on/off time (min/min) | COD removal (%) | N removal (%) | References |
|---|---|---|---|---|---|
| **SBR** | Slaughterhouse wastewater | 50/50 | 96 | 96 | (Li et al., 2008) |
| **Intermittent aeration tank** | Anaerobically Digested Swine Wastewater | 60/60 | 57 | 91 (as TKN) | (Cheng and Liu, 2001) |
| **Modified Ludzack-Ettinger** | Municipal wastewater | 51/45 | 96 | 89 | (Liu and Wang, 2017) |
| **MMBR** | Rural wastewater | 60/60 | 77.7 | 54.3 (as TIN) | (Luan et al., 2022) |
| **Fixed-bed reactor** | Effluent of a UASB treating slaughterhouse | 60/120 | >88 | 62 | (Barana et al., 2013) |
| **Combo system (FBR-MBR)** | Synthetic wastewater | 60/60 | 99.5±0.5 | 99.1±0.8 (as $NH_4^+$-N) | (Guadie et al., 2014) |
| **MBR** | Synthetic wastewater | 60/60 | 96% | 92.9% | (Choi et al., 2008) |
| **MBR** | Domestic wastewater | 90/60 | 87.6 - 98.1 | 35-70% | (Lim et al., 2004) |
| **MBR** | Domestic wastewater | 50/70 | 95.5 | 86.9 | (Lim et al., 2007) |

The DO concentration in an intermittently aerated system is very decisive in ensuring the effective operation of the process and generally depends on the aeration on/off times tested (Hidaka et al., 2002). Additionally, to ensure simultaneous effective removal, it is significant to determine the presence of relevant bacteria and how their performance is affected under different operating conditions (Mota et al., 2005). Therefore, optimizing the aeration on/off time according to each treatment process configuration and wastewater characterization is crucial to achieve maximum removal efficiency in the system with intermittent aeration. Moreover, although MBRs provide excellent removal performances in terms of carbon, nitrogen, color, solids, and high-quality treated wastewater for reuse strategies during the textile wastewater treatment, a detailed study on the optimization of intermittent aeration for textile wastewater treatment in an MBR has not yet been found.

## 1.3 Unique Aspect

Organic recovery for further energy production, reducing operating costs, water recovery, and nutrient removal are challenges that must be addressed during textile wastewater treatment. Although organic recovery and effective wastewater treatment have been investigated for domestic and synthetic wastewaters using MBRs operated at low SRTs (He et al., 2023; Ng and Hermanowicz, 2005a; Orhon et al., 2016), no research on textile industry wastewater has been located. Therefore, the textile wastewater treatment in the high-rate MBR system was investigated in detail for the first time in this thesis.

Dye removal in a dynamic MBR using synthetic wastewater was optimized by intermittent aeration process (Sahinkaya et al., 2017), however no studies were found in the literature on optimizing nutrient removal in an MBR using real wastewater with an ultrafiltration membrane under different aeration patterns. In addition, the determination of the ammonium oxidation rate by using a respirometer and the specific denitritation and denitrification rate by setting up batch reactors at different aeration on/off times adds a distinctive originality to the thesis. In addition, energy consumption and sludge production were compared in all conditions tested in the systems. All studies carried out in the thesis have provided innovative results that will shed light on pilot- and full-scale processes.

## 1.4 Organization of the Thesis

The thesis consists of an introduction (Chapter 1) and four data chapters (Chapters 2-5). Lastly, in the sixth chapter (Chapter 6) the comprehensive conclusion of the thesis and recommendations for future studies are given.

Chapter 2 includes the data obtained during the real textile wastewater treatment in two aerobic lab-scale MBRs at SRTs of 30, 20 and 10 d, which are often used in the literature and considered as conventional SRT values. The COD, color and nutrients removal performances, membrane filtration performances and membrane foulant characterizations, $Y_{obs}$ values, sludge amount and energy requirements were compared for the SRTs of 30, 20 and 10 d.

Chapter 3 includes the data obtained during the real textile wastewater treatment in an aerobic lab-scale MBR at SRTs of 5, 3, 2, 1 and 0.5 d and SRT/HRT ratio of 5, 10 and 20, respectively. The COD, color and nutrients removal performances, organic matter recovery performances, membrane filtration performances and membrane foulant characterizations, $Y_{obs}$ values, amount of sludge produced and energy requirements for aeration were compared for all tested different SRTs and SRT/HRT combinations.

Chapter 4 includes the data obtained during the optimization of intermittent aeration process for a lab-scale MBR for nitrogen removal from real textile wastewater at SRT of 30 d and HRT of 1 d, respectively. The nitrogen, COD and color removal performances, membrane filtration performances and membrane foulant characterizations, $Y_{obs}$ values, amount of sludge produced and energy requirements for aeration were compared for all tested different aeration patterns.

Chapter 5 includes the batch tests results and respirometric studies carried out to investigate the variations of specific ammonium, denitrification, and denitrification rates of microorganisms exposed to different DO concentrations and different aeration on/off times in the MBR, results of which provided in the previous chapter.

Lastly, chapter 6 provides this thesis' conclusions and recommendations for future research.

# 2. EFFECT OF SLUDGE RETENTION TIME ON THE PERFORMANCES AND SLUDGE FILTRATION CHARACTERISTICS OF AN AEROBIC MEMBRANE BIOREACTOR TREATING TEXTILE WASTEWATER

## 2.1 Introduction

Textile industries generate a huge amount of wastewater containing toxic pollutants such as dyes, salts, metals and surfactants (Friha et al., 2015; Leaper et al., 2019). It has been reported that 0.06-0.40 $m^3$ of water is consumed per kg of manufactured material in the textile industry (Laqbaqbi et al., 2019). According to the World Bank, about 17 to 20% of industrial water pollution occurs during the wet processing stages of manufacturing. (Holkar et al., 2016). Biological, physical, and chemical treatment methods are widely preferred in the treatment of textile wastewater (Holkar et al., 2016). These conventional treatment options inevitably yield high amounts of sludge to be properly disposed of. Biological methods have several advantages over the others such as low cost and less sludge production (Holkar et al., 2016). In particular, membrane bioreactors (MBRs) provide opportunities such as less sludge production and high-quality effluent compared to other treatment systems (Khouni et al., 2020). One of the most decisive factors during the operation of MBR processes is the solid retention time (SRT). Both short and long SRTs provide different advantages in the operation. Operating an MBR at long SRTs ($\geq$ 25 d (Ng and Hermanowicz, 2005a)) offers advantages such as higher performance due to higher suspended solid (SS) concentrations (Van den Broeck et al., 2012) and lower sludge production. However, long SRTs decrease the amount of active microorganisms in the sludge (El-Fadel et al., 2018) and cause an increase in the oxygen requirement (Jiang et al., 2008). Short SRTs reduce oxygen and energy requirements (Duan et al., 2009) but increase the excess sludge amount, which constitutes a significant portion of the operating cost

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This chapter based on the paper 'Yilmaz, T., Demir, E. K., Asik, G., Başaran, S. T., Çokgör, E. U., Sözen, S., & Sahinkaya, E. (2023). Effect of sludge retention time on the performance and sludge filtration characteristics of an aerobic membrane bioreactor treating textile wastewater. Journal of Water Process Engineering, 51, 103390, DOI: 10.1016/j.jwpe.2022.103390'.

(Zhang et al., 2021). Excess sludge generated in wastewater treatment plants may be used for energy generation (Berktay and Nas, 2007; Seiple et al., 2020).

Membrane fouling and membrane cleaning costs are also significant disadvantages for the MBRs (You et al., 2006). In terms of membrane filtration and fouling, longer SRTs may provide better filterability, while shorter SRTs result in faster membrane fouling profiles. This may be due to the amounts of soluble microbial products (SMP) and extracellular polymeric substances (EPS) production depending on the SRT (Jiang et al., 2008). It is known that the amount of SMP increases, as the SRT decreases (Al-Halbouni et al., 2008; Kimura et al., 2009). However, different results were reported about the relationship between EPS generation and SRT (Massé et al., 2006).

In a study, synthetic textile wastewater containing 2000 mg/L COD, 2000 mg/L sulfate, and 200 mg/L dye was fed to sequential anaerobic and aerobic MBRs operated at different SRTs (infinite, 60, and 30 d). The COD concentrations in the anaerobic and aerobic MBR permeates averaged 579±231 mg/L and 58±19 mg/L despite different SRTs in all the operational conditions (Yurtsever et al., 2017). In another study, the treatment performance of synthetic wastewater containing 3500 mg/L COD and 100 mg Reactive Black 5 dye was investigated at SRT and HRT values of 15 d and 36 h, respectively, in an aerobic MBR system equipped with a membrane module with 0.4 µm pore diameter. The COD and color removal efficiencies were reported as 94.8 and 72.9%, respectively (Yun et al., 2006). You et al. (2006) studied the treatment of Kuagnin Industrial Park wastewater using an aerobic MBR equipped with a 0.4 µm pore-sized membrane. They reported COD and color removal efficiencies as 79% and 54%, respectively. In another study using fungi for treatment, synthetic wastewater containing 2 g/L TOC and 100 mg/L dye was fed to an MBR containing a membrane module with a pore diameter of 0.4 µm in which 97% TOC and 99% color removal efficiencies were obtained (Hai et al., 2006).

Ultrafiltration membrane (UF) is generally preferred as a pre-treatment before NF (Debik et al., 2010) and/or RO (De Jager et al., 2014) processes during the recovery of treated textile wastewater. In a pilot-scale study using a submerged UF membrane (0.04 µm pore size) module, the treatment of real textile industry wastewater containing 1411 mg/L COD, 49.2 mg/L total nitrogen and 2447 Pt-Co color was investigated. Two different SRTs (infinite and 25 d) were used and the average permeate COD, TN and color concentrations were 37 mg/L, 10.5 mg/L and 53 Pt-Co, respectively (Yigit et al., 2009).

There are many studies on textile wastewater treatment using anaerobic and/or aerobic MBRs as reported above. However, synthetic textile wastewater was used in some of the studies (Deowan et al., 2013; Yurtsever et al., 2017) which may not exactly reflect the process performance for a real wastewater treatment. In some of the studies, real textile wastewater was treated using MBR processes at long SRTs (Cinperi et al., 2019; Deowan et al., 2019; Kozak et al., 2021; Lorena et al., 2011) without focusing on the impact of SRT on the performance, filtration characteristics and energy requirement of the process. Hence, to fill the gap in the literature, this study aims to investigate and compare the treatment performance of a real textile industry wastewater at different SRTs (30, 20 and 10 d) in an aerobic MBR equipped with a hollow fiber UF membrane module with a pore diameter of 0.04 μm. Membrane fouling trends and filtration performances under different SRTs were discussed. Also, observed biomass yield, air and energy requirements were calculated at different SRTs.

## 2.2 Materials and Methods

### 2.2.1 Bioreactors

Two laboratory-scale submerged membrane bioreactor (MBR) systems (with an active volume of 7.0 L) were used (Figure A1). A feed tank was used to feed the systems with wastewater, and the influent and permeate flow rates were adjusted using peristaltic pumps (Seko (PR7) and Longer Pump (BT600-2J, respectively). ZeeWeed 500-1M model PVDF hollow fiber membrane module with a pore diameter of 0.04 μm was used in the systems and the membrane surface area was 0.23 $m^2$. In both systems, air with a flow rate of 3 L/min was supplied to the system through a compressor. Hence, air provided both for biological treatment and cake scouring on the membrane was 0.78 $m^3$-air/($m^2$-membrane.h). Transmembrane pressure (TMP) during filtration was monitored with a manometer placed in the permeate line.

### 2.2.2 Wastewater and biomass source

The wastewater used in this study was obtained from a plant treating industrial zone wastewater in Bursa, Turkey. It is known that 64 companies supply wastewater to the plant with a total flow rate of 50,000-60,000 $m^3$/d and 95% of the wastewater originates from textile industry (Sahinkaya et al., 2019). The characterization of the wastewater, during the operating periods, is given in Table 2.1, which remained fairly constant throughout the study. Total COD, color and soluble TN of the wastewater averaged 927±277 mg/L and 910±287 Pt-Co and 39±10 mg/L, respectively. The wastewater characteristics were similar to the ones reported earlier by other studies in the literature conducted with wastewater from the same plant (Sahinkaya et al., 2019). The seed biomass (~ 6 g VSS/L) for the MBRs was also taken from the sludge recirculation line of the same wastewater treatment plant.

**Table 2.1 :** Characterization of wastewater used throughout the study depending on the operating periods.

| Parameter | Unit | Average ± stdv |
|---|---|---|
| **pH** | | 7.9±0.4 |
| **Total COD** | mg/L | 927±277 |
| **Soluble COD** | mg/L | 472±121 |
| **Color** | Pt-Co | 910±287 |
| **Soluble TN** | mg/L | 39±10 |
| **$N\text{-}NH_4^+$** | mg/L | ≤1 |
| **$PO_4\text{-}P$** | mg/L | 2.5±0.8 |
| **Alkalinity** | mg $CaCO_3$/L | 824±124 |
| **Conductivity** | μS/cm | 6141±833 |
| **$N\text{-}NO_2^-$** | mg/L | ≤1 |
| **$N\text{-}NO_3^-$** | mg/L | ≤1 |

### 2.2.3 Operating conditions

The two aerobic MBRs were operated in parallel. One of them was operated for 256 days at an SRT of 30 d and the other for 84 and 68 days at SRTs 20 and 10 d, respectively. The HRT was kept constant at 1 d in both MBRs operated at room temperature. Excess sludge was removed daily from both systems to keep the SRTs constant at the tested values. The filtration process was run intermittently (5 min suction, 1 min rest) to reduce membrane fouling. Instantaneous (or gross) flux values were calculated as 1.23, 1.20 and 1.14 LMH at SRTs of 30 d, 20 d, and 10 d, respectively. Due to the intermittent operation of the systems, the net flux values varied as given in Table 2.2.

**Table 2.2 :** Operating conditions of aerobic MBRs.

| SRT (d) | HRT (d) | SRT/HRT | $Q_{daily\ sludge}$ (L/d) | $Q_{permeat}$ (L/h) | Net Flux (LMH) | Gross Flux (LMH) | $Q_{air}$ (L/min) |
|---|---|---|---|---|---|---|---|
| **30** | 1 | 30 | 0.233 | 0.282 | 1.23 | 1.47 | ≥ 3 |
| **20** | 1 | 20 | 0.350 | 0.277 | 1.20 | 1.45 | ≥ 3 |
| **10** | 1 | 10 | 0.700 | 0.263 | 1.14 | 1.37 | ≥ 3 |

In order to determine the treatment performances, COD, color, alkalinity, conductivity, total nitrogen, ammonium nitrogen, SS, and VSS were measured in the influent, supernatant (the soluble part within the MBR), and permeate of the MBRs. In addition to determining the membrane filtration performance, TMP, specific resistance to filtration (SRF), supernatant filterability (SF), and capillary suction time (CST) were regularly monitored/measured. SMP and EPS were also measured for the characterization of membrane foulants.

### 2.2.4 Critical flux

One of the factors determining the filtration performance and fouling frequency in an MBR is the selected flux value. Generally, MBRs are operated at subcritical flux with limited fouling (Navaratna and Jegatheesan, 2011). Hence, a critical flux test was conducted in the MBR operated at an SRT of 30 d (Figure A3). In the test, the MBR was operated at different fluxes for a total duration of 175 min. The flux value was gradually increased from 1.25 to 35 LMH and the filtration duration at each flux was 20 min. At each flux change, filtration was interrupted for 5 min to improve the physical cleaning of the membrane surface and to remove the foulants from the previous filtration step.

### 2.2.5 Specific energy requirements

The energy requirement for aerobic treatment is dominated by oxygen transfer during the aeration process. The overall oxygen requirement of an aerobic system is calculated from the following equations (Park et al., 2015):

$$\mathrm{OD_{theory}} = \mathrm{Q(S_0 - S_e) + 4.32QN_{ox} - 2.86Q\left(N_{ox} - NO_{3,e}\right) - 1.42}P_{X,bio} \quad (2.1)$$

$$\mathrm{OD_{field}} = \left(\frac{\mathrm{OD_{theory}}}{\mathrm{E}}\right)\left(\frac{\mathrm{SOTR}}{\mathrm{AOTR}}\right) = \left(\frac{\mathrm{OD_{theory}}}{\mathrm{E}}\right)\left(\frac{\mathrm{DO_{S,20}}}{\alpha.\,(\beta \mathrm{DO_s} - \mathrm{DO}).\,\theta^{\mathrm{T}-20}}\right) \quad (2.2)$$

$\mathrm{OD_{theory}}$: Theoretical oxygen requirement, (mg/d)

$OD_{field}$: Field oxygen requirement, (mg/d)

Q: Wastewater flow rate, (L/d)

$S_0$: Influent COD concentration, (mg/L)

$S_e$: Effluent COD concentration, (mg/L)

$N_{ox}$: Oxidizable ammonia concentration, (mg/L)

$NO_{3,e}$: Nitrate nitrogen concentration, (mg/L)

$P_{x,bio}$: Biological sludge wasted daily, (mg VSS/d)

E: Oxygen transfer efficiency of diffuser, (unitless)

SOTR: Standard oxygen transfer rate

AOTR: Actual oxygen transfer rate

$DO_{S,20}$: Oxygen saturation concentration, (mg/L)

DO: actual dissolved oxygen concentration, (mg/L) (assumed 2 mg/L)

α: Oxygen transfer correction factor (typical 0.3-1)

α is assumed a function of MLSS $\alpha = e^{-0.08788.MLSS}$

β: Wastewater correction factor for oxygen solubility (assumed 0.95)

Θ: Temperature correction factor (assumed 1.024)

T: temperature (assumed 20 °C)

Air flow rate is calculated via the following equation:

$$\mathrm{Q_{air}} = \frac{\mathrm{OD_{field}}}{\mathrm{d_{air}}.\,(\text{kg O2 in 1 kg air})} \cdot \left(\frac{1\ \text{day}}{24\ \text{hr}}\right) \qquad (2.3)$$

$Q_{air}$: Required air flow rate, (L/h)

$d_{air}$: Density of air (assumed 1.2 kg/m$^3$)

The specific energy requirement for aeration is the aeration energy requirement per m$^3$ of wastewater being treated and it is calculated based on the air flow rate requirement. The specific energy requirement for aeration was calculated for each SRT based on the equations provided above.

The energy requirement in an MBR may vary between 0.6 and 2.3 kWh/m$^3$ depending on the size of the plant and the selected treatment processes and operational conditions (Krzeminski et al., 2017). In our short-term critical flux experiment, it was observed that the sustainable flux may be in the range of 20-30 LMH and the MBR may be successfully operated at ~20 LMH with a maximum TMP of 0.3 bar. The energy requirement for permeate pumping may be calculated based on the equation provided by Kim et al. (2011).

$$P = \frac{Q\gamma E}{1000} \tag{2.4}$$

Where P is the required power as kW, Q is the permeate flow rate as m$^3$/s, ɣ is 9800 N/m$^2$, and E is hydraulic head as m.

### 2.2.6 Observed yield

$Y_{obs}$ is the amount of biomass generated per substrate utilized, which can be calculated from the following relation.

$$Y_{obs} = \frac{Px}{Q(S_0 - S)} \tag{2.5}$$

$P_X$: Biomass generation and assumed excess sludge wasted daily as g SS/d.

$S_0$: COD concentration in the influent

S: COD concentration in the permeate

### 2.2.7 Analytical methods

COD, alkalinity, SS, and VSS were measured according to Standard Methods (APHA, 2005). Dionex ICS-5000+ (Thermo Fisher Scientific, USA) ion chromatography equipped with an AS9-HC column was used for nitrate and nitrite measurements. HACH HQ40d multimeter 190 was used for conductivity and pH measurements. TN, ammonium nitrogen, and $PO_4$-P were measured using HACH kits according to the manufacturer's instructions. SMP and EPS samples were prepared according to the methods described by Judd and Judd, (2006) and Rahman et al., (2017), respectively. SMP and EPS were measured as carbohydrates (Dubois et al., 1956) and protein (Bradford, 1976) concentrations in the samples. For SRF analysis, the sludge sample was filtered with a 0.45 µm pore-sized membrane using a dead-end system under a pressure of 0.5 bar in the absence of mixing. In the SF analysis, the supernatant obtained by centrifugation of the sludge for 10 min was filtered through a membrane filter with a pore diameter of 0.22 μm using a dead-end filtration apparatus under constant pressure (0.5 bar) and stirring (Dereli et al., 2014). Standard filter papers supplied by Trion Electronics and the Trion Capillary Suction Timer were used for CST measurements. Viscosity of sludge samples was measured using a Brookfield DV-E brand viscometer and the values were calculated using the Newtonian model. Molecular weight distribution of soluble organic matter was determined using an Agilent 1260 Infinity brand GPC equipped with two PL Aquagel-OH Mixed-H columns.

#### 2.2.8 Statistical analysis

The statistical analysis of the data was performed by one-way analysis of variance (ANOVA) using the Data Analysis Tool of Excel 2019 (Microsoft Corporation, USA) to statistically compare the values of the different parameters monitored during each SRT. The significant difference was set at 95% ($p \leq 0.05$).

### 2.3 Results and Discussion

#### 2.3.1 Treatment performances in aerobic MBRs

Two aerobic MBRs were operated in parallel with real textile wastewater. One of the MBR systems was operated for 256 days at an SRT of 30 d. No significant changes were observed in the influent composition except for total COD increasing up to 1660 mg/L for very short durations around days 100, 155, and 230 (Figure 2.1). Organic matter removal performance was not affected by the COD surges experienced in this period, and effluent COD averaged 77±20 mg/L in permeate (Figure 2.1) corresponding to an average COD removal efficiency of around 91% at an SRT of 30 d. The average color in the permeate was decreased to 396±101 Pt-Co, corresponding to 55% color removal (Figure 2.2). In permeate, the alkalinity decreased to an average of 512±59 mg $CaCO_3$/L corresponding to approximately 38% alkalinity consumption during the treatment process.

The second aerobic MBR was operated in parallel, again with textile wastewater, initially at an SRT of 20 d for 84 days. MBR performance was not affected by the decrease of the SRT and/or the 10% increase in the influent soluble COD concentration. The average COD was observed around 70±33 mg/L in the permeate and the removal efficiency was 93% (Figure 2.1). Color in the influent and permeate averaged 930±291 Pt-Co and 356±83 Pt-Co, respectively, corresponding to 60% color removal at this SRT (Figure 2.2). During this period, a slight increase in pH was observed while the alkalinity was measured as 501±38 mg $CaCO_3$/L in the permeate corresponding to an average of 32% alkalinity consumption.

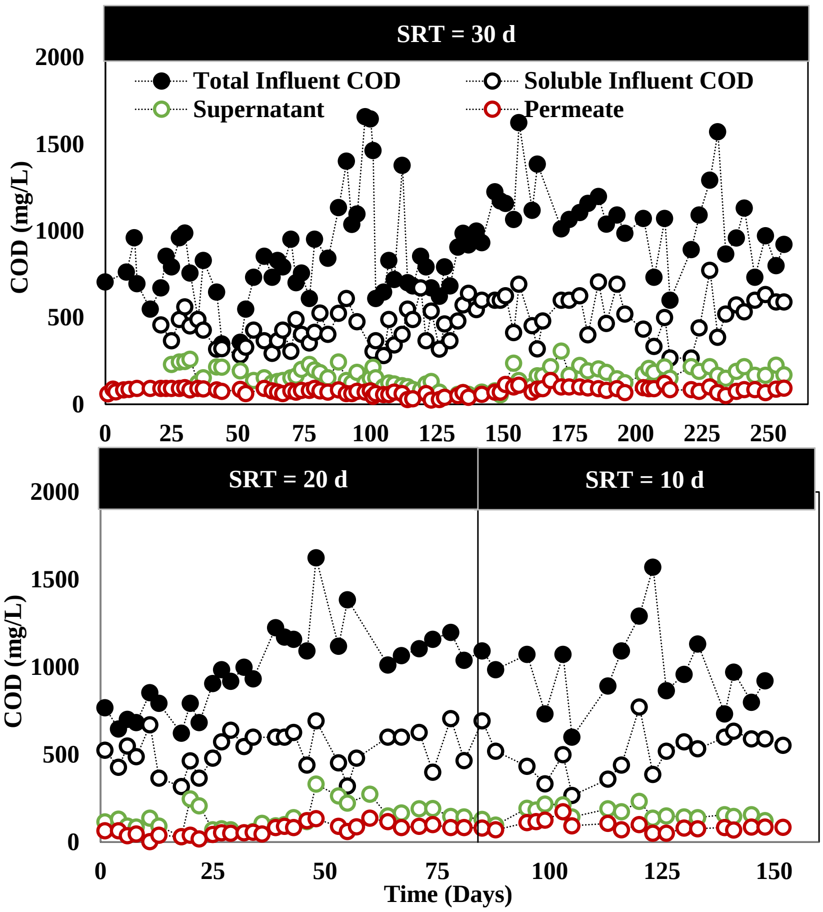



**Figure 2.1 :** Variations of COD concentrations in the MBRs throughout the study.

In the last period, the SRT was kept at 10 d and the MBR was operated for approximately 68 days. While the influent composition remained essentially the same, decreasing the SRT to 10 d resulted in an average permeate COD of 90±28 mg/L (90% removal efficiency) (Figure 2.1). The average color, which was 1091±331 Pt-Co in wastewater, decreased to 51899 Pt-Co in the permeate. While the color removal efficiency was 55 and 60% at SRTs of 30 d and 20 d, respectively, it decreased to 50% at the SRT of 10 d ($p < 0.05$). (Figure 2.2). The average alkalinity concentration in the permeate was 561±92 mg $CaCO_3$/L, and around 40% of the alkalinity was consumed at the SRT of 10 d.

Employing an SRT in the range of 30 to 10 d for the treatment of textile wastewater with MBR revealed that the system was able to perform at high organic removal efficiencies above 90% at all times ($p < 0.05$). The overall organic removal performance in the MBR was a result of biological degradation. The amount of the SMP generated in the reactor and its size distribution which determines its rejection by the cake layer are affected by the systems SRT. The impact of the membrane and cake layer and the fate of SMP in the system are further discussed in the following sections.

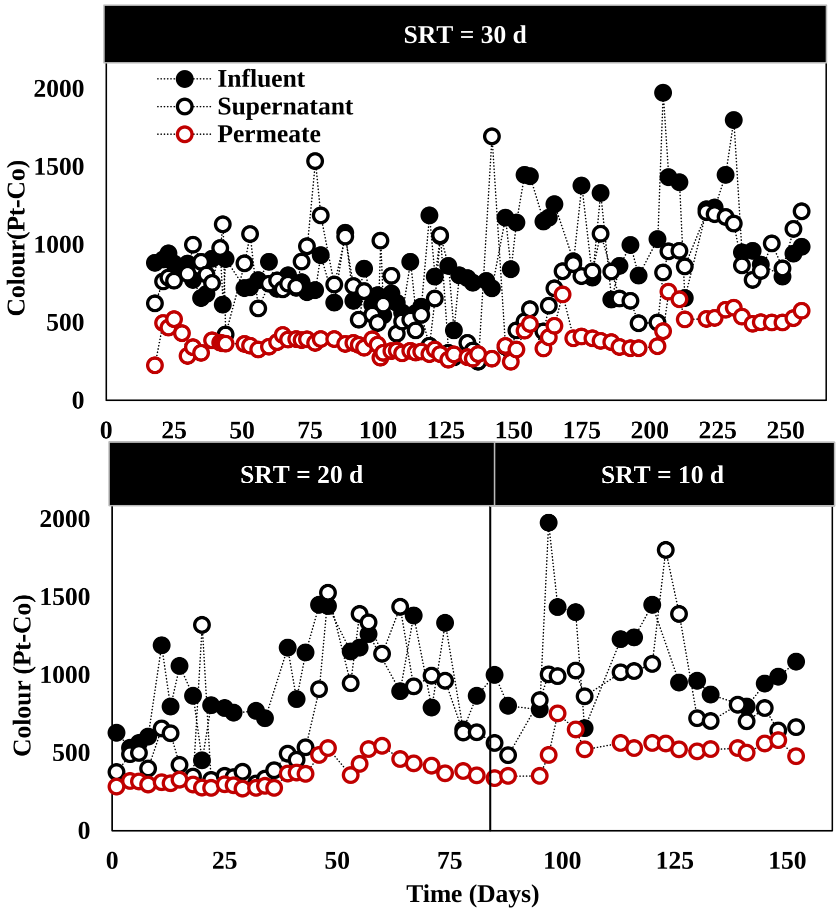



**Figure 2.2 :** Variations of color concentrations in the MBRs throughout the study.

Textile industry uses different types of dyes including reactive, disperse, acid, basic, azo, and sulfur dyes in its processes. Dyes that are commonly used include azo dyes that are insoluble hence they are more easily separated. Others including direct, reactive, acid, and basic dyes are known to be highly soluble hence conventional separation techniques are inefficient. As reported in the literature, biodegradation plays a limited part due to the persistent nature of textile dye chemicals and the main mechanism is often the adsorption of dye molecules onto biomass in the case of insoluble dyes (Li et al., 2016). In this study, color removal decreased to 50% as SRT was decreased to 10 d from an average of 56% at SRTs of 30 d and 20 d. Consistent with the lower amount of biomass contribution retained in the bioreactor the contribution of cake layer to color removal also decreased with decreasing SRTs to 10 d.

No appreciable changes were observed in the pH and conductivity during the whole operating periods testing SRTs of 10 d, 20 d, and 30 d. These results resonate well with the reported literature for MBR treatment of textile wastewaters where similar trends were observed in terms of organics and color removal.

In their review, Jegatheesan et al. (2016) summarized numerous findings from aerobic MBR treatment of textile wastewater, where COD removal efficiencies ranged between 60 and 97%, mostly around 90% regardless of SRT, color removal on the other hand ranged between 20 and 98%, averaging around 50%. In the study by Yang et al. (2020), a UF membrane was used to investigate the treatment of textile wastewater and they reported COD and color removal efficiencies as 91% and 80%, respectively. In another study, COD and color removal efficiencies from synthetic textile wastewater using an aerobic MBR at SRT of 30 d were reported as 93.1 and 87.1%, respectively (Sari Erkan et al., 2020). Cinperi et al. (2019) reported 70 and 62% COD and color removals from textile industry wastewater using a pilot-scale MBR at infinite SRT. In the current study, although the SRT value was reduced to 10 d, the COD and color removal efficiencies remained above approximately 90% and 50%, respectively (Figure 2.2).

### 2.3.2 Nitrification and phosphorus removal performances

Nitrogen content is expressed as total Kjeldahl nitrogen (TKN) and/or total nitrogen (TN) in wastewater. While ammonia nitrogen and organic nitrogen form TKN content, total nitrogen (TN) includes nitrite and nitrate-based nitrogen besides TKN (Deng et al., 2021). Ammonium, nitrite, and nitrate in the wastewater were below 1 mg N/L throughout the study (Table 2.1). In this case, the nitrogen source of the wastewater was likely to be organic nitrogen. Soluble or dissolved organic nitrogen refers to the filtered organic nitrogen (Zheng et al., 2021).

The average soluble nitrogen concentration of wastewater was 39±10, 31±8 and 41±13 mg N/L at the SRTs of 30 d, 20 d, and 10 d, respectively. Soluble nitrogen removal efficiencies averaged 14%, 22% and 12% at the SRTs of 30 d, 20 d, and 10, respectively ($p > 0.05$). As mentioned, the highest TN removal efficiencies were achieved at the SRT of 20 d, similar to other parameters. During the periods in which SRT was 30 d and 10 d, the average N-$NO_3^-$ was measured as 37±13 mg/L and 41±11 mg/L in the permeate, respectively. However, the average N-$NO_3^-$ in the permeate was only 21±7 mg/L at SRT of 20 d. Ammonium in the permeate was around 1 mg N/L at all the SRTs. N-$NO_2^-$ accumulation was observed on certain days of MBR operation. For example, at SRT of 30 d, during days 154 and 168, the permeate nitrite increased up to 30 mg N/L. Likewise during operation at the SRT of 20 d, corresponding to the 39$^{th}$ and 60$^{th}$ days, N-$NO_2^-$ concentration increased around 32 mg N/L. These deteriorations were observed very shortly and the system quickly recovered back to the previous steady-state values. For the MBR operation at an SRT of 10 d, unlike SRTs of 30 d and 20 d, nitrite accumulation was detected between 7 and 19 mg N/L in the permeate during the first 35 days indicating a lower nitrification efficiency. However, in the following days, the nitrification process performance eventually recovered eliminating nitrite accumulation in the permeate.

It is known that nitrification performance is enhanced at higher SRTs since this allows sufficient time to secure the slow-growing nitrifiers in the system (Innocenti et al., 2002). Badani et al. (2005) reported 70% ammonium oxidation performance during real textile wastewater treatment in a pilot-scale aerobic MBR (Badani et al., 2005). In another study, COD and $NH_3$ removal efficiencies of over 90% from domestic wastewater were reported at all the tested SRTs (5, 10, 20 and 40 d) (Huang et al., 2001). In our study, the average $PO_4$-P concentrations in influent averaged 2.5±0.8, 2.5±0.9, and 2.8±1.1 mg/L at SRTs of 30, 20, and 10 d, respectively. In permeate, $PO_4$-P averaged 1.6±0.7, 1.6±0.8 and 1.9±1 mg/L, respectively, with a corresponding removal efficiency of around 35% for all SRTs.

### 2.3.3 Impact of membrane cake layer on the treatment performances

The cake layer is formed by the accumulation of microorganism and their residues, organic substances and inorganic substances on the membrane surface during filtration in an MBR (C. Wang et al., 2015), and its amount is directly related with SRT, HRT (Huang et al., 2011), SS concentration (Chang and Kim, 2005) and filtration flux. In addition, the cake layer on the membrane surface can contribute positively to the degradation and/or rejection of organic and inorganic substances (Yurtsever et al., 2015). Table 2.3 shows the removal efficiencies of the main parameters and the contribution of the membrane and/or cake layer to them. For total COD, the removal efficiency was above 90% at all operated SRTs.

**Table 2.3 :** Variation of removal efficiencies at different SRTs.

| **Parameter** | **COD** | | | **Color** | | |
|---|---|---|---|---|---|---|
| **SRT (d)** | **30** | **20** | **10** | **30** | **20** | **10** |
| **Removal efficiency inside of reactor (%)** | 82±10 | 86±7 | 83±5 | 21±23 | 31±24 | 23±15 |
| **Removal efficiency due to membrane and cake layer (%)** | 10±7 | 8±7 | 8±3 | 40±24 | 29±24 | 32±21 |
| **Total Removal efficiency (%)** | 91±4 | 93±2 | 90±4 | 55±12 | 60±10 | 50±13 |

The contribution of membrane and cake layer to the color and COD removals decreased with decreasing SRT, which should be due to the reduced thickness of the cake layer depending on the SS concentration in the MBR. The color removal within the MBR operated at an SRT of 20 d was 31% compared to 21-23% at SRTs 30 and SRT 10 d. This may be due to the differences in the feed wastewater composition, which could be more biodegradable during the operation of the MBR at the SRT of 20 d. Despite the decrease in SS and VSS concentrations in the reactor due to the SRT decrease, the treatment performances were very close at all the tested SRTs.

### 2.3.4 Impact of SRT on membrane fouling and filtration performances

The average SS and VSS were 2849±296 mg/L and 2276±192 mg/L, respectively, during the first 147 days of the operation at the SRT of 30 d. In the following days, SS and VSS increased by an average of 43% and 50%, respectively (Figure 2.3). This increase corresponds to the period when the total COD and color of the wastewater were increased. Compared with the SRT of 30 d, SS and VSS decreased by approximately 19% and 26%, respectively, at the SRT of 20 d (Figure 2.3). This decrease in SS and VSS concentrations reached 35% and 34%, respectively, at SRT of 10 d. It was observed that the VSS/SS ratios ranged between 0.77 and 0.86.

In the short-term critical flux test, an increase in TMP was observed at a flux of 30 LMH. As a result, the critical flux was decided to be between 20 and 30 LMH. No fouling was observed in the MBR throughout the operation due to subcritical flux operation (<1.5 LMH) and TMP remained below 10 mbar throughout the study (Figure A2).

SRF provides information on sludge filterability and compaction capacity of the cake layer (Szabo-Corbacho et al., 2022). In this study, the average SRF was $5x10^{14}$±$1.73x10^{14}$ m/kg, $5.71x10^{14}$±$1.07x10^{14}$ m/kg and $8.34x10^{14}$±$2.39x10^{14}$ m/kg at SRTs of 30, 20 and 10 d. A significant increase in SRF occurred, especially at SRT of 10 d. This may be due to the increase in colloidal substances in the system due to the decreased SRT and the pore blocking of the membrane with the colloidal organics. In the study conducted with synthetic textile wastewater with sequential anaerobic and aerobic MBRs, the averages of SRF and CST were measured as 1.33±$0.4x10^{14}$ m/kg and 27±9 s at SRTs of infinite, 60 d and 30 d, in an aerobic MBR and it was reported that there was no correlation between SRT and the SRF and CST values (Yurtsever et al., 2017).

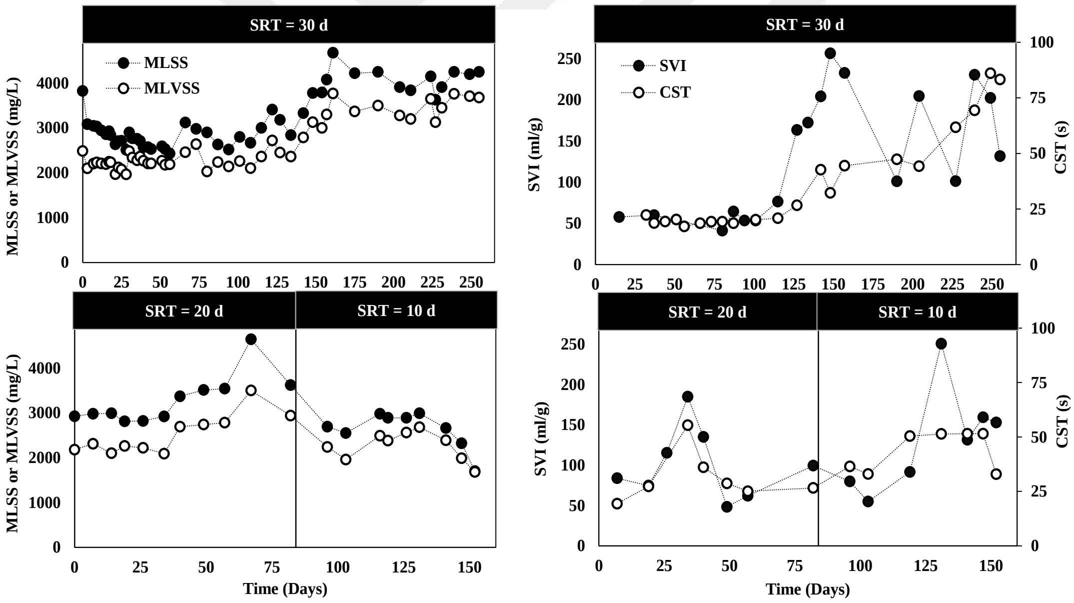



**Figure 2.3:** Variations of SS, VSS, SVI and CST in the MBRs throughout the study.

Supernatant filterability is about the presence of substances that causes mainly pore blocking (Dereli et al., 2014). It was observed that the SRF increased by 1.7 times as the SRT decreased from 30 d to 10 d. No significant change was observed in the SF by decreasing the SRT from 30 d to 10 d, and it remained between 0.77 and 0.86 mL/min. The average viscosities of the sludge were 2.85±0.11, 2.48±0.1, and 2.17±0.11 cP, respectively, at SRTs of 30, 20, and 10 d. As expected, sludge viscosity increases at high SS concentrations in the MBR (Meng et al., 2007). During the first

127 days at an SRT of 30 d, SVI and CST were 71±39 mL/g and 20±2 s, respectively (Figure 2.3). Subsequently, SVI and CST increased to 257 mL/g and 86 s, respectively, and both parameters showed a fluctuating pattern at an SRT of 30 d. SVI and CST were measured as 100±44 mL/g and 31±12 s, respectively, at an SRT of 20 d. By reducing the SRT to 10 d in the MBR, the SVI increased from 76±19 mL/g to 174±53 mL/g in the last days of the period, while the CST remained at 44±9 s during this period. Hence, no exact correlation between SVI and CST was observed. In a study conducted for the treatment of chromium-containing synthetic textile wastewater, a dynamic membrane bioreactor was operated under intermittent aeration conditions at an SRT of 60 d. The MBR was constantly aerated and no chrome was added during the first two periods. In these periods, the SRF was measured as $2.2x10^{12}$ m/kg. Also, it was reported that CST and SVI values were not affected by the changes in operating conditions and were measured as 21.42±2.9 s and 90±32 mL/g, respectively, throughout the study (Sahinkaya et al., 2017).

The average SMP in the supernatant ranged between 31 and 36 mg COD/L at all the SRTs. Approximately 53, 35, and 46% of the SMP in the supernatant were rejected by the membrane and/or cake layer at the SRTs of 30, 20, and 10 d, respectively. EPS averaged 33±3, 31±4 and 41±7 mg COD/g VSS at the SRTs of 30, 20 and 10 d, respectively. Although EPS production changed depending on the SRT, the content of EPS in terms of carbohydrates and protein was not changed. In the MBRs, tightly bound EPS was between 64% and 68%, while loosely bound EPS was between 36% and 32%. In addition, it was observed that the carbohydrate and protein contents of the produced SMP and EPS were close to each other even at different SRTs (Figure 2.4).

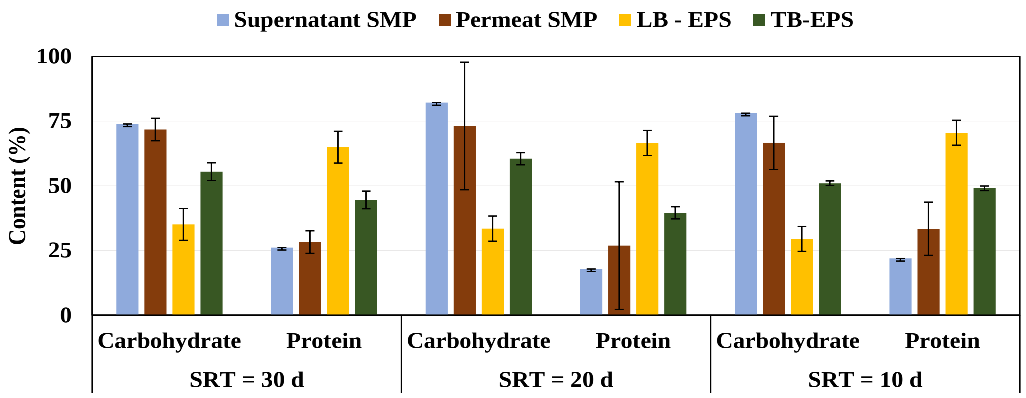



**Figure 2.4 :** Variations of SMP and EPS content produced under different SRTs in terms of carbohydrate and protein.

While the carbohydrate content of SMP in the supernatant ranged from 74% to 82%, it decreased to approximately 67-73% in the permeate. The highest protein content was detected in loosely bound EPS, it ranged from 65% to 70% at all the SRTs. The carbohydrate and protein content of the tightly-bound EPS were 51-60% and 40-49%, respectively, for all SRT values. In a study without SRT information, the textile wastewater treatment performances and effect of HRTs and powdered activated carbon (PAC) addition were investigated in sequential anaerobic moving bed bioreactor (AnMBBR) and aerobic MBR. In the absence of PAC addition, the carbohydrate content of SMPs in the supernatant and permeate were 32.83 and 25.48 mg/L, while the protein content of SMPs was 4.1 and 3.7 mg/L, respectively. The carbohydrate and protein contents of EPS were reported as 7.16 and 1.17 mg/g MLVSS (Kozak et al., 2021), and it was lower than those obtained in this study. It has been reported that the amount of EPS decreases as SRT increases (Massé et al., 2006; Mutamim et al., 2013). In this study, EPS increased from 31 mg COD/g VSS to 41 mg COD/g VSS, especially by reducing SRT to 10 d.

Gel Permeation Chromatography (GPC) was used to measure the molecular weight and presence of the organic substances in the wastewater and permeate. In GPC analyses, four main peaks were observed in the wastewater corresponding to 30, 10, 2.6 and 0.3 kDa molecular weights (Figure 2.5). In the permeate, only three peaks were observed corresponding to 20, 3 and 0.57 kDa at an SRT of 30 d. The peaks observed during the SRT of 20 d correspond to approximately 21, 2.8, 1 and 0.47 kDa. Also, similar peaks were obtained at an SRT of 10 d, corresponding to 22, 3.7, 1.4 and 0.5 kDa. No peak corresponding to 1 kDa was observed at the SRT of 30 d. There is a significant difference between the peak heights observed in GPC analyses depending on the SRT. In particular, the peak signals corresponding to low molecular weight organic compounds decreased as SRT decreased. Therefore, the amount of low molecular weight SMP may be higher at longer SRTs.

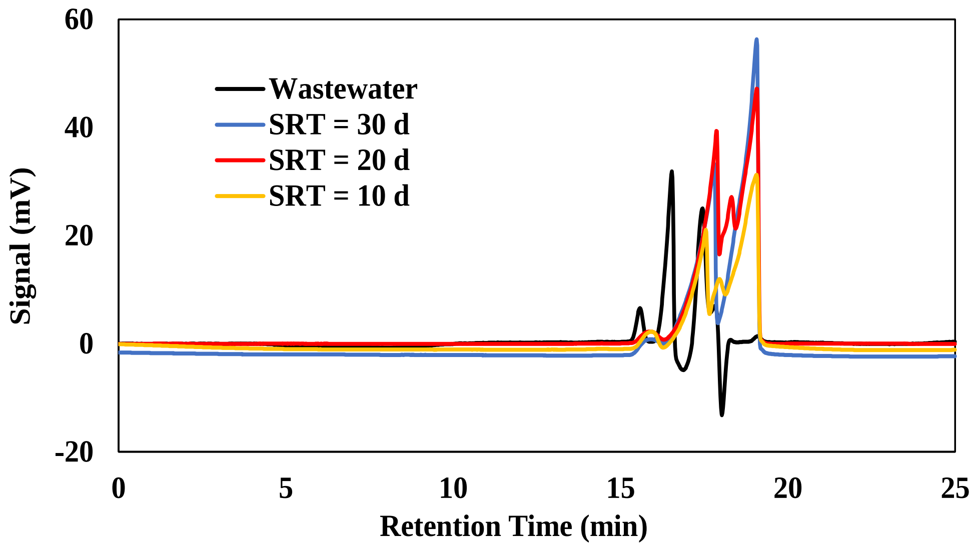



**Figure 2.5 :** The impact of SRT on the presence of organic molecules in the permeate.

### 2.3.5 Impact of SRT on observed yield and daily sludge waste in MBR

The variations of the calculated specific energy requirement and $Y_{obs}$ values concerning employed SRTs are plotted in Figure 2.6. At constant HRT, a high amount of sludge was retained in the bioreactor and low sludge yields ($Y_{obs}$) were attained at high SRTs. Conversely, a lower amount of sludge was retained which results in a high sludge amount to be further handled at shorter SRTs. In the study, $Y_{obs}$ values increased with decreasing SRT as it was calculated as 0.125, 0.192 and 0.314 mg SS/mg COD at SRTs of 30, 20, and 10 d, respectively.

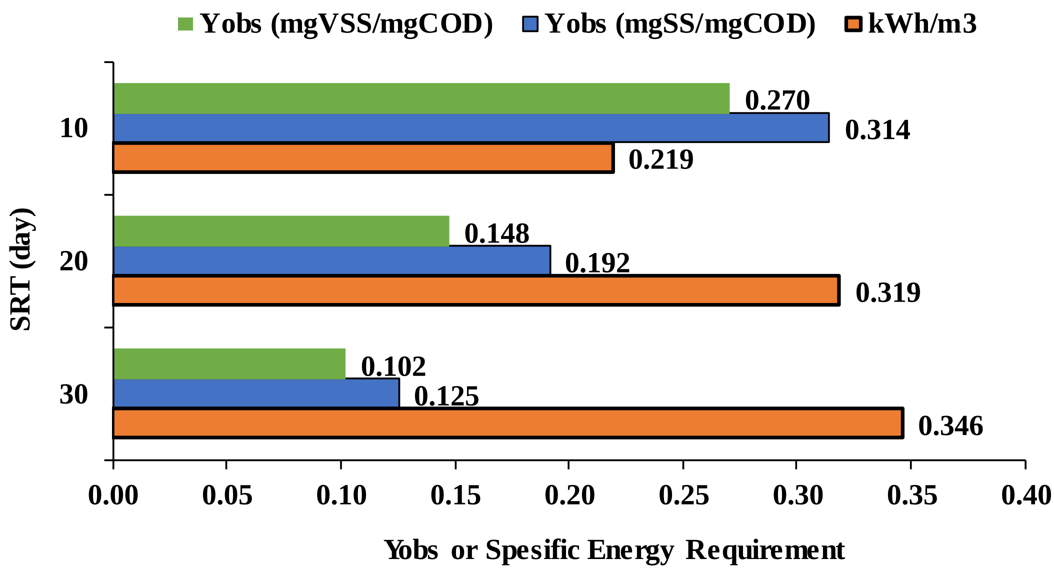



**Figure 2.6 :** The impact of SRT on the $Y_{obs}$ and specific energy requirement.

In a study investigating petrochemical wastewater treatment in aerobic MBR, $Y_{obs}$ values were reported as 0.614±0.165 g VSS/g COD, 0.521±0.0864 g VSS/g COD, and 0.441±0.0997 g VSS/g COD at SRTs of 10, 20, and 30 d (Huang et al., 2020). In a study by Castellanos et al. (2021), nutrient removal for domestic wastewater was studied in the aerobic granular sludge system and the biomass yield coefficient (Y) was reported as 0.48 g VSS/g COD, 0.61 g VSS/g COD and 0.68 g VSS/g COD at SRT of 30 d, 20 d, and 15 d (Castellanos et al., 2021).

Although the sludge amount to be handled increases at short SRTs, it may be preferable if the excess sludge is used for energy generation. As illustrated in Figure 2.6, the generated sludge amount increased almost 2.5 times as SRT decreased from 30 to 10 d. It is well known that as the SRT decreases the specific amount of methane generation during anaerobic digestion of the sludge increases significantly (Carrera et al., 2022). Bolzonella et al. reported that the specific biogas generation increased significantly from 0.07 $m^3$/kg-$VSS_{fed}$ to 0.18 $m^3$/kg-$VSS_{fed}$ as SRT decreased from 35 to 8 d (Bolzonella et al., 2005). Hence, low SRT may be preferred to generate energy from waste sludge without sacrificing the desired treatment performance.

### 2.3.6 Impact of SRT on oxygen and energy requirements in MBR

The specific energy requirement is expected to increase with SRT as a result of higher biomass concentrations in the bioreactor. Sözen et al. (2019) determined the specific energy requirement for an SRT of 4 d was 0.25 kWh/$m^3$, whereas the same for an SRT of 25 d was estimated between 0.45 and 0.52 kWh/$m^3$. It has been reported that the total energy requirement in aerobic MBR is between 0.6 and 1.2 kWh/$m^3$, of which approximately 30-40% for aeration (Martin et al., 2011). In our study, the specific aeration energy requirement for biological oxidation only was estimated as 0.22-0.35 kWh/$m^3$ in the studied SRT range. The lowest SRT of 10 d exerted the lowest energy requirement, which was 37% and 31% lower than the SRT of 30 d and 20 d, respectively (Figure 2.6). Linear relationships between SRT and both specific energy requirement ($r^2$= 0.90) and $Y_{obs}$ ($r^2$=0.97) were observed.

As SRT decreased from 30 d to 10 d, sludge generation increased around 2.5 times and the energy requirement for aeration decreased by 37%. Hence, considering the energy requirement for biological treatment, shorter SRT values can be chosen without sacrificing treatment performance, especially if excess sludge is to be used for energy generation.

The specific energy for permeation was calculated as 0.0083 kWh/m$^3$. Additional energy will also be required for course bubble air scouring if the membranes are located in a separate membrane tank. Energy requirement, in this case, will completely depend on the airflow rate, which may be decided by membrane fouling propensities. In a recent study, the aeration requirement for physical cleaning by air-scouring of a ceramic membrane used as flocculation-assisted direct filtration was reported to be 0.082 kWh/m$^3$ (Ozcan et al., 2022). Hence, total specific energy requirements including biological treatment, air scouring of the membranes and permeation at SRTs of 30, 20 and 10 d were estimated as 0.44, 0.41 and 0.31 kWh/m$^3$, respectively.

## 2.4 Conclusion

In the study, the treatment of real textile wastewater was investigated in two aerobic MBRs operated under different SRTs conditions (10, 20 and 30 d). The highest COD and color removal efficiencies were observed at SRT 20 d. The COD removal efficiency was over 90% at all operated SRTs. The highest color removal efficiency of 60% was achieved at an SRT of 20 d, which was 8% and 17% higher than the values observed at SRTs of 30 and 10 d, respectively. In addition, the highest removal efficiencies by the cake layer were observed at an SRT of 30 d. Due to the low flux, no serious fouling was observed in the MBRs however, SRF values were increased by reducing the SRT to 10 d. In conclusion, this study asserts that aerobic MBR operated in the SRT values down to 10 d, which is practically more environmentally friendly than MBR operation conventionally employing very long SRTs, is a very effective and reliable treatment option for organic carbon removal from actual composite textile effluents, while being able to provide a certain level of color removal also. As SRT decreased, the waste sludge amount increased while the energy requirement for aeration decreased. The increased sludge disposal costs due to the increased amount of waste sludge at short SRTs may be compensated by the increased methane production if the excess sludge is anaerobically digested.

# 3. PERFORMANCES OF A HIGH-RATE MEMBRANE BIOREACTOR FOR ENERGY-EFFICIENT TREATMENT OF TEXTILE WASTEWATER

## 3.1 Introduction

The rapid development of textile industry on global scale has resulted in serious water consumption as per kg of product may need 0.06-0.40 $m^3$ water consumption (Mokhtar et al., 2014). It is also a major concern for the receiving environment with the discharge of wastewater containing various chemicals, dyes reagents, and metals (Mani et al., 2019). However, biological systems such as conventional activated sludge systems (CAS) cannot provide an effective treatment due to the toxic content of textile wastewater, and other activated sludge processes related operational problems such as frequently encountered sludge bulking phenomena (T.-H. Kim et al., 2007). Membrane bioreactors (MBRs) are highly preferred due to numerious advantages containing small footprint, less sludge generation, high organic matter and nutrient removal performances and high discharge quality (Kim et al., 2019). Aerobic MBRs are generally used in textile wastewater treatment due to their higher chemical oxygen demand (COD) removal (over 90%) capacity (Samsami et al., 2020) and the readiness of the permeate to be fed for further water recovery processes, such as reverse osmosis. For example, Yang et al. (2020) investigated treatment efficiency of a real textile wastewater in different biological proceses of  CAS, MBR, and moving bed biofilm reactor (MBBR). Higher COD, color, and total solid (TS) removal efficiencies were obtained in the MBR. Sari Erkan et al. (2020) reported 93.1% COD and 87.1% color removal efficiencies in an aerobic MBR. However, MBRs require higher energy consumption mainly due to the higher oxygen demand of biomass at long sludge retention times (SRTs) (Jiang et al., 2008), and membrane filtration and cake scouring (Yap et al., 2012).

Undoubtedly, the new approaches in sustainable wastewater treatment need to be implemented in MBRs or other biological processes to increase the number of energy neutral or positive treatment plants (Hao et al., 2018). This can be achieved with a high-rate MBR systems operated at an SRT of only 0.5-2 days, providing high sludge production that can be used for further energy production through partial oxidation of the wastewater (Rocco et al., 2023). In the high-rate MBRs, not all of the COD is biologically oxidized to carbon dioxide due to low SRT and hydraulic retention time (HRT) values (Faridizad et al., 2022), only a part of the soluble organic matter (or COD) is used for intercellular storage, microbial growth and carbon oxidation (Canals et al., 2023), and the particulate COD in the wastewater may coagulate with the sludge and be rejected by the membrane. Therefore, high quality effluent discharges are obtained due to membrane rejection of particulate and colloidal organic matters together with creating a very suitable raw material source for biomethane production as particulate and colloidal organic parts in wastewater remain in the sludge by bioflocculation (Başaran et al., 2014). Although high-rate MBRs have been used for energy-efficient synthetic domestic wastewater (Başaran et al., 2014; Ng and Hermanowicz, 2005a; Sözen et al., 2014), and sewage treatment (Akanyeti et al., 2010; Faust et al., 2014), to the best of our knowledge, there is no study conducted with textile industry wastewater.

In addition, bioflocculation, which plays a major role in the recovery of organic matter from wastewater, is highly associated with extracellular polymeric substances (EPS) production (Ng and Hermanowicz, 2005a). However, quite conflicting results have been reported regarding the effect of SRT on EPS production and the carbohydrate and protein content of the produced EPS (Faust et al., 2014). Also, the most limiting disadvantage of high-rate MBR systems is the rapid formation of membrane fouling profiles (Rocco et al., 2023). However, membrane fouling is affected by operating conditions (SRT and HRT), wastewater (Isma et al., 2014) and biomass characteristics (biomass concentration, viscosity, type and concentration of microbial products) (Han et al., 2005) and therefore it should be examined separately for each wastewater and operating conditions.

The present study aims to determine and compare the performances of a high-rate MBR treating real textile industry wastewater at varying SRTs (0.5–5 d) and SRT/HRT ratios (5, 10 and 20). At each SRT, observed biomass yield ($Y_{obs}$), the amount of waste sludge, air and energy requirements were calculated. Furthermore, the effects of low SRTs and varying SRT/HRT ratios on membrane filtration performance and fouling tendency in the high-rate MBR were discussed in detail. The variations of sludge filterability properties were investigated with analyses such as specific resistance to filtration (SRF), supernatant filterability (SF), capillary suction time (CST) and viscosity. In addition, organic and inorganic membrane foulants under the different SRTs, where SRT/HRT ratio was 10, were characterized with the scanning electron microscopy (SEM) and SEM coupled with energy dispersive spectroscopy (SEM-EDS).

## 3.2 Materials and Methods

### 3.2.1 Bioreactor

A completely automated aerobic MBR (7 L active volume), previously operated under aerobic conditions at an SRT of 10 d and an HRT of 1 d for around 68 days (Yilmaz et al., 2023a), was used in the present study. A submerged ZeeWeed 500M-1M module including ultrafiltration (UF) membrane with a 0.04 μm pore diameter and 0.23 $m^2$ surface area was used for the filtration. Influent and permeate flow rates were adjusted using Seko (PR7) and Longer Pump (BT600-2 J) peristaltic pumps, respectively. Waste sludge was removed daily from the system with an additional pump. A completely automated digital control panel was used to set and monitor the operating parameters such as start-stop, pump speed adjustment and trans membrane pressure values (TMP) in the MBR system. Air flow rates were varied between 3 and 8 L/min with a compressor to adjust the dissolved oxygen (DO) concentration in the MBR at ≥ 3 mg/L (Figure B1). The reactor was kept in the room at a temperature of 25±1 °C.

### 3.2.2 Wastewater and biomass source

Wastewater was obtained from a full scale treatment plant, which serves 64 textile industries with a total flow rate of 50,000-60,000 $m^3$/day and 95% of the wastewater originates from the textile industry (Sahinkaya et al., 2019). The average total COD, color and $N\text{-}NH_4^+$ in the wastewater were 834±143 mg/L and 1037±407 Pt-Co and 51±11 mg/L, respectively (Table B1). The inoculum sludge for the high-rate MBR was obtained from a lab-scale aerobic MBR treating textile wastewater at an SRT of 10 d with a volatile suspended solid (VSS) of 2,263±326 mg/L (Yilmaz et al., 2023a).

### 3.2.3 High-rate MBR operation

The details of each operating condition tested in the system are given in Table 3.1. The MBR was operated for approximately 250 days in five main different periods with SRTs ranging from 5 to 0.5 d. HRTs were changed in the system to adjust the SRT/HRT ratios at 5, 10, and 20 in each period, except at SRTs of 1 and 0.5 d where the SRT/HRT ratio was kept constant at 10. During the study, the influent and permeate flow rates were varied between 7-140 and 5.6-126 L/d, respectively. Intermittent filtration with 5 min suction and 1 min relaxation duration was applied to reduce membrane fouling. Due to changes of HRTs, average and instantaneous fluxes varied between 1-11.4 and 1.2-13.7 LMH (liters per square meter per hour), respectively. Physical and chemical cleanings were applied when the TMP exceeded 0.2 and 0.4 bar, respectively, as described by Yurtsever et al. (2017).

**Table 3.1 :** Operating conditions tested in the high-rate MBR.

| Parameter | Unit | Periods | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | P1.1 | P1.2 | P1.3 | P2.1 | P2.2 | P2.3 | P3.1 | P3.2 | P3.3 | P4 | P5 |
| **Reactor volume** | L | | | | | | 7 | | | | | |
| **Membrane Area** | $m^2$ | | | | | 0.23 | | | | | | 0.46 |
| **SRT** | d | | 5 | | | 3 | | | 2 | | 1 | 0.5 |
| **HRT** | h | 24 | 12 | 6 | 14.4 | 7.2 | 3.6 | 9.6 | 4.8 | 2.4 | 2.4 | 1.2 |
| **SRT/HRT** | - | 5 | 10 | 20 | 5 | 10 | 20 | 5 | 10 | 20 | 10 | 10 |
| $\mathbf{Q_{wastewater}}$ | L/d | 7 | 14 | 28 | 12 | 23 | 47 | 17.5 | 35 | 70 | 70 | 140 |
| $\mathbf{Q_{daily\ waste\ sludge}}$ | L/d | | 1.4 | | | 2.3 | | | 3.5 | | 7 | 14 |
| $\mathbf{Q_{permeate}}$ | L/d | 5.6 | 12.6 | 26.6 | 9.3 | 21 | 44.3 | 14 | 31.5 | 66.5 | 63 | 126 |
| **Average Flux** | LMH | 1 | 2.3 | 4.8 | 1.7 | 3.8 | 8 | 2.5 | 5.7 | 12 | 11.4 | 11.4 |
| **Inst. Flux** | LMH | 1.2 | 2.7 | 5.8 | 2 | 4.6 | 9.6 | 3 | 6.85 | 14.5 | 13.7 | 13.7 |

### 3.2.4 Analytical methods

Analyzes were carried out on samples at least three times a week. COD, suspended solids (SS), VSS, and alkalinity were performed as described in Standard Methods (APHA, 2005). The color was measured as Pt-Co at 420 nm in a spectrophotometer (HACH, DR/5000). Dionex ICS-5000+ (Thermo Fisher Scientific, USA) ion chromatography equipped with an AS9-HC column was used to measure nitrate and nitrite concentrations. HACH HQ40d and WTW Multi 3420 multimeter were used for DO/Oxidation-reduction potential (ORP) and pH/conductivity measurements, respectively. Total nitrogen (TN), ammonium nitrogen and total phosphorus (TP) concentrations were monitored with HACH kits. In order to test the sludge filterability, SRF and SF analyzes described by Dereli et al. (2014) were performed. Type 304M Capillary Suction Timer (Triton Electronics Ltd., England) and DV-E viscometer (Brookfield Ltd., Canada) were used for CST and viscosity (with Newtonian model), respectively. SMP and EPS samples were extracted as described by Judd and Judd (2006) and Rahman et al. (2017), respectively and carbohydrate (Dubois et al., 1956) and protein (Bradford, 1976) analyzes was applied. Agilent 1260 Infinity was used in gel permeation chromatography (GPC) analyses. SEM-EDX samples was prepared in a dead-end filtration device with a pore size of 0.22 μm as described by Yilmaz et al. (2023b), and the analyses were performed using the GeminiSEM 500 device at Osmangazi University Central Research Laboratory Application and Research Center (ARUM, Eskişehir, Turkey), respectively.

Observed sludge yield ($Y_{obs}$) and energy requirement for aeration in each SRT and SRT/HRT ratio were calculated as explained by Yilmaz et al. (2023a). Statistical analysis of data were performed using ANOVA as described in our previous study (Yilmaz et al., 2023a).

## 3.3 Results and Discussion

### 3.3.1 COD and color removal in the high-rate MBR

The removal efficiencies of the system operated for 250 days are given according to periods in Table 3.2. The pH and conductivity in wastewater were 8±0.5 and 5588±1084 μS/cm, respectively, and remained at similar values in permeate throughout the study (Figure B2).

COD in permeate averaged around 100 mg/L with the highest value of 227 mg/L, observed at SRT of 0.5 d (Figure 3.1). The total COD removal performance ranged from 86 to 92% for all SRTs between 5 and 2 d. However, a slight reduction in COD removal performance was observed when the SRT was decresed to less than 2 d ($p < 0.05$). Further decreasing the SRT to 1 and 0.5 d resulted in decreasing COD removal efficiencies to 82 and 77%, respectively.

**Table 3.2 :** Wastewater characterization and removal efficiencies at different periods.

| Parameter | | Unit | Period No | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | P1.1 | P1.2 | P1.3 | P2.1 | P2.2 | P2.3 | P3.1 | P3.2 | P3.3 | P4 | P5 |
| Operating Conditions | SRT | d | | **5** | | | **3** | | | **2** | | **1** | **0.5** |
| | SRT/HRT | | **5** | **10** | **20** | **5** | **10** | **20** | **5** | **10** | **20** | **10** | **10** |
| COD | Wastewater | mg/L | 746±173 | 906±123 | 888±127 | 684±148 | 776±49 | 781±68 | 845±191 | 943±141 | 872±96 | 838±79 | 842±68 |
| | Supernatant | mg/L | 130±54 | 180±49 | 270±44 | 149±19 | 114±26 | 196±20 | 182±31 | 204±38 | 624±29 | 636±74 | 1094±191 |
| | Permeate | mg/L | 81±17 | 75±16 | 82±18 | 84±7 | 61±14 | 89±17 | 101±19 | 71±16 | 120±41 | 148±25 | 197±21 |
| | Total Removal Efficiency | % | **89** | **92** | **91** | **88** | **92** | **89** | **88** | **92** | **86** | **82** | **77** |
| Color | Wastewater | Pt-Co | 879±304 | 952±301 | 941±128 | 828±78 | 909±143 | 774±98 | 950±292 | 1515±313 | 1735±746 | 1105±167 | 1848±580 |
| | Supernatant | Pt-Co | 755±126 | 902±204 | 1463±321 | 1081±266 | 703±95 | 999±206 | 900±174 | 848±127 | 2497±445 | 2062±479 | 4508±889 |
| | Permeate | Pt-Co | 530±22 | 496±83 | 570±143 | 748±25 | 462±91 | 570±98 | 567±40 | 448±25 | 804±258 | 661±150 | 893±348 |
| | Total Removal Efficiency | % | **40** | **48** | **39** | **10** | **49** | **26** | **40** | **70** | **54** | **40** | **52** |
| Alkalinity | Wastewater | $mgCaCO_3/L$ | 1037±50 | 953±109 | 954±57 | 828±39 | 778±36 | 808±35 | 798±64 | 814±56 | 919±36 | 950±23 | 797±72 |
| | Permeate | mg/L | 655±69 | 521±68 | 600±74 | 491±26 | 505±61 | 589±66 | 527±90 | 700±41 | 899±53 | 906±37 | 775±30 |
| | Decrease | % | **37** | **45** | **37** | **41** | **35** | **27** | **34** | **14** | **2** | **5** | **3** |
| $N\text{-}NH_4^+$ | Wastewater | mg/L | 0±0 | 55±14 | 43±2 | 49±3 | 38±14 | 59±13 | 47±9 | 48±3 | 59±0.1 | 61±1 | 25±3 |
| | Permeate | mg/L | 0±0 | 0.4±0.8 | 1±1.4 | 0.1±0.2 | 5±7 | 22±6 | 9±10 | 18±9 | 50±9 | 51±1 | 19±3 |
| | Total Removal Efficiency | % | **-** | **99** | **98** | **99.8** | **87** | **62** | **81** | **63** | **15** | **16** | **23** |

In the literature, SRT in textile wastewater treatment using MBRs is mainly close to 25 d to reach COD removal efficiencies over 90% (Jegatheesan et al., 2016). Kapdan and Ozturk (2005) investigated the treatment of synthetic dyestuff-containing wastewater in an SBR at different SRTs (12-30 d), and reported that the removal performances were not affected by increasing SRT from 15 to 30 d. However, color and COD removal efficiencies decreased at SRT of 12 d. High-rate MBRs have been generally tested for domestic wastewater treatment. Ng and Hermanowicz (2005a) investigated synthetic wastewater (400±15 mg COD/L) treatment at relatively short SRTs (0.25 – 5 d) and HRTs (3 – 6 h). COD removal performances were between 97.3 and 98.4% for all the applied SRTs. Başaran et al. (2014) examined synthetic wastewater (250 mg/L acetate-based COD) in a laboratory scale side-stream aerobic MBR. SRTs in the system were varied between 2 and 0.5 d at a constant HRT of 1.0 h, and reported that as the SRT decreased, the COD in effluent increased, however it did not exceed 20 mg/L for all the SRTs.

In the present study, as the SRT/HRTs ratio increased at a constant SRT (i.e. as HRT was decreased), organic matter accumulation in the MBR supernatant increased, which was more evident even at decreased SRTs (except for SRT of 3 d) (Figure 3.1). The COD accumulation in the supernatant with respect to total COD in the wastewater ranged from 15 and 30% up to the Period 3.2 (at SRT of 2 d and HRT of 4.8 h). A sudden increase in COD accumulation was observed in the system with HRT reduction to 2.4 and 1.2 h, as it reached to 72, 76 and 130% at SRT of 2 (P3.3), 1 (P4) and 0.5 d (P5). Hence, the COD in the supernatant exceeded that in the feed, which should be due to accumulation of slowly biodegradable COD and soluble microbial products within the MBR as a function of SRT/HRT ratio (Hocaoglu and Orhon, 2010). The COD in the permeate was between 8 and 12% of the feed COD between P1.1 and P3.2, while it reached 14, 18 and 23% at P3.3, P4 and P5 coinciding with the increased COD accumulation in the MBR supernatant.

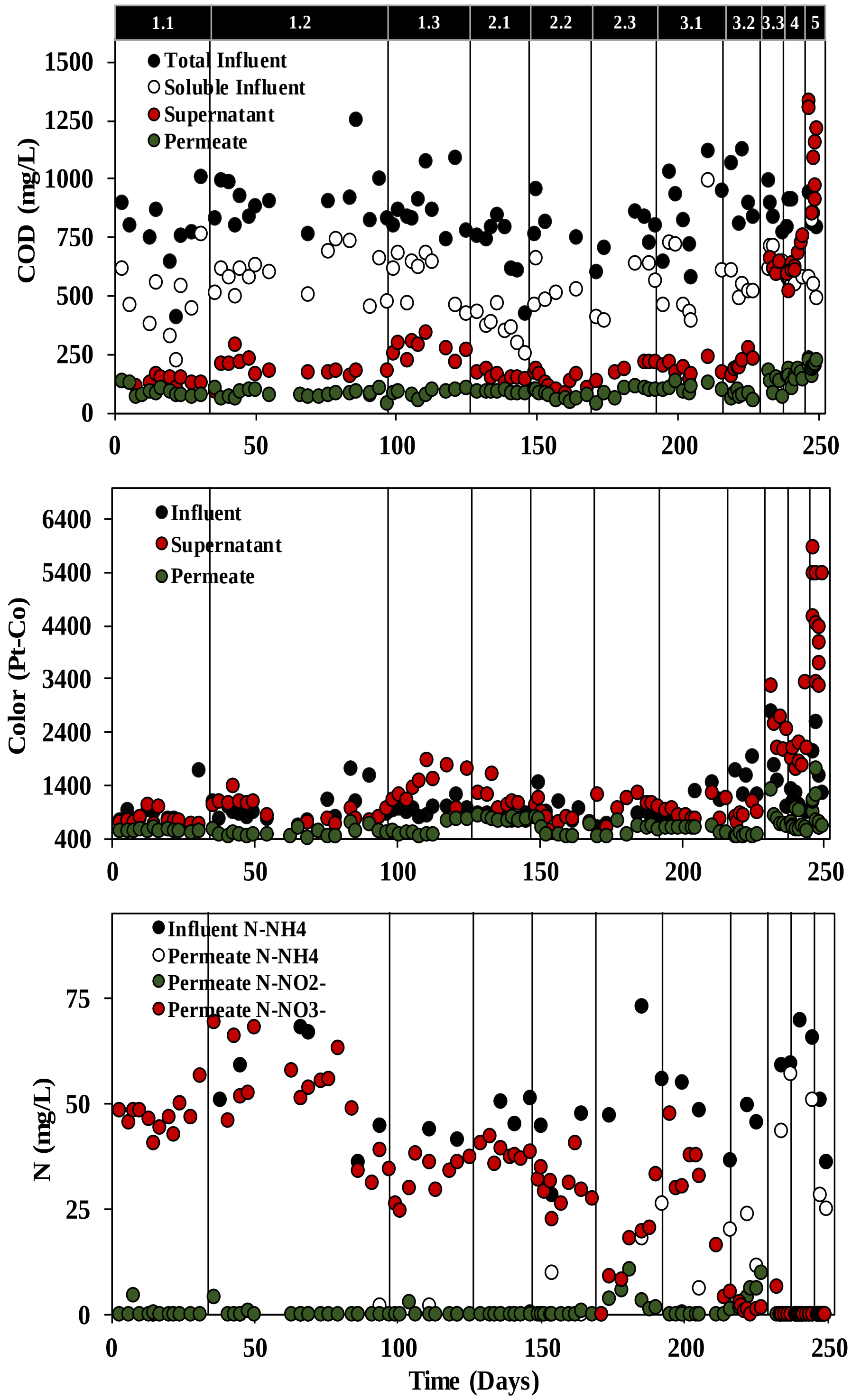



**Figure 3.1 :** COD, Color, N-$NH_4^+$, N-$NO_3^-$ and N-$NO_2^-$ concentrations in the high-rate MBR.

It is well known that the effective membrane filtration pore size decreases significantly as a results of cake or gel layer formation on the membrane, which increases membrane rejection performance and causes COD accumulation within MBR (Hocaoglu and

Orhon, 2010). This accumulation may be useful in terms of energy recovery as a part of organic may not be oxidized and can be recovered for further energy recovery together with sludge. Organic matters accumulate within MBRs as a function of SRT/HRT value (Hocaoglu and Orhon, 2010), and the accumulation-corrected residual soluble COD in the bulk ($S_{RBE}$), which was calculated according to Equation 3.1 (Hocaoglu and Orhon, 2010), as a function of SRT is illustrated in Figure 3.2. In a conventional activated sludge process, soluble organic matter retains in the bioreactor for the duration of HRT, whereas, in an MBR a part of soluble organic matter may be kept in the bioreactor for the period of SRT due to reduced membrane pore size (Hocaoglu and Orhon, 2010). Therefore, increased COD accumulation at SRTs of 1 and 0.5 d can be attributed to accumulation of the slowly biodegradable substrate and SMPs in the bulk due to reduced retention time.

$$\mathrm{S_{RBE}} = (S_{\mathrm{RB}})/(\frac{\mathrm{SRT}}{\mathrm{HRT}}) \quad (3.1)$$

Where $S_{RB}$ is the residual soluble bulk COD (Hocaoglu and Orhon, 2010).

$$\%\mathrm{S_{RBE}} = \frac{\mathrm{S_{RBE}}}{\mathrm{S_0}}.100 \quad (3.2)$$

Where $S_0$ is the total feed COD.

Sutton et al. (2011) reported 80% COD accumulation in the system due to insufficient time provided for the processes such as hydrolysis, bioflocculation and adsorption for all COD components at low SRTs. In another study investigating sewage treatment in an MBR at SRTs 1, 0.5 and 0.25 d and HRT of 1.2 h, approximately 54, 41 and 27% of the total COD were mineralized in the system, while about 13, 16 and 19% of the total COD remained in permeate in respective order (Akanyeti et al., 2010). Consequently, as SRT decreased, lower total removal was obtained, but the amount of COD remaining in the sludge increased. Therefore, the most economical, perhaps carbon neutral or carbon positive, system would be possible at the minimum SRT and HRT values, where the highest possible amount of COD accumulates as sludge or colloidal COD in the system without being oxidized and acceptable permeate COD values are provided.

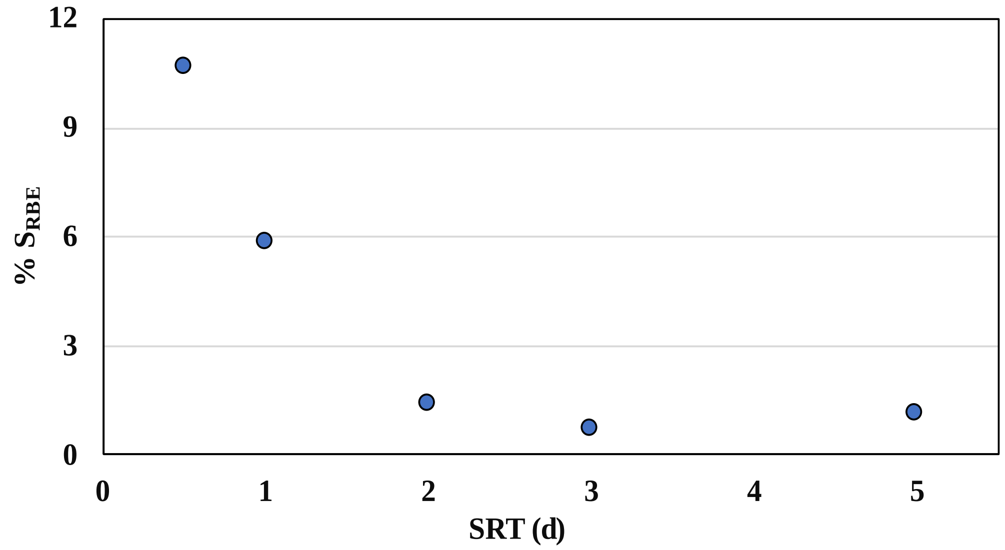



**Figure 3.2 :** Variations of accumulation corrected COD in the high-rate MBR.

No correlation was observed between color removal efficiency and SRT or SRT/HRT ratios (Figure 3.1 and Table 3.2). Color removal efficiencies ranged from 10 to 70% ($p < 0.05$), and the highest removal performance was obtained at SRT of 2 d and HRT of 4.8 h. Similar accumulation trends were observed in color, except for the SRT of 3 d, and the accumulation rates of color in the system increased as SRT and HRT decreased. Although color significantly accumulated in the system at the lowest SRTs of 1 and 0.5 d, the color in permeate was still much lower compared to those in supernatant (Figure 3.1). For example, color in wastewater, supernatant and permeate averaged 1848, 4508 and 893 Pt-Co, respectively at SRT 0.5 d. Although color in the wastewater mostly soluble and pass through the 0.45 µm pore-sized filter, significant amount of color rejection during the filtration was observed, which should be due to decreased pore size of the membrane resulting from cake or gel layer development.

It has already been reported that aerobic systems are not effective for the azo dye removal. Generally, biological azo dye removal mechanisms include the destruction of bonds in the anaerobic condition to aromatic amines and their further degradation in the aerobic condition (Sarayu and Sandhya, 2012). Işık and Sponza (2006) investigated acid dyeing wastewater treatment in a sequential anaerobic UASB/aerobic CSTR system, and obtained 87–80% color removal efficiency. After a biological treatment, an expensive tertiary treatment stages such as membrane filtration, adsorption with activated carbon, ozonation, coagulation-flocculation, and photocatalytic degradation may be needed for further decolorization (Vilaseca et al., 2010). For example, De Jager et al. (2014) achieved to reduce 2070 ADMI units color in wastewater to 12 ADMI with RO process after biological treatment including anaerobic, aerobic, and anoxic processes.

### 3.3.2 Nutrient removal in the high-rate MBR

The variation of the nitrogen in the MBR during the study is given in Figure 3.1. The average TN in the wastewater was 60±11 mg/L, and average nitrogen consumed ranged from 11 to 24 mg/L in the periods, excluding P1.2 and P2.3 ($p < 0.05$). The ammonium oxidation efficiency was above 98% at the SRT of 5 d. However, ammonium oxidation efficiency decreased with decreasing SRT and HRT to 3 d and 7.2 h, respectively ($p < 0.05$). Further decreasing the SRT to 2 d and HRT to 4.8 h in the system ceased nitrification process, and nitrite and/or nitrate production was completely eliminated. The average nitrate in permeate was between 34 and 50 mg N/L in P1 and P2.1, corresponding to ≥98% ammonium oxidation performance. With the decrease of SRT and HRT, the average nitrate in permeate dropped down to 1 mg N/L. No nitrite accumulation was occurred at SRTs of 1 d and 0.5 d, and it averaged less than 5 mg N/L in all periods.

SRT is an important parameter on the nitrification efficiency because of the slow growth rate of autotrophic bacteria (Duan et al., 2013). Duan et al. (2009) reported more than 87% ammonia removal at SRT of 3, 5, and 10 d (at HRT of 6 h). Ng and Hermanowicz (2005a) reported that nitrification performance ceased at an SRT <2.5 d in an MBR treating synthetic domestic wastewater. Therefore, the data obtained in our study are consistent with the literature, and in general, the nitrification process stopped completely when the SRT was decreased to ≤2 d.

It is also known that 7.14 mg of alkalinity is consumed per mg of ammonium nitrogen during nitrification (Srivastava and Kazmi, 2020). Decreasing alkalinity consumption in the system can be associated with the deterioration in the nitrification performance. Alkalinity consumption in the system was between 27 and 45% when the nitrification process proceeds at high performances. Further decreasing SRTs led to deterioration in the nitrification performance and decreasing alkalinity consumption down to 2% (Figure B2 and Table 3.2).

The TP consumption in the process ranged between 45 and 82%, and the highest consumption occurred as 80% at the SRT of 1 d. However, by reducing the SRT to 0.5

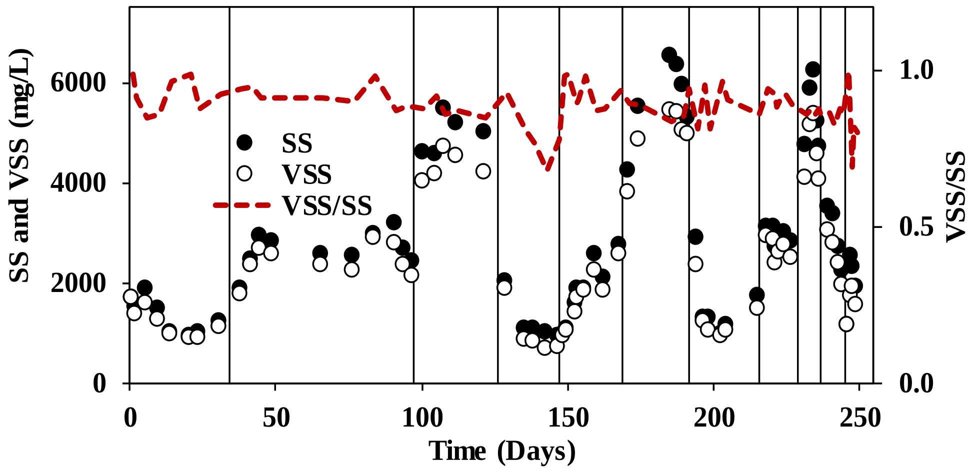
d, TP removal decreased to 64% again, which may be due to the lower biomass production associated with the lower COD consumption as discussed previously.

### 3.3.3 Waste sludge generation in the high-rate MBR

Biomass amount in the bioreactor is a function of SRT for the same HRT value, and sludge concentration in the bioreactor increases with increasing SRT (Equation 3.3).

$$X = \left(\frac{SRT}{HRT}\right) \cdot \frac{(S_0 - S_e) \cdot Y}{(1 + k_d \cdot SRT)} \tag{3.3}$$

(So: Influent substrate concentrations, mg/L, Se: Effluent substrate concentrations, mg/L); Y: Yield coefficient, g VSS/g COD; $k_d$: decay rate, 1/d).



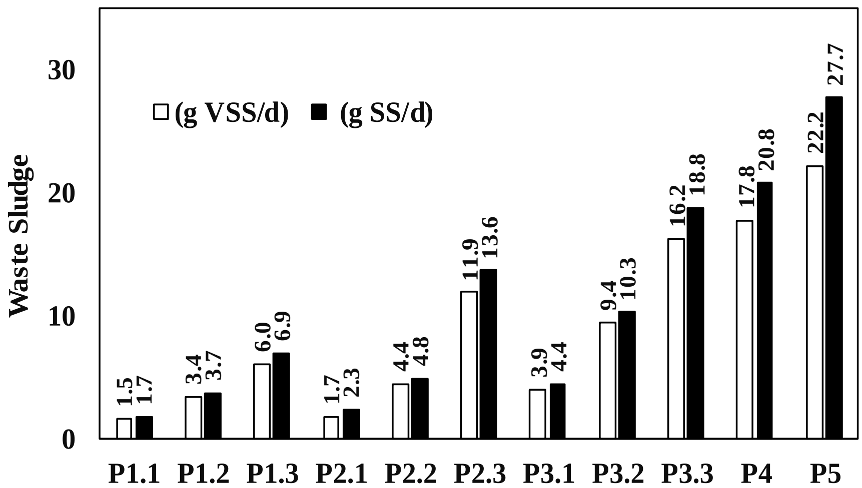




**Figure 3.3 :** SS, VSS concentrations (upper figure) and waste sludge amount in the high-rate MBR.

As the SRT/HRT ratio increased (as HRT decreased) at each SRT, the SS and VSS concentrations increased in the system (Figure 3.3). For example, VSS in the system was 1145±216, 2682±208 and 4640±598 mg/L for SRT/HRT of 5, 10 and 20, respectively, at SRT of 2 d. In addition, SS and VSS were close to each other at the same SRT/HRT ratios for each SRT. VSS concentrations was 2484±257, 1937±411, 2682±208 and 2538±481 mg/L at SRT of 5, 3, 2 and 1 respectively, for SRT/HRT ratio of 10. However, when SRT was reduced to 0.5 d, VSS concentration in MBR decreased to 1585±327 mg/L at SRT/HRT ratio of 10, which should be due to reduced COD removal efficiency in the system. Although, average VSS/SS ratio was 0.9 at SRT/HRT of 10 when SRT was 5, 3, 2 and 1 d, respectively, it decreased to 0.8 at SRT of 0.5 d, which may be due to variations in the feed wastewater. The amount of waste sludge produced for each period is given in Figure 3.3. The amount of waste sludge increased as the SRT decreased.

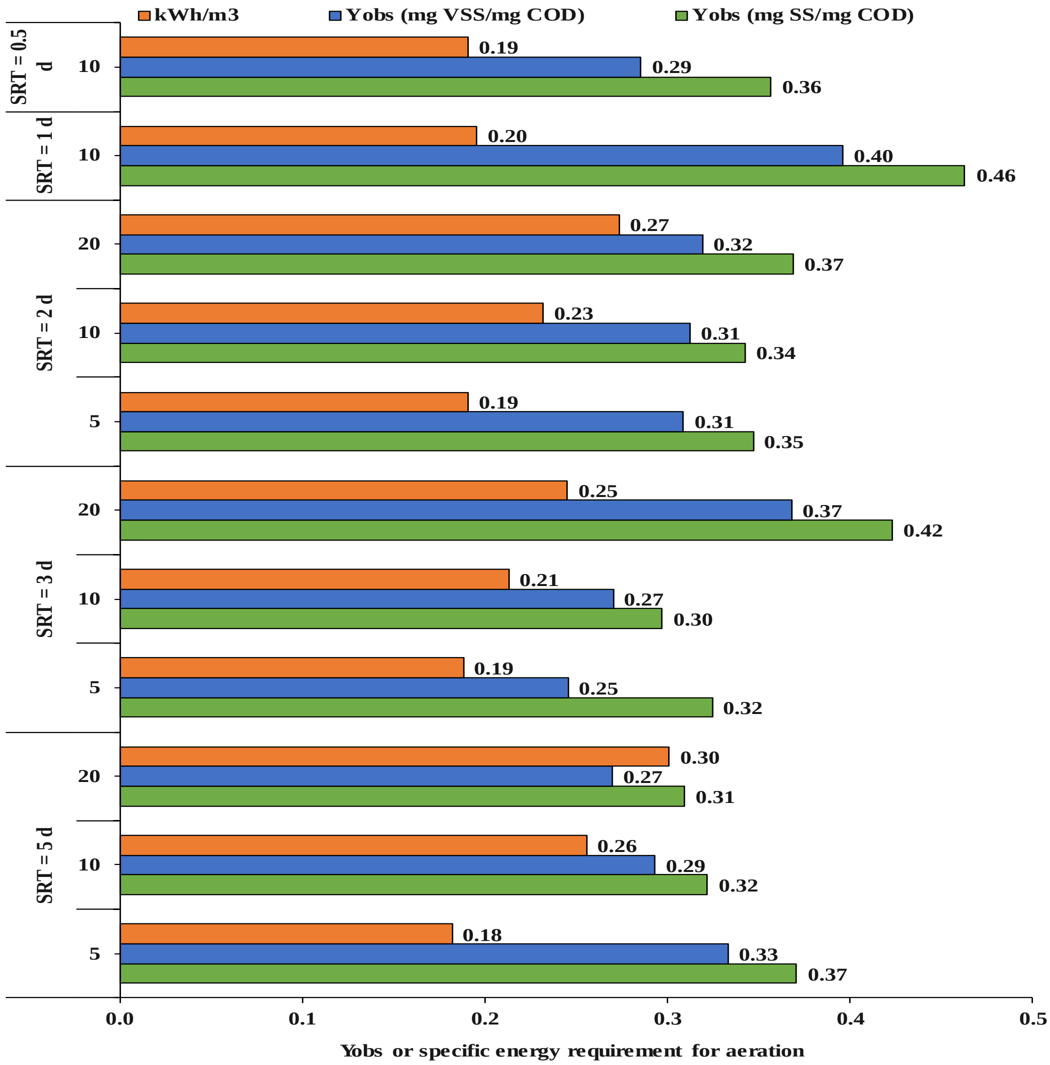



**Figure 3.4 :** The impact of SRT on the $Y_{obs}$ and specific energy requirement in the MBR.

$Y_{obs}$ values ranged from 0.3 to 0.42 mg SS/mg biodegraded COD for all SRT/HRT ratios for SRT of 5, 3 and 2 d. While the highest $Y_{obs}$ value was 0.46 mg SS/mg COD at SRT of 1 d, and it was around 0.36 SS/mg biodegraded COD at SRT of 0.5 d. A clear increase in $Y_{obs}$ was observed with decreasing SRT at SRT/HRT ratio of 10, and it was 0.32, 0.30, 0.34 and 0.46 mg SS/mg biodegraded COD at SRT of 5, 3, 2 and 1 d, respectively (Figure 3.4). In our previous study, $Y_{obs}$ increased from 0.125 to 0.314 mg SS/mg COD by reducing SRT from 30 to 10 d (Yilmaz et al., 2023a). In another study, the observed sludge yield values were 0.38, 0.51 and 0.68 g COD/g COD at SRTs of 1, 0.5 and 0.25 d, respectively (Akanyeti et al., 2010).

### 3.3.4 Membran fouling and filtration performances

#### 3.3.4.1 TMP and flux

The variability of TMP and flux under various SRTs and HRTs throughout the study are given in Figure 3.5. The instantaneous flux in the system were 1.2 (P1.1), 2.7 (P1.2) and 5.8 LMH (P1.3), respectively at SRT of 5 d. The highest TMPs were <5, <20, and 69 mbar at 1.2, 2.7, and 5.8 LMH, respectively, and no cleaning was performed due to low TMP values.

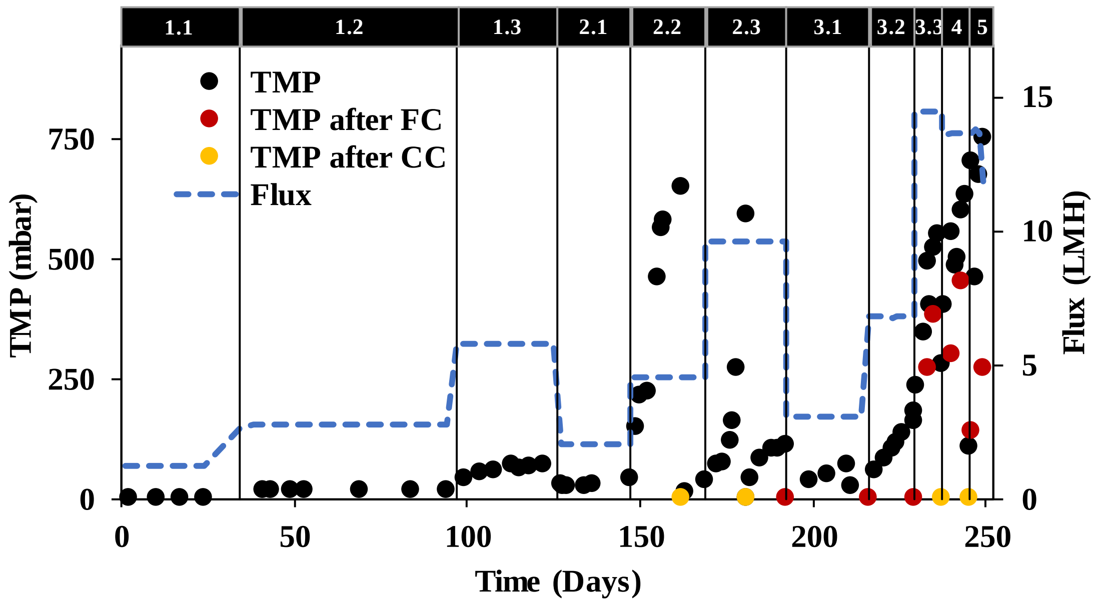



**Figure 3.5 :** The TMP and flux variations in the high-rate MBR.

With the decreasing of the SRT to 3 days, the instantaneous flux changes were as 2 (P2.1), 4.6 (P2.2) and 9.6 LMH (P3.3), respectively. In the P2.1, the average TMP was 27±5.6 mbar, and showed a similar trend with the SRT of 5 d. However, as the flux increased to 4.6 and 9.6 LMH in P2.2 and P2.3, respectively, TMP reached approximately 600 mbar, and chemical cleaning was performed in both periods.

The instantaneous fluxes were 3 (P3.1), 6.85 (P3.2), and 14.5 LMH (P3.3) at SRT of 2 d. When flux was 3 LMH, TMP showed a similar trend to that observed at SRT 5 d and the highest TMP was 70 mbar. As the flux increased to 6.8 LMH, the highest TMP increased to 180 mbar. However, membrane cleaning was not required at this period. Then, the flux was increased to 14.5 LMH, leading to a rise of TMP to 500 mbar on day 4$^{th}$. To reduce fouling, the airflow rate was first increased from 5 to 8 L/min, which was useless, and then backwashing with 30 LMH and physical cleaning resulted in TMP drop to 270 mbar. However, after 2 days, the TMP increased back to 520 mbar again and the previous procedure was repeated until the end of the period.

The instantaneous flux was 13.7 LMH at SRT of 1 d, and TMP increased up to 630 mbar despite physical cleaning every four days.

At SRT of 0.5 d, a second membrane module was added to system, due to required frequent membrane cleaning at SRT 1 d, and the flux was adjusted 13.7 LMH. However, TMP quickly rose up to 750 mbar in the system, and regular physical cleaning was required. Although the flux at SRTs 0.5 and 1 d were the same, a faster fouling profile occurred at the SRT of 0.5 d.

Membrane fouling may increase at high SS concentrations in the bioreactor due to cake deposition. Furthermore, fouling may also be related to the COD in the supernatant, and colloidal matter in the range of 0.01–1.0 μm (Tchobanoglus et al., 2003) and flocks (Jinsong et al., 2006). While SS concentration in the system accelerates the fouling profiles, also sludge characteristics, viscosity and SMP-EPS values can have significant effects (Han et al., 2005). Considering the SRTs of 5 (P1) and 3 (P2) when the COD and color accumulations in the bioreactor were relatively lower, the highest SS concentrations were reached at SRT/HRT ratio of 20 for both SRTs, and faster fouling profiles were formed compared to the lower ratios (corresponding to lower SS concentrations) at the same SRT. A long-term filtration process can be applied without a sudden increase in TMP due to low organic matter at longer SRT and HRT values (Isma et al., 2014). In our study, higher TMPs and rapid fouling profiles were observed during periods when SRT and HRT were quite low. Especially with the decrease of SRT to 2 d and below, high amounts of COD and color accumulation in the system may be one of the possible reasons for observing faster fouling. Also, the high membrane fouling rates are thought to be due to the high SMP concentrations and low EPS concentrations produced at short SRTs (Jiang et al., 2008). As the SRT/HRT ratio increased for each SRT value, the SMP in the bioreactor increased while the EPS decreased (as detailed in the following headings). While the fastest membrane fouling profiles were observed especially at SRT/HRT ratio of 20, it was considerably slower at SRT/HRT of 5.

#### 3.3.4.2 Variations of SRF, SF, CST, viscosity and SVI

Variations of CST, SVI, SRF, SF and viscosity in the system under different SRTs and SRT/HRT combinations throughout the study are given in Table B2. In general, as the SRT decreased, the SRF increased, while the SF decreased in the system. In particular, the highest SRF were observed as $1.3\times10^{15}\pm1.7\times10^{14}$, $3.0\times10^{15}\pm5.2\times10^{14}$ and $1.15\times10^{16}\pm4.9\times10^{15}$ mg/kg at the SRT of 2 (only at SRT/HRT ratio of 20), 1 and 0.5 d, respectively, when COD and dye accumulated in the supernatant of MBR leading to faster TMP increase, while SF values decreased to 0.3 mL/min. Generally, higher SRF values may be observed at higher suspended solids, EPS and dispersed bacteria in MBRs (Pontoni et al., 2015).

CST and viscosity are closely related to SS and colloidal matters, which also play significant roles in membrane fouling (Laera et al., 2009). The highest CST and viscosity values were obtained at SRT/HRT ratios of 20, while the lowest values were obtained at the SRT/HRT ratio of 5. The viscosity values were generally ranged from 2 to 3 cP at SRT/HRT ratios of 5 and 10 in all SRTs. SVI was generally not correlated with SRT and SRT/HRT ratios in any periods (Figure B3 and Table B2).

#### 3.3.4.3 Variations of SMP and EPS

SMP-EPS and their carbohydrate and protein contents are presented in Figure 3.6. The SMP in the supernatant increased especially at SRTs $\leq$ 2 d. Furthermore, the SMP in supernatant clearly increased as the SRT/HRT ratio increased. For example, the SMP in supernatant was 35, 45 and 79 mg COD/L at SRT/HRT ratios of 5, 10 and 20, respectively, during the SRT of 2 d. The increase in the SMP concentrations at an increased SRT/HRT ratio is due to the higher accumulation rate of slowly biodegradable fraction of SMP. The highest SMP concentration was observed at the shortest SRT studied, i.e. 0.5 d, which was over 100 mg COD/L. Membrane was an effective barrier and rejected high amount of SMP, which polished the permeate generated. However, as it was discussed before, this caused accumulation of SMPs in the supernatant depending on the SRT/HRT ratio, resulting in faster fouling of the membrane.

The average SMP in the permeate ranged from 13 to 24 mg COD/L, and the highest SMP concentration in permeate was observed at SRT of 0.5 d. Especially in the last periods (P3.3, P4 and P5), the rejection of SMP in the supernatant remained close to 80%, and the carbohydrate part of SMP in the permeate was relatively higher compared to the protein content.

While higher SMP production occurs at lower SRTs, especially the utilization-associated byproducts (UAP) portion of SMP with high biodegradability potential and hydrophilic characteristics increases (Ni et al., 2011). The higher SMP concentrations were observed as SRT decreased, and SMP in the MBR bulk accumulated especially at SRT 1 and 0.5 d. This should be due to the reduced biodegradation potential of the organics accumulated in the bulk at reduced SRTs.

There are conflicting results with EPS production at low SRTs in the literature as some have reported increased EPS production at low SRTs (Malamis and Andreadakis, 2009), while some have reported the opposite (Ng and Hermanowicz, 2005a). In this study, a significant increase in total EPS production was observed especially at SRTs of 1 and 0.5 d. EPS concentrations were 53±30, 60±9, 58±1, 80±3 and 95±6 mg COD/g VSS for SRT of 5, 3, 2, 1 and 0.5 d, respectively, at the SRT/HRT ratio of 10. Although total EPS produced increased as the SRT decreased, it decreased as the SRT/HRT ratio increased in each SRT.

No significant changes were observed in loosely (LB-EPS) and tightly bounded (TB-EPS) contents of EPS, as they were 43±3 and 57±3% for P1.1 and P1.2, and averaged 30±4 and 70±4% in the following periods, in respective order. Also, very close values were observed in our previous study at SRTs of 30, 20, and 10 d (36-32% LB-EPS and 64-68% TB-EPS) (Yilmaz et al., 2023a). SRT clearly had no effect on the content of EPS. The carbohydrate contents of TB-EPS and LB-EPSs varied between 34–65% and 43-75%, respectively, while protein contents varied between 35-66 and 25-57%.

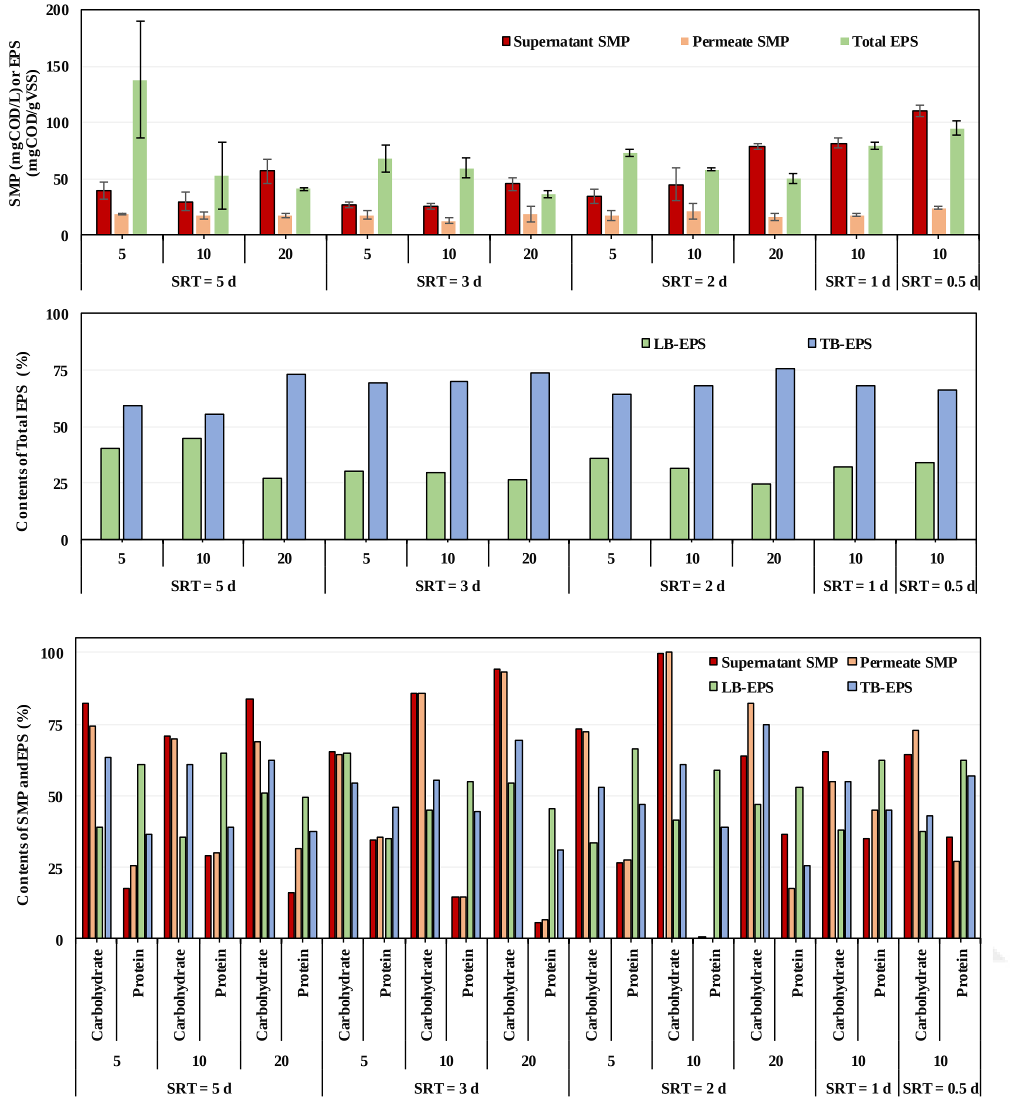



**Figure 3.6:** Variations of SMP and EPS in the MBR.

### 3.3.4.4 Molecular weight of dissolved organic molecules

Figure B4 represent the GPC results in the supernatant and permeate for SRT 5, 3, and 2 d at SRT/HRT ratio of 10. Four main peaks were detected corresponding to 24, 5.8, 1, and 0.3 kDa in all samples. The peaks obtained in the supernatant and permeate were quite close, and no change was observed despite very low SRTs. Similarly, in our previous study examining the real textile wastewater treatment performance in an MBR at SRT 30, 20, and 10 d, the supernatant and permeate peaks were quite similar, and four peaks varying between 20-22, 3 - 3.7, 1-1.4, and 0.47-0.57 kDa was detected (Yilmaz et al., 2023a).

### 3.3.4.5 SEM-EDS analysis of the foulant layer

The SEM images and EDS result of the clean and cake-deposited membrane surfaces for the SRTs 5, 3 and 2 d are given in Figure B5 and B6, respectively. The predominant C and O peaks were identified in the cake layer at SRT of 5 d while N was also predominant at SRTs of 3 and 2 d. Although elements such as Al, Si and Fe have an important contribution on the creation of membrane cake layer (Pendashteh et al., 2011), they were detected in all SRTs below 2%.

### 3.3.5 Impact of SRT on energy requirements for aeration

Although MBRs are preferred worldwide to achieve better effluent quality (Verrecht et al., 2010) which may be due to higher biomass concentrations at longer SRTs (Han et al., 2005), other approaches are currently being explored to prioritize energy minimization or energy neutral plants. One of these approaches is to convert organic materials or biosolids in wastewater into methane (Ng and Hermanowicz, 2005a) instead of oxidizing them to carbon dioxide (Akanyeti et al., 2010), thus reducing energy cost for aeration and carbon footprint as well as energy recovery (Ng and Hermanowicz, 2005a). In MBRs operated at low SRTs, although particulate and colloidal organics are separated by membrane, limited removal of soluble biodegradable COD may be observed. The limited oxidizing capacity of the process helps saving energy cost and keeping the calorific value of biomass high to be used for energy recovery (Orhon et al., 2016).

The variations of energy requirement for aeration under different SRT and SRT/HRT ratios are provided in Figure 3.4. As SRT decreased, there was a relative decrease in the specific energy requirement for aeration, and it was between 0.2-0.28 kWh/$m^3$ for 5, 3 and 2 d at SRT/HRT ratio of 10, while it was around 0.19 kWh/$m^3$ at SRTs of 1 and 0.5 d. In particular, as the SRT/HRT ratio increased for each SRT value, a clear increase was observed in specific energy requirement. For example, specific aeration energy requirements were 0.19, 0.23 and 0.31 kWh/$m^3$ for SRT/HRT ratio of 5, 10 and 20 at SRT of 2 d, respectively. In our previous study, the specific energy requirement for aeration was calculated as between 0.35, 0.32 and 0.22 kWh/$m^3$ at SRTs of 30, 20 and 10 d, respectively (Yilmaz et al., 2023a). In this study, lower energy requirement for aeration was observed at as SRT decreased.

### 3.4 Conclusion

It can be possible to draw the following conclusions;

- High COD removal efficiency (77% removal) was obtained even at an SRT of 0.5 d and HRT of 1.2 h
- Nitrification completely ceased at SRTs ≤ 2 d.
- Membrane fouling rates increased at short SRTs, which may be due to SMP and COD accumulation in the MBR.
- $Y_{obs}$ increased from 0.3 to 0.46 mg SS/mg COD by reducing SRT from 5 to 1 d (at SRT/HRT ratio of 10).
- Specific aeration energy requirement decreased to 0.19 kWh/m$^3$ (0.3 kWh/kg-$COD_{removed}$) at SRT of 0.5 d.
- Further pilot-scale studies are needed.

# 4. IMPACT OF AERATION ON/OFF DURATIONS ON THE PERFORMANCES OF AN INTERMITTENTLY-AERATED MBR TREATING REAL TEXTILE WASTEWATER

## 4.1 Introduction

Textile industry produces 50-400 L of wastewater per kg product and it causes many serious environmental problems in the receiving environment due to its toxic content (Cinperi et al., 2019). Textile industry wastewater contains high levels of organic matter, color, solids, dyestuffs, metals, and nitrogen (Al-Mamun et al., 2019). The nitrogen concentration of textile wastewater varies depending on the method and technology used in the dyeing process (Yılmaz and Sahinkaya, 2023). In the literature, higher nitrogen concentrations in textile industry wastewater have been reported in recent studies. For example, the total nitrogen (TN) and ammonium-nitrogen ($N\text{-}NH_4^+$) in wastewater in the balancing tank of a fabric printing factory located in the textile region of Como (Italy), were 105.9±24.9 mg N/L and 93.1±23.4 mg N/L, respectively (Lotito et al., 2012). Around 120–140 mg TN/L in the influent of textile wastewater taken from a small-sized factory making zippers in Shenzhen (Guangdong, China) was reported (Zhou et al., 2021). The total Kjeldahl nitrogen (TKN) concentration of wastewater from the J textile factory in Seoul, Korea was measured between 86 and 145 mg/L (Chang et al., 2002).

Uncontrolled discharge of nitrogen-containing wastewater into the receiving environment causes eutrophication and adverse effects on ecological balance (Song et al., 2021). Nitrification and denitrification are the two preferred biological processes for nitrogen removal from wastewater. In the nitrification process, ammonium is oxidized to nitrite and nitrate in aerobic conditions (Thakur and Medhi, 2019) by ammonia-oxidizing and nitrite-oxidizing bacteria, respectively (Liu et al., 2020).

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This chapter is based on the paper ‘Yilmaz, T., Demir, E. K., Başaran, S. T., Çokgör, E. U., & Sahinkaya, E. (2023). Impact of aeration on/off duration on the performance of an intermittently aerated MBR treating real textile wastewater. Journal of Water Process Engineering, 54, 103886, DOI: 10.1016/j.jwpe.2023.103886’.

In the denitrification process, nitrate or nitrite is converted to nitrogen gas by heterotrophic and/or autotrophic microorganisms under anoxic conditions (Asik et al., 2021).

The intermittent aeration process provides many advantages such as high nitrogen removal performance due to creating appropriate conditions for simultaneous nitrification and denitrification, its suitability for low-performing wastewater treatment plants retrofitting, lower energy requirement, and less sludge problems (Miao et al., 2022). Intermittent aeration provides energy savings between 33 and 45% compared to conventional processes, which may be due to the possibility of denitrification over nitrite (Dotro et al., 2011). Intermittent aeration process is preferred in many different systems such as sequencing batch reactors (SBR) (Asadi et al., 2012; Mansouri et al., 2014), constructed wetlands (Jie Wang et al., 2020; Zhang et al., 2018), activated sludge systems (Insel et al., 2006), baffled reactors (Lucio et al., 2022), and membrane bioreactors (MBR) (Guo et al., 2013). In particular, MBRs provide advantages such as effective filtration, higher effluent quality, less sludge production, low footprint as well as high nitrogen removal efficiency compared to conventional biological systems (Tang et al., 2022). There are some studies in the literature on wastewater treatment performances of intermittently aerated MBRs under varying operational conditions. In one of these studies conducted by Kim et al. (2007), COD and TN removal efficiencies were 95.2% and 72.7%, respectively, in a full-scale treatment plant consisting of an anaerobic reactor, two modified intermittent aeration reactors, a deoxygenation (deox) tank and aerobic MBR for sewage treatment (H. S. Kim et al., 2007). In another study, a pilot scale MBR process, consisting of aerobic/anoxic, membrane, and deox tanks, was operated for municipal wastewater treatment at different aeration on/off cycle durations. The highest efficiencies obtained at the aeration on/off cycle durations of 80 min/100 min were 96±9%, 78±27%, and 75±14% for nitrification, denitrification and total nitrogen removal, respectively (Capodici et al., 2015).

Instead of achieving the nitrification and denitrification processes in two separate tanks with internal recirculation, optimum conditions for both processes may be created in a single system to minimize the size of treatment plants (Bhattacharya and Mazumder, 2021). The success of the nitrification and denitrification processes in a single system may be highly affected by parameters such as dissolved oxygen (DO), carbon/nitrogen

(C/N) ratio, hydraulic retention time (HRT), and aeration/anoxic cycles (Di Capua et al., 2022). Anoxic denitrifying bacteria are sensitive to the presence of oxygen (Rajta et al., 2020) and theoretically at least 2.86 mg COD/mg N must be provided in the system for heterotrophic denitrification (Li et al., 2022). Also, slow-growing autotrophic ammonia-oxidizing bacteria are generally recognized as the rate-limiting step in the nitrification process, and an adequate amount of nitrifiers should be present in the system for effective biological nitrogen removal (Jingyin Wang et al., 2020).

There are limited studies in the literature, on the real textile wastewater treatment using an MBR process in which intermittent aeration is appalled. In the study by Sahinkaya et al. (2017), the effect of different aeration on/off cycle durations (5 min/3 min, 1 min/10 min, and 1 min/15 min) on the dye and chromium removal performances of a dynamic membrane bioreactor treating synthetic textile wastewater was evaluated. The ammonium nitrogen in the synthetic wastewater was 33±2 mg/L, and it decreased 1.3±1 mg/L in the permeate at the aeration on/off cycle of 5 min/3 min and 1 min/10 min whereas it increased up 28 mg/L at the aeration on/off cycle of 1 min/15 min. The average COD removal efficiency was 96% until the aeration on/off durations of 1 min/15 min.

The present study aims to determine and compare carbon and nitrogen removal performances of a real textile industry wastewater at varying DO concentrations (DO of 6 and 3 mg/L) and the different aeration on/off cycle durations (from 2 min/2 min to 90 min/360 min) in an intermittently aerated MBR including a hollow fiber ultrafiltration (UF) membrane. Membrane filtration performance and fouling tendency for each operating condition were discussed in detail. Also, organic and inorganic membrane foulants under the different aeration/off cycle durations were characterized with the FT-IR, SEM, SEM-EDS, GPC and PSD analyses.

## 4.2 Materials and Methods

### 4.2.1 Bioreactor

In the study, a cylindrical lab-scale submerged MBR system with an effective volume of 7 L was used. Influent, permeate, and recirculation flows were controlled with peristaltic pumps (Longer Pump-BT600-2J, Seko-PR7). A hollow fiber PVDF 0.04 μm pore-sized ultrafiltration membrane (SUEZ) with a surface area of 0.23 $m^2$ was used in the MBR. In order to reduce membrane fouling, filtration was applied intermittently in 6 min cycles employing 5 min of permeation and 1 min of relaxation. Differential pressure values in the system were monitored with a manometer. Daily excess sludge was removed from the system manually. The system was aerated through a compressor operated according to the predetermined on/off durations with a timer. In addition, an automation system was used for adjusting intermittent aeration and pump operation when feeding was done only in anoxic conditions.

### 4.2.2 Wastewater and biomass source

The system was fed with wastewater, 95% of which was of textile wastewater origin, obtained from the Demirtaş Organized Industrial Zone (DOSAB) wastewater treatment plant located in Bursa, Turkey. General information about the treatment plant was previously provided by Sahinkaya et al. (2019), and the wastewater characterization for this study is given in Table C1. The MBR previously operated under aerobic conditions at an SRT and an HRT of 30 d and 1 d, respectively, for around 250 days (Yilmaz et al., 2023a) was used in the present study.

### 4.2.3 Operating conditions

The system operated under nine different main periods (P1-P9) for approximately 472 days. The HRT and SRT in the system were constant at 1 d and 30 d, respectively. The average and instantaneous fluxes in the system were 8.3 and 10 LMH, respectively, and a certain portion of the permeate was continuously fed back into the system to keep HRT constant at 1 day. For this case, average influent, permeate, and recirculation flow rates were set to 7, 46 and 39 L/d, respectively.

During the first two periods (P1-P2), the system was continuously aerated to have DO of 6 and 3 mg/L, respectively. Then, the system was tested under intermittent aeration conditions. In the periods between P3 and P7.1, aeration on/off cycle durations were varied between 2 min/2 min and 90 min/90 min (Table 4.1). In order to improve the TN removal performance in the system, the temperature in the reactor was first increased to around 30°C (close to the operating temperature of the real treatment plant), and then 2.5 L sludge (2515 mg VSS/L) taken from a real scale 5-stage bardenpho MBR process was added to system in P7.2. Up to P7.3, the system was fed intermittently in both on and off cycles, similar to filtration process, then the wastewater was fed to the system only during the aeration-off cycles. By this way, the system was tested at different aeration intermittencies while observing the effects of employing different feeding patterns, elevated temperature and culture supplementation. In P8 and P9.1, aeration on/off durations were set to 90 min/180 min and 90 min/360 min, respectively.

**Table 4.1:** Conditions tested throughout the study (SRT = 30 d, HRT = 1 d, average Flux = 8.3 LMH and instantaneous flux = 10 LMH).

| Period No | DO | Aeration On/Off durations | Temperature | Feed type | Feeding time |
|---|---|---|---|---|---|
| | mg/L | min/min | °C | | |
| **P1** | 6 | | | | |
| **P2** | 3 | | | | |
| **P3** | | 2/2 | | | |
| **P4** | | 15/15 | 18-25 | | Intermittent (5 min feed and 1 min cease) in all phase |
| **P5** | | 30/30 | | | |
| **P6** | | 60/60 | | Real TW | |
| **P7.1** | | | | | |
| **P7.2** | | 90/90 | | | |
| **P7.3** | | | | | |
| **P8** | | 90/180 | | | |
| **P9.1** | | | 33±0.9 | | Continuous only in anoxic phase |
| **P9.2** | | 90/360 | | Real TW + 200 mg COD/L (acetate source) | |
| **P9.3** | | | | Real TW | |

In order to increase the COD/N ratio in P9.2, 200 mg COD/L acetate was externally supplied to the wastewater and its effect on the TN removal efficiency was investigated. In the last period, the system was fed with a new batch wastewater with a relatively high COD content.

TMP was monitored regularly to determine membrane filtration performance. According to the procedure provided by Yurtsever et al. (2017), physical and chemical cleanings were performed when TMP exceeded 200 and 400 mbar in the system, respectively. The removal performance of the system was followed via regular measurements of COD, TN, nitrate, nitrite, color, etc. The DO and oxidation reduction potential (ORP) values were regularly monitored in the system. For the sludge filterability properties, regular measurements of supernatant filterability (SF), specific resistance to filtration (SRF), sludge volume index (SVI), viscosity, and capillary suction time (CST) were conducted. Soluble microbial products (SMP) and extracellular polymeric substances (EPS) concentrations as carbohydrate and protein fractions were determined at different aeration on-off durations. The GPC, FT-IR, SEM–EDX, and PSD analyses were performed to characterize the organic and inorganic foulants. Observed sludge yield ($Y_{obs}$) and energy requirement for aeration in each operating condition were calculated as given by Yilmaz et al. (2023a). The airflow requirements were calculated by assuming the average DO concentration during the aeration cycles of intermittent aeration periods around 2.5 mg/L and the oxygen transfer efficiency of the diffuser as 5%/m. Then, energy requirements were calculated based on the theoretically determined airflow rates and the actual airflow rates measured during the experiments.

### 4.2.4 Analytical methods

The regular parameters (alkalinity, COD, SS, and VSS) were measured following the Standard Methods (APHA, 2005). Nitrate and nitrite were measured using an ion chromatography (Dionex ICS-5000+, Thermo Fisher Scientific, USA). Multimeters (HACH HQ40d, WTW Multi 3420) were used to measure conductivity, pH, DO and ORP parameters inside the bioreactor. HACH kits were used for TN, ammonium, and total phosphorus. SMP and EPS samples prepared following the procedure provided by Judd and Judd (2006) and Rahman et al. (2017) were used to measure carbohydrates (Dubois et al., 1956) and protein (Bradford, 1976). SRF and SF were measured as described by Dereli et al. (2014). CST and viscosity were determined using Trion Capillary Suction Timer and Brookfield DV-E viscometer (with using the Newtonian model), respectively. Molecular weights of dissolved organic molecules were measured with the GPC (Agilent 1260 Infinity) using two PL Aquagel-OH Mixed-H columns (Mobile phase = 0.02% $NaN_3$ (w/v), T= 30°C, Q=1 mL/ min). FTIR samples were prepared according to the method given by Yurtsever et al. (2016), and measured with Perkin Elmer Spectrum Two FTIR device. PSD on the samples taken from the bioreactor was measured using a Mastersizer (Malvern 2000MU) at Yildiz Technical University, Application and Research Center for Science and Technology (BİTUAM, Istanbul, Turkey). For SEM-EDS measurements, the operating conditions in the bioreactor were simulated in the dead-end filtration system, since no observable cake layer was formed on the hollow fibre membrane used in the MBR. For this purpose, 200 mL sludge sample was drawn from the MBR, and sludge cake was formed in the dead-end filtration system, by filtration at 0.5 bar through the membrane with 0.22 µm pore size. SEM images were taken on the membrane sample using the GeminiSEM

500 device at Osmangazi University Central Research Laboratory Application and Research Center (ARUM, Eskişehir, Turkey). EDS analyzes were performed on the same membrane for detecting inorganic contaminants in the cake layer.

The statistical analysis of the data was performed according to our previous study (Yilmaz et al., 2023a).

## 4.3 Results and Discussion

### 4.3.1 Performance of intermittently aerated MBR

#### 4.3.1.1 Variations of pH, conductivity, COD, color, alkalinity and phosphate in the MBR

The pH and conductivity of the feed wastewater showed no appreciable change throughout the study averaging 8.2±0.5 and 5673±1168 µs/cm, respectively (Figure C1). Total COD in wastewater averaged 793±173 mg/L, of which approximately 70% was soluble COD. In the continuously aerated periods (P1 and P2), the removal efficiencies were above 91%, and the permeate COD averaged 64±13 mg/L at DO of 3 mg/L (Figure 4.1). Employing different aeration on/off durations resulted in COD removal efficiency to vary only between 84 and 90%. Different DO concentrations and on/off durations did not negatively affect the overall COD degradation efficiency ($p < 0.05$). Interestingly, when the operating DO concentration was decreased from 6 mg/L to 3 mg/L, the COD in the supernatant was reduced from 295±83 mg/L to 133±18 mg/L. The supernatant COD in the intermittently aerated periods was also lower than that in P1, which may be due to high agitation conditions in P1 causing the release of a part of EPS to the supernatant. Approximately 24% of the total COD was rejected by the membrane and the cake in the first period. In the following periods, the rejection by membrane and cake layer varied between 3% and 14%, while the removal due to the cake layer increased as the aeration-off duration increased (Table 4.2).

**Table 4.2 :** Variations of COD, color, total nitrogen and ammonium concentrations in MBR.

| Parameters | | P1 | P2 | P3 | P4 | P5 | P6 | P7.1 | P7.2 | P7.3 | P8 | P9.1 | P9.2 | P9.3 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **COD (mg/L)** | **Total Influent** | 879±74 | 872±111 | 672±138 | 859±197 | 720±146 | 763±256 | 780±177 | 694±90 | 755±163 | 659±224 | 711±157 | 919±240 | 862±141 |
| | **Soluble Influent** | 571±67 | 568±102 | 433±126 | 570±166 | 482±125 | 546±184 | 555±188 | 542±48 | 550±0 | 540±231 | 538±134 | 627±288 | 537±180 |
| | **Supernatant** | 295±83 | 133±18 | 86±30 | 132±29 | 119±47 | 148±47 | 154±22 | 185±21 | 212±6 | 178±55 | 183±6 | 222±16 | 177±37 |
| | **Permeate** | 81±21 | 64±13 | 65±29 | 87±23 | 78±26 | 93±19 | 90±21 | 108±4 | 109±5 | 108±10 | 111±21 | 107±9 | 107±19 |
| | **Removal Efficiency (%)** | 91 | 93 | 90 | 90 | 89 | 88 | 88 | 84 | 86 | 84 | 84 | 88 | 88 |
| **Color (Pt-Co)** | **Influent** | 825±161 | 931±110 | 842±187 | 1109±431 | 1134±387 | 1563±557 | 1318±316 | 1917±526 | 1543±251 | 1161±0 | 1266±422 | 1349±282 | 1230±363 |
| | **Supernatant** | 1133±235 | 701±153 | 566±202 | 625±69 | 751±183 | 1034±366 | 996±89 | 1035±88 | 1092±4 | 881±235 | 825±57 | 1168±82 | 1139±298 |
| | **Permeate** | 446±40 | 536±113 | 504±163 | 526±51 | 590±122 | 645±71 | 566±11 | 614±62 | 587±6 | 602±28 | 576±40 | 622±78 | 586±58 |
| | **Removal Efficiency (%)** | 46 | 42 | 40 | 53 | 48 | 59 | 57 | 68 | 62 | 48 | 55 | 54 | 52 |
| **Total Nitrogen (mg/L)** | **Influent** | 74±17 | 61±10 | 54±5 | 53±9 | 50±9 | 70±21 | 78±9 | 86±1 | 72±0 | 72±8 | 81±11 | 93±10 | 54±5 |
| | **Permeate** | 54±10 | 44±2 | 38±5 | 28±10 | 28±8 | 40±10 | 49±16 | 61±19 | 54±0 | 35±5 | 46±1 | 28±2 | 22±6 |
| | **Removal Efficiency (%)** | 27 | 28 | 29 | 46 | 43 | 43 | 36 | 29 | 25 | 51 | 44 | 70 | 60 |
| **$N\text{-}NH_4^+$ (mg/L)** | **Influent** | 71±13 | 45±4 | 43±10 | 43±10 | 41±11 | 49±17 | 60±0 | 51±22 | 59±0 | 50±10 | 63±12 | 85±6 | 46±8 |
| | **Permeate** | 0 | 0 | 0.6±0.8 | 12±9 | 2±4 | 6±7 | 4±0 | 4±6 | 4±0 | 4±0.5 | 5±3 | 9±11 | 4±3 |
| | **Removal Efficiency (%)** | 100 | 100 | 99 | 72 | 95 | 89 | 93 | 92 | 93 | 91 | 92 | 89 | 91 |

The average color in the feed and permeate were 1171±458 Pt-Co and 570±108 Pt-Co, respectively (Figure 4.1). No correlation was observed between color removal efficiency and different DO concentrations and aeration on/off cycle durations ($p < 0.05$). While the lowest removal efficiency was observed at aeration on/off durations of 2 min/2 min, the highest removal efficiency was obtained as 68% at the aeration on/off duration of 90 min/90 min. Considering the supernatant and permeate colors, approximately 7 to 45% contribution to the total removal efficiency, depending on the periods, was due to the cake layer. Color accumulation was observed in the system especially at DO of 6 mg/L as the color in the supernatant increased up to 1133 Pt-Co levels, even higher than the influent concentration. With the employment of intermittent aeration, the color in the supernatant decreased to 566 Pt-Co at aeration on/off durations of 2 min/2 min. The highest color removal due to membrane and cake layer was 45% observed in the aeration on/off cycle of 90 min/360 min, which had the longest aeration-off period.

Jegatheesan et al. (2016) summarized the studies on the treatment of textile wastewater in aerobic MBRs, and noted that the COD and color removals ranged between 60-97% and 20-98%, respectively. In the present study, the COD removal efficiency ranged between 84 and 91%, and no adverse effect of intermittent aeration on the performance was observed. The color removal performance was highly variable in the literature, and detailed studies with real textile wastewaters under intermittently aerated MBRs are needed. Around 280,000 tons/year textile dyes are released into the receiving environment with wastewater, and 60% of these are azo dyes, which may have carcinogenic and mutagenic properties (Shah, 2014). Conventional aerobic biological systems are not sufficient for azo dye removal, and combined processes including anaerobic biological ones may be required for more effective color removal (Wang et al., 2011). In the biological processes, azo dye must be reduced to aromatic compounds under anaerobic conditions. However, the presence of molecular oxygen in the environment inhibits the enzyme responsible for azo dye reduction (Sandhya et al., 2005). In a study investigating the intermittent aeration effect on azo dye removal, batch reactors containing glucose and Direct Black 22 were aerated at different numbers of cycles (0, 4 and 8 numbers/d), where each cycle was run 1 h with 2.5 L/min of air flowrate. The lowest decolorization was observed in the highest aeration cycle due to the oxygen usage as an electron acceptor instead of azo dye. Also the authors reported that the intermittent aeration process increased the biodegradation of azo dyes but decreased the rate of decolorization (Oliveira et al., 2020).

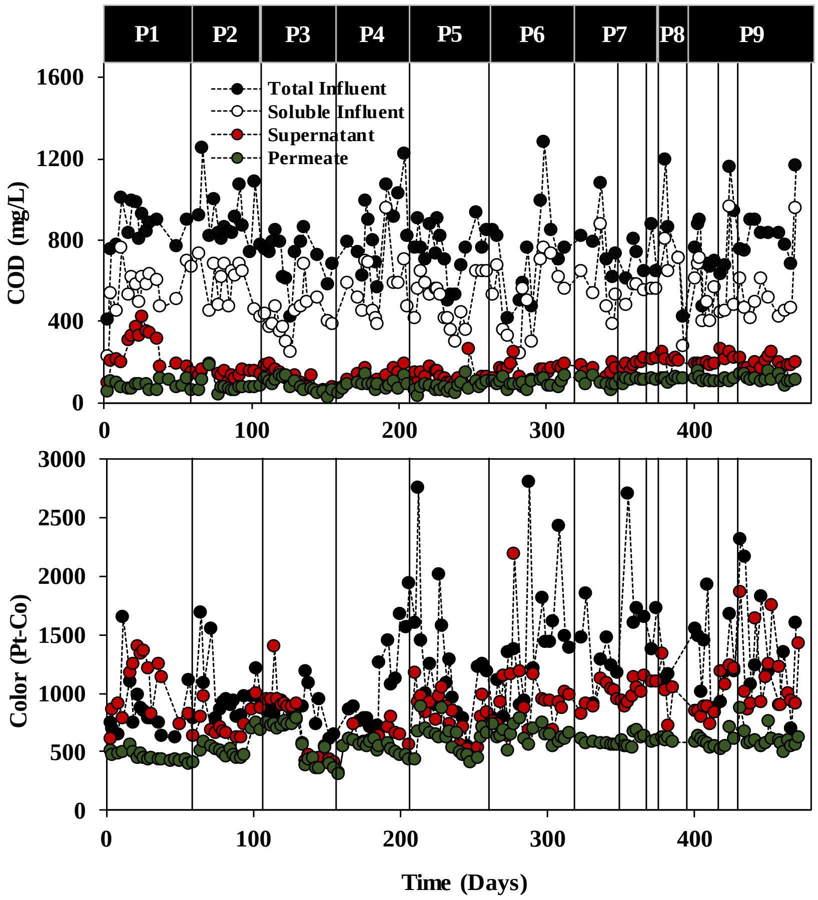


**Figure 4.1 :** COD and color variations in the MBR.

The average alkalinity of the wastewater was 974±195 mg $CaCO_3$/L, and different consumption rates were observed depending on the nitrification and denitrification performances in the periods (Figure C1). While there was approximately 51% alkalinity consumption in the first period, the consumption decreased to around 38% with the decreasing DO to 3 mg/L and the aeration on/off durations of 2 min/2 min. In the following periods, alkalinity consumption gradually decreased to 20-26% until on/off durations of 90 min/90 min (P7). The alkalinity consumption increased to 43% in P7, and varied again between 22 and 34% in the following periods. In the nitrification process, 7.14 mg $CaCO_3$ per mg N-$NH_4^+$ is consumed, while 3.57 mg $CaCO_3$ is produced per mg N-$NO_3^-$ in the denitrification process (Li and Irvin, 2007).

The TP in the wastewater averaged 4±1 mg/L, and it decreased to an average of 2.3±0.9 in the permeate, throughout the study (Figure C2). While the phosphorus removal efficiency was between 63 and 66% in the periods when the DO was 6 and 3 mg/L, the phosphorus removal efficiency decreased to 42-43% with the intermittent aeration process at the on/off time of 60 min/60 min. A rapid decrease in the phosphorus removal performance was detected at the aeration on/off cycle of 90 min/90 min as it ranged between 15-25%. However, it increased to 47-48% again by increasing the aeration off duration to 360 min. Biological P removal relies on the selection and proliferation of phosphorus-accumulating organisms (PAOs) that need to be favored due to their disadvantageous of lower yield compared to heterotrophic bacteria. Their survival is attained by employing an anaerobic phase for rapid substrate uptake with no electron acceptor, which enables the growth of PAO in subsequent anoxic and aerobic conditions. Slightly higher removal efficiency in the continuous aerobic phases is attributed to assimilation. Intermittent aeration may have enabled the growth of PAOs that could be responsible for a certain fraction of P removal, which is supported by the 48% removal achieved at P9, however the overall removal mechanism is believed to be dominated by assimilation since the results reveal less P removal with decreasing VSS in the intermittent aeration phases.

#### 4.3.1.2 Nitrogen Removal

Complete ammonium oxidation was achieved in the system during the first three periods (Figure 4.2 and Table 4.2). The decrease of DO concentration to 3 mg/L and the changing aeration on/off durations according to Table 4.1, somehow affected the nitrification performance. By increasing the aeration on/off durations to 15 min/15 min, the ammonium concentration in the permeate reached 24 mg/L N-$NH_4^+$ and the oxidation performances in the period averaged 72%. However, with increasing the on/off durations to 30 min/30 min, the ammonium oxidation efficiency increased to 95%, and remained above 89% in the following periods ($p > 0.05$). The optimum temperature for nitrification is known to be 30-35 °C (Luostarinen et al., 2006), and the nitrification efficiency was already above 90% before the temperature was increased in the system (Figure 4.2).

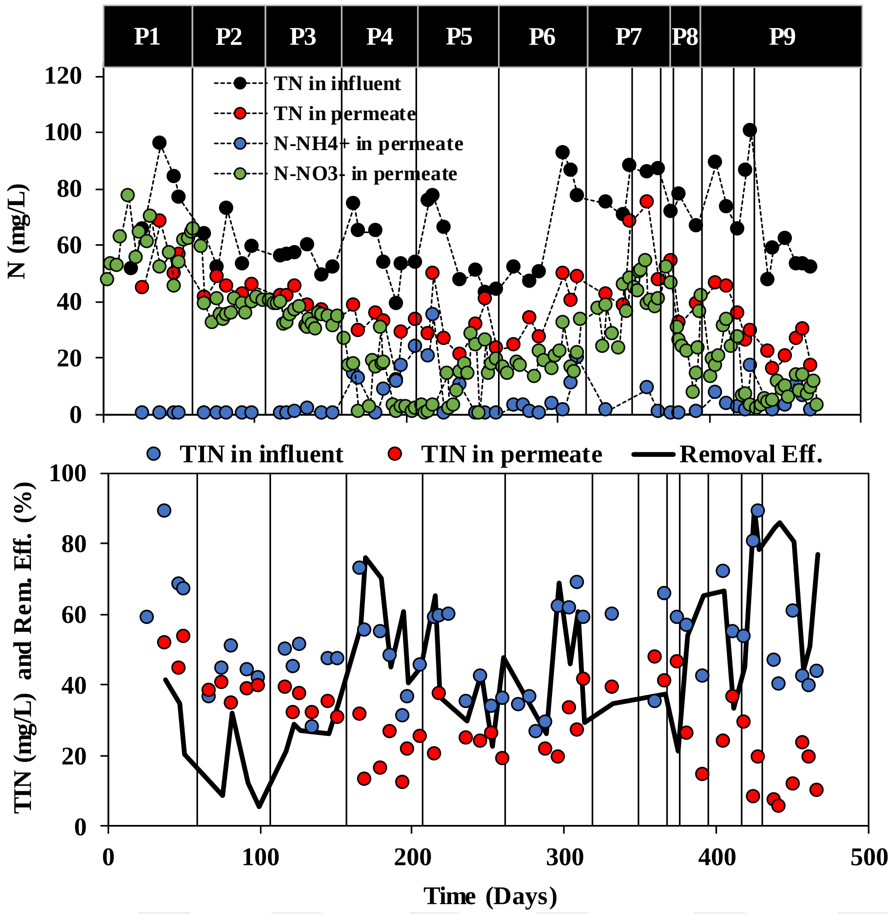


**Figure 4.2 :** TN, N-$NH_4^+$, N-$NO_3^-$ and N-$NO_2^-$ concentrations (upper figure) and variations of TIN (bottom figure) in the MBR.

During the denitrification process, nitrite accumulation occurs mostly due to the slower reduction of nitrite than nitrate (Hongwei et al., 2009). In our study, nitrite was not detected in permeate, except for three measurements, throughout the study. The highest N-$NO_3^-$ in permeate was observed at DO of 6 mg/L. The average nitrate concentration, which was 60±8 mg N/L in the first period, gradually decreased to 9±10 mg N/L at aeration on/off durations of 15 min/15 min. This led to testing longer aeration durations in the following periods, which recovered the nitrification performance as the nitrate in the permeate raised to 49±4 mg N/L at aeration on/off durations of 90 min/90 min (P7.3). By changing the aeration on/off duration to 90 min/180 min, the nitrate in permeate decreased to 24±12 mg N/L due to increased denitrification performance. The highest nitrogen removal efficiency was observed in the last period and the average nitrate in the permeate was 8±3 mg N/L and decreased down to 1.5 mg N/L.

The average TN removal efficiency for first two periods was 27-28%, and lowering the dissolved oxygen level did not affect TN removal. In the following periods, when intermittent aeration process was employed, TN removal did not change at aeration on/off durations of 2 min/2 min, and remained at the level of 29%. With the increase of the aeration on/off durations to 15 min/15 min in the P4, the TN removal efficiency increased to 46%. While TN removal remained at a level of 43% at aeration on/off cycles of 30 min/30 min and 60 min/60 min, it decreased to 36% at the aeration on/off cycles of 90 min/90 min (P7.1). After this period, as stated above, new denitrifying biomass was added to the bioreactor together with increasing the temperature to around 33$^{o}$C (P7.2), but the nitrogen removal efficiency decreased to 29%. In P7.3, the system was fed only during the anoxic phase (aeration off cycle) in order to increase the COD/N ratio during the anoxic phase. However, no increase in TN removal efficiency was observed, and it even decreased to 25%. Increasing the aeration on/off duration to 90 min/180 min and 90 min/360 min (P8 and P9.1) provided positive contributions to the TN removal efficiency as it increased to 51 and 44%, respectively. Afterward, 200 mg/L COD (acetate based) was externally added to wastewater at on/off durations of 90 min/360 min, and the TN removal raised to 70%. In the last period, the MBR was fed with a new batch of wastewater in the absence of external COD supplementation and the TN removal performance reached up 74% ($p < 0.05$) (Figure 4.2). Throughout the study, the permeate organic nitrogen concentration was generally ≥10 mg-N/L, which may have originated from non-biodegradable nitrogen-containing dyes (Clagnan et al., 2021), non-biodegradable nitrogen-containing organics in the influent, and protein part of SMPs (Arshad et al., 2021). In Figure 4.2, total inorganic nitrogen (TIN) variations in the influent and effluent and the removal performance were illustrated. In the last period, the average TIN removal efficiency increased up to 85-90%, which may be resulted from increasing non-aerated duration and feeding the reactor only in the non-aerated cycle.

In the intermittent aeration operations, high DO improves nitrification performance during the aeration period, while high denitrification performance can be achieved in the presence of a sufficient amount of carbon at the aeration off cycle (Fan et al., 2013b). In addition, the durations of on/off cycles are very important to obtain optimum conditions for a high TN removal (Mota et al., 2005). In order for the nitrification performance to remain at approximately 90% and above, the aeration cycle duration should be sufficient, i.e. 30 min or longer according to the present study. However, the TN removal efficiency remained above 70% only when the aeration off duration was 360 min. In the study by Lim et al. (2007), BOD removal efficiency and nitrification performance remained above 97% and 99%, respectively, regardless of the aeration on/off durations tested, but it was observed that at least 70 min off duration was required for obtaining denitrification efficiency above 82% in an MBR operated under intermittent aeration treating domestic wastewater. In another study, domestic sewage treatment was investigated in a mesh filtration bio-reactor operated under continuous and intermittent aeration conditions. While the BOD degradation performances were similar in all aeration cycles, the highest TN removal performance of 80% was attained at the aeration on/off cycle of 1 h/1 h (Kiso et al., 2000). In a study by Lim et al. (2004), domestic wastewater treatment was investigated in an MBR operated at intermittent aeration condition (90 min aeration/60 min non-aeration). While the COD removal performance ranged from 87.6% to 98.1%, the TN removal performance was between 35 and 70%. Also, the authors reported that the length of the anoxic period may not be sufficient for a complete denitrification (Lim et al., 2004). Although several studies have been conducted on domestic wastewater treatment using intermittently aerated MBR, a limited amount of studies were conducted on real textile wastewater treatment. Hence, the optimum operating conditions differ a lot, which should be due to the different rates and biodegradability of organics present in the both type of wastewaters.

In the study by Fan et al. (2013a), a step-feeding strategy increased the removal efficiencies of organics, ammonium, and total nitrogen in batch-operated vertical flow constructed wetlands operated under intermittently aerated conditions. The COD/N ratio in the anoxic period increased with feeding in the anoxic period only (in P7.3), and it also increased the amount of SS and VSS. In another study, the impacts of different C/N ratios (4.5, 7 and 10) on the treatment performances of an intermittently aerated MBR were investigated at an aeration on/off cycle of 60 min/60 min (HRT = 12 h). While the COD removals remained above 97% for all C/N ratios, the nitrogen removal efficiency increased as the C/N ratio increased. Nitrogen removal efficiency was 62.4, 89.1 and 92.9% for C/N ratios of 4.5, 7 and 10, respectively (Choi et al., 2008). In our study, although the COD/TN ratio was around 12, lower TN removal efficiencies may be due to the decreased rate of COD utilization in the denitrification phase and the non-biodegradable organic nitrogen in the influent.

Increasing the temperature from 18-25°C to approximately 33°C did not have a clear positive effect on denitrification performance. In the study of Elefsiniotis and Li (2006), the temperature in the denitrifying batch tests was increased from 10°C to 20°C and 30°C, respectively. The greatest positive effect on specific denitrification and carbon consumption rates was observed by the temperature increase from 10°C to 20°C.

#### 4.3.1.3 Variations of DO and ORP

Complete DO concentration above 1.5 mg/L was aimed during the aeration cycles. The air flow rate was increased when the DO decreased below 1.5 mg/L due to sudden changes in the organic load to the MBR in some periods (Figure C3). However, the average DO concentration at aeration on/off durations of 15 min/15 min decreased to 0.88±0.68 mg/L. The decrease in nitrification performance at the eration on/off cycle of 15 min/15 min is thought to be due to insufficient oxygen concentration during the aerobic cycle. In the anoxic phase, the DO decreased to ≤0.07 mg/L after the P7.1 (the aeration on/off of 90 min/90 min) (Figure C3). In addition, the variations of DO and ORP in aerobic and anoxic phases were followed regularly in each period (Figure C4). DO concentration in the aeration-off cycles decreased below 0.25 mg/L after the first 30 min, especially in periods when the anoxic duration was 60 min and longer (Figure C4). In the intermittent aeration process, the presence of oxygen and insufficient organic due to its consumption during the aeration cycle limit denitrification performance (Hu et al., 2012). In a study, a 35% reduction in the specific denitrification rate in the presence of 0.09 mg/L oxygen was reported (Oh and Silverstein, 1999).

During the first and third periods, in which anoxic conditions were limited, the average ORP varied from 89 to 108 mV. In the following periods, while the ORP at the end of the aerobic cycle varied between -118 and 108 mV, it varied between -432 and 70 mV at the end of the anoxic cycles (Figure C3). In a study by Choi et al. (2009), the treatment of synthetic domestic wastewater in MBR containing microfiltration membrane under different SRTs was investigated by applying an intermittent aeration cycle of 60 min on/60 min off. The maximum and minimum ORP values were reported as 246.5 and 11.6 mV, respectively, at an SRT of 30 d and a C/N ratio of 4.5 (Choi et al., 2009). Cheng and Liu (2001) investigated, anaerobically digested swine wastewater treatment by applying intermittent cycle of 60 min on/60 min off. DO values for the aerobic and anoxic cycles were 2-4.6 mg/L and 0.2-1 mg/L, respectively. While the ORP value varied between 80 and 100 mV in the aerobic phase, it was about 0 mV at the end of the anoxic cycle. Fuerhacker et al. (2000) reported the ORP range for simultaneous nitrification and denitrification as −60 and +198 mV.

### 4.3.2 Impact of aeration on/off durations on fouling and the membrane filtration performances

#### 4.3.2.1 Variations of TMP

The instantaneous flux in the system was 10±0.17 LMH throughout the study. The TMP in the system showed changes depending on the variations of the intermittent aeration on/off durations and DO concentration (Figure 4.3). TMP increased up to 130 mbar in the system, and no membrane cleaning was required at DO of 6 mg/L in P1. After the DO was reduced to 3 mg/L in P2, TMP increased to 420 mbar in the first five days. Backwash and physical cleaning were applied to relieve the membrane fouling, and the TMP remained below 25 mbar until the end of the period. After starting the intermittent aeration cycles, fouling formed more rapidly, especially when the aeration off duration was 60 min (P6) or longer. TMP was measured separately in aerobic and anoxic phases at aeration on/off durations of 90 min/180 min. While no fouling or cake formation was observed in terms of TMP measurement in the aerobic phase, it increased up to 180 mbar during the anoxic phase at aeration on/off durations of 90 min/180 min. By increasing the off duration to 360 min in the last period, an increase in TMP was observed even in the aerobic phase. The highest TMP values observed in the system were 130 and 420 mbar for aerobic and anoxic cycles, respectively, at aeration on/off durations of 90 min/360 min. During the study, physical cleaning was sufficient, and the TMP in the system decreased near to zero after physical cleaning as seen in Figure 4.3. Chemical cleaning was performed only during the period transitions to better observe the effect of the tested operating conditions on TMP changes.

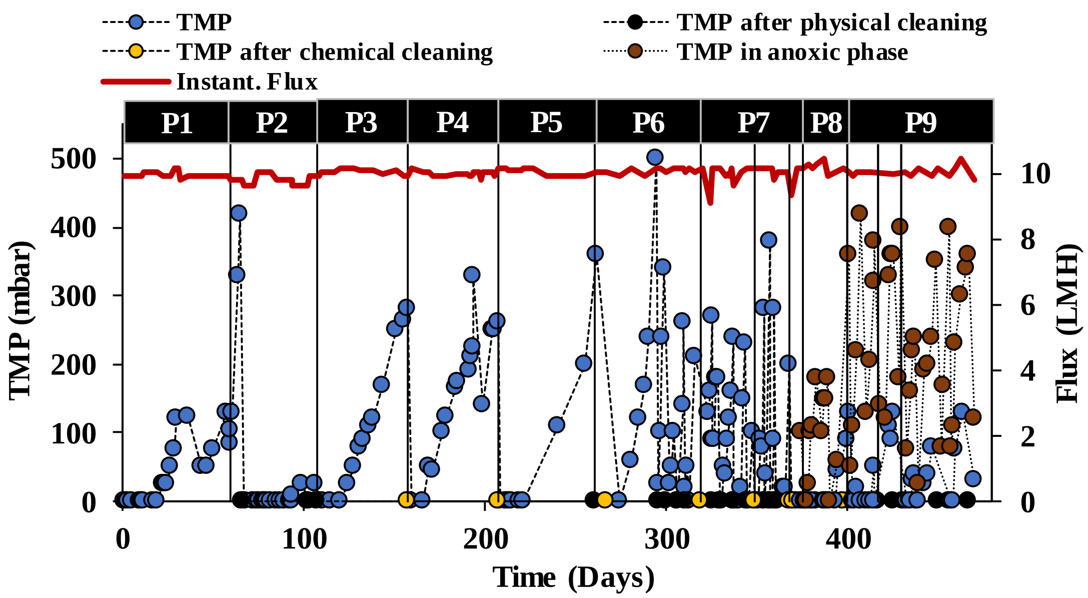


**Figure 4.3 :** TMP and flux variations in the MBR.

#### 4.3.2.2 SVI, CST, SS, VSS, SRF, SS and viscosity variations

The SS and VSS in the system were highly variable (Figure C5). In the first period, SS and VSS were 3963±192 and 3583±184 mg/L on average, and increased to 4668±438 and 4108±338 mg/L, respectively, by reducing the dissolved oxygen to 3 mg/L. Although a decrease was observed in the SS and VSS in the P3 and P4 with the initiation of the intermittent aeration process, the average SS and VSS increased to 4352±255 mg/L and 3558±253 mg/L, respectively, at the aeration on/off cycle of 30 min/30 min. After P7.3, the system was fed during the aeration off cycles. In the last periods, SS and VSS dropped by 56% and 54%, respectively, compared to the P2 (DO of 3 mg/L). In the study by Jung et al. (2006), sludge reduction was investigated in different aerobic and anaerobic cycles and the highest sludge reduction (as MLSS) rate was 69.9% at the aeration on/off cycle of 4 h/4 h.

No correlation was observed between SVI and CST values and different DO concentrations and different on/off durations (Figure C5). In general, high SVI values were observed in the system until the aeration-off duration of 360 min. The average SVI was 237±27 mL/g, and it decreased to 195±8 mL/g by decreasing DO from 6 to 3 mg/L. The average SVI in the system with intermittent aeration ranged from 136±11 to 229±2 mL/g for P3 to P7. In the last period, the lowest SVI values in the system were observed, and the SVI averaged 56±7 mL/g. The average CST decreased from 37±8 to 20±0.3 s with decreasing DO from 6 m/L to 3 mg/L in the system. The intermittent aeration process had no adverse effect on the CST, which averaged between 15±1 s and 23±6 s. Sahinkaya et al. (2017) observed that the intermittent aeration had no impact on CST and SVI values, but in the present study, a partial improvement was observed.

There was no change in the SRF in the P1 and P2, and it remained around $1.28x10^{14}$ m/kg. SRF decreased to $1.58x10^{13}$ m/kg and $5.87x10^{13}$ at the aeration on/off durations of 2 min/2 min and 30 min/30 min, respectively. However, as the aeration off duration was extended in the following periods, SRF increased to $1.61x10^{14}$ m/kg, $1.99x10^{14}$ m/kg, and $4.17x10^{14}$ m/kg at the aeration on/off durations of 60 min/60 min, 90 min/90 min and 90 min/360 min, respectively. The highest SF values were observed as 1.49 mL/min, 1.52 mL/min and 1.86 mL/min in P1 (DO of 6 mg/L), P4 (the aeration on/off durations 30 min/30 min) and P5 (the aeration on/off durations 30 min/30 min) respectively, and it varied between 0.82 mL/min and 1.13 mL/min in other periods without observing a clear trend.

Similar trends were observed in the viscosity analysis. The viscosity increased from 2.24 cP to 2.67 cP as the DO was reduced from 6 mg/L to 3 mg/L. This increase continued with the intermittent aeration process, and it was 3.05 cP, 3 cP and 4.71 cP at the aeration on/off durations of 2 min/2 min, 15 min/15 min, and 30 min/30, respectively. Contrary to SRF, a decrease in viscosity was observed by increasing the aeration on/off duration further and it decreased to 2.11 cP at the aeration on/off cycle of 90 min/360 min. The decreases and increases in viscosity are thought to be related to the SS concentrations. In another study conducted by Kornboonraksa et al. (2009), sludge viscosity increased as the MLSS increased at aeration on/off cycles of 60 min/60 min. In the study by Nagaoka and Kudo (2002), intermittent aeration process had no effect at high organic loadings, but resulted in a lower viscosity at lower organic loadings.

#### 4.3.2.3 SMP and EPS variations

During the P2 (DO of 3 mg/L), SMP in the supernatant was 29±1.7 mg COD/L and decreased to 23±2 mg COD/L until the aeration on/off durations of 60 min/60 min. The SMP in supernatant increased to 32±1 mg COD/L at the aeration on/off durations of 90 min/90 min, but decreased back to 24±11 mg COD/L when the off duration extended to 360 min. SMP decreased in the system with the initiation of intermittent aeration. One reason may be that with the intermittent aeration in the system, bacteria are exposed to biochemical stress due to the low aeration intensity, resulting in a lower concentration of SMP (Wu and He, 2012). The carbohydrate and protein content of SMP in supernatant varied between 58-76% and 42-24%, respectively, independent of the operating periods. A more stable trend was observed in the average SMP concentration in permeate, and it was 15±3.5 mg COD/L throughout the study. The average carbohydrate and protein contents of SMP in permeate were 54-62% and 46-38%, respectively, and higher carbohydrate rejection by the membrane and cake layer was observed.

No significant changes were detected in EPS concentrations throughout the study as it averaged 32±7 mg COD/g VSS, with approximately 23 and 77% of loosely (LB-EPS) and tightly bound EPS (TB-EPS), respectively. While the carbohydrate and protein contents of LB-EPS ranged between 35-47 and 65-53% until the aeration on/off durations of 90 min/90 min, both contents were 58 and 42% at the aeration on/off cycle of 90 min/360 min respectively. The contents of carbohydrate and protein in the TB-EPS were 57-67 and 43-33%, respectively. Carbohydrate and protein contents are closely related to fouling, and a high P/C ratio cause high sludge hydrophobicity (Satyawali and Balakrishnan, 2008). In our previous study, similar SMP and EPS concentrations were obtained in the MBR system treating real textile wastewater under continuous aeration at SRT of 30 d. However, while the rejection of supernatant SMP by the membrane and the cake was 53% (Yilmaz et al., 2023a), it decreased to 24% in this study. Also, a higher percentage of LB-EPS and protein contents in the SMP and EPS were observed in the present study (Figure 4.4).

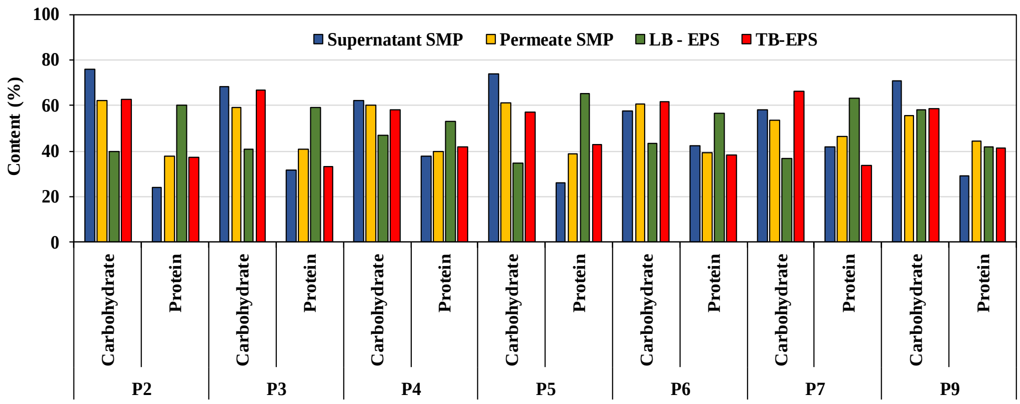


**Figure 4.4 :** SMP and EPS content variations in MBR.

#### 4.3.2.4 Molecular weights of dissolved organic molecules

Prior to GPC analyses, samples were filtered through a 0.45 pore-sized filter. The GPC results of wastewater, supernatant and permeate samples are illustrated in Figure C6. Although the permeate peaks were relatively lower, similar peaks were detected in the wastewater, supernatant and permeate of MBR. In the all samples, only four peaks were observed corresponding to approximately 23 kDa, 5 kDa, 1 kDa and 0.3 kDa until the last period. While, peaks corresponding to approximately 22 kDa, 4 kDa, 0.7 kDa and 0.3 kDa were observed in the wastewater and supernatant, another peak of 1.8 kDa was identified in the permeate at the aeration on/off cycles of 90 min/360 min. In addition, it was observed that the peak signals in all periods were very close to each other except for the P5.

#### 4.3.2.5 Particle size distribution of MBR mixed liquor

Particle size distribution (PSD) analyzes for each operating condition are shown in Figure C7. According to the PSD results, particles generally clustered between 1 and 955 μm in the supernatant. An increase in particle size was observed with decreasing DO concentration and increasing aeration-off duration, with the highest mean diameter observed at the aeration on/off cycle of 90 min/360 min. The mean diameter in the supernatant was 34 and 58 μm at the DO concentration of 6 and 3 mg/L, respectively. With the start of intermittent aeration in the system, it increased to 70 μm at the aeration on/off cycle of 2 min/2 min, and then it decreased to 38 μm at the aeration on/off durations of 15 min/15 min, which may be due to limited aeration during the aeration-on cycle as mentioned before. In the aeration on/off cycle of 30 min/30 min, the mean diameter increased to 71 μm and remained between 111 and 131 μm in the following periods. Floc formation is closely related to the aeration process, and high aeration intensity caused dispersion in the sludge flocs (size ranges from 100 to 500 μm), and therefore micro flocs (size ranges from 10 to 15 μm) are formed inside the reactor (Wu and He, 2012). In the present study, a high mean diameter of over 100 μm was observed, especially in the prolonged aeration-off periods.

#### 4.3.2.6 FTIR analyses

FT-IR spectra results for each operating condition throughout the study are given in Figure C8. Similar characteristic peaks in each condition were observed at 618, 800, 875, 1021, 1100, 1260, 1406, 1450, 1540, 1640, 1720, 2850, 2920 and 3280 1/cm. In the study conducted by Sahinkaya et al. (2017), 2920 1/cm and 2850 1/cm peaks were observed, which may be due to aliphatic C-H stretching. In addition, 1540 1/cm and 1245–1260 1/cm peaks were observed, which may be due to unique protein structure, specified amides II (N-H in plane) and III (C-N stretching), respectively. 1720 1/cm corresponds to the carboxylic groups associated with the typical properties of humic and fulvic acids (Jarusutthirak et al., 2002). The spectrum between 3411 1/cm and 3288 1/cm represents the O-H stretching of the O-H bond in the hydroxyl functional groups (An et al., 2009). 875 and 1640 1/cm can be attributed to strong carbonate bands and amide bands I (C=O vibration), respectively (Smidt and Parravicini, 2009). The spectrum around 600 and 900-1100 1/cm may be attributed to inorganic sulfur compounds and Si–O stretch of clay minerals and Si–O–Si vibration of silica, respectively (Carballo et al., 2008). 1450 1/cm can be attributed (C-H bending) for C-H group (Ashokkumar and Ramaswamy, 2014).

#### 4.3.2.7 SEM-EDS analysis and inorganic components in the MBR

The SEM images of the clean and cake-deposited membrane surfaces are given in Figure C9. Ca, Mg, Si and Na peaks were identified in the cake layer in period 9 (Figure 4.5). Similar results were obtained in other periods. Mg, Al, Fe, Ca and Si have important effects on gel layer formation (Wang et al., 2008), and contribute to a dense cake layer formation bridging cells and biopolymers (Yurtsever et al., 2017).

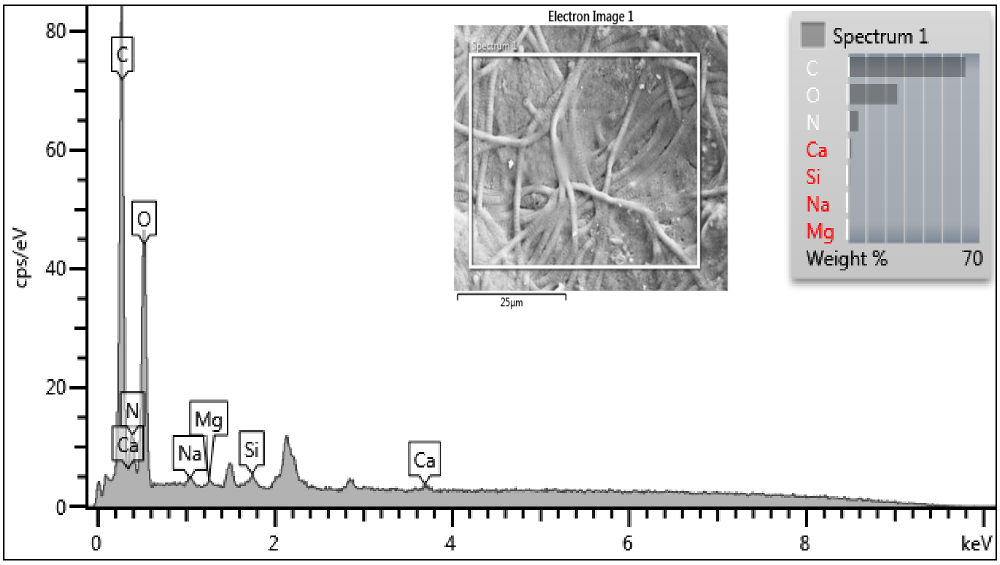


**Figure 4.5 :** EDS result of cake-deposited membrane surface at the aeration on/off durations of 90 min/360 min.

### 4.3.3 Impact of aeration on/off cycles on observed yield and spesific energy requirements for aeration

The variation of $Y_{obs}$ values and energy requirements for aeration at different operating conditions are shown in Figure 4.6. $Y_{obs}$ varied between 0.16 and 0.23 mg SS/mg COD in between P1 and P5 (the aeration on/off durations of 30 min/30 min). However, with the increase of the aeration off duration, a serious decrease was observed in the $Y_{obs}$, and it ranged between 0.09 and 0.13 SS/mg COD in the following periods. In the literature, a 12.6% decrease in sludge yield was reported in the longer non-aeration durations (Guglielmi and Andreottola, 2011) due to the consumption of organic matter during denitrification rather than aerobic oxidation (Miao et al., 2022). As the aeration on/off duration increased, 48% less waste sludge was generated in the last period compared to P1 (continuous aeration). Silva et al. (2018) investigated post-treatment of anaerobic process effluent in a structured-bed reactor, less sludge production was reported with the intermittent aeration process. In another study, sludge production was observed as 8.56, 8.35, 7.39 and 5.79 kg/d for continuous aeration and the aeration durations of 2.5 h/0.5 h, 2 h/1 h, 1.5 h/1 h, respectively, in pilot-scale fixed-film integrated activated sludge system. Hence, intermittent aeration has several benefits including less sludge generation compared to the conventional aerobic process.

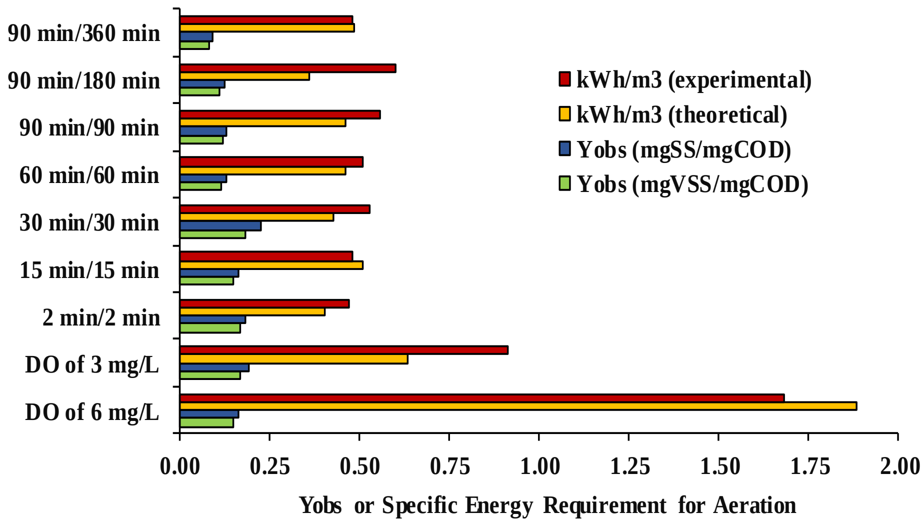


**Figure 4.6 :** The impact of aeration on/off durations on the $Y_{obs}$ or specific energy requirements for aeration.

In general, the amount of energy requirement for aeration constitutes approximately 40–75% of the total energy amount (Mamais et al., 2015). Nitrogen and carbon removal in a single reactor with intermittent aeration offers an opportunity to minimize the energy requirement (Singh et al., 2018) due to several reasons including the consumption of organic matter in an anoxic condition instead of an aerobic condition, the acceleration of nitrifying bacterial activities under intermittent aeration condition (Miao et al., 2022), and the increased possibility of short-cut denitrification. Doan and Lohi (2009) reported energy savings of 27-58% with intermittent aeration for wastewater treatment in their study. On the other hand, Dan et al. (2021) reported 60% energy savings with intermittent cycle extended aeration systems. In our study, while the experimentally determined specific senergy requirement for aeration was 1.7 $kWh/m^3$ during continuous aeration at DO of 6 mg/L, it decreased to 0.9 $kWh/m^3$ with the reduction of DO to 3 mg/L. The specific energy requirement averaged 0.53±0.06 $kWh/m^3$ under intermittent aeration conditions corresponding to around 69 and 42% reductions compared to P1 and P2, respectively.

## 4.4 Conclusion

Real textile wastewater treatment was evaluated in an MBR under different DO concentrations (6 and 3 mg/L) and the intermittent aeration periods (on/off durations from 2 min/2 min to 90 min/360 min). The highest TN removal efficiency was observed at the aeration on/off durations of 90 min and 360 min. COD removal was over 84% throughout the operation. During the treatment of real textile wastewater with the intermittent aeration process, the aeration duration should be ≥30 min for an efficient nitrification performance, and the aeration-off cycle duration should be ≥180 min for an acceptable denitrification performance. Appreciable reductions in $Y_{obs}$ and aeration energy requirements were observed with intermittent aeration. While the SS, VSS, SF and viscosity decreased under intermittent aeration conditions, SRF increased. No changes were observed in the molecular weights of organic matters in supernatant and permeate. In general, the average particle size increased as the aeration-off cycle duration increased. Ca, Mg, Si and Na were the main inorganics detected in the cake layer.

# 5. SPECIFIC AMMONIUM OXIDATION AND DENITRIFICATION RATES IN AN MBR TREATING REAL TEXTILE WASTEWATER FOR SIMULTANEOUS CARBON AND NITROGEN REMOVAL

## 5.1 Introduction

Nitrogen-derived substances in wastewater cause significant problems in the receiving environment, and great efforts are put on nitrogen removals from domestic and industrial effluents. In the conventional nitrogen removal processes, organic nitrogen/ammonium ($NH_4^+$-N) must first be bio-oxidized to nitrite/nitrate under aerobic conditions through the nitrification process, and in the second stage, nitrate/nitrate is converted to nitrogen gas under anoxic conditions through denitrification (Miao et al., 2022). However, conventional systems involving both processes separately are known for their high operating costs due to high aeration requirement in the nitrification process and high amount of sludge production during denitrification (Al-Hazmi et al., 2022). Another preferred approach to limit carbon and energy requirements, and hence, increase the process performance, is the nitritation-denitritation process (Zou et al., 2020) where nitrite produced by the partial ammonium oxidation is directly converted to $N_2$ gas during the denitritation process, reducing the carbon and aeration requirement by 40% and 25%, respectively, for nitrogen removal (Regmi et al., 2014). Hence, achieving simultaneous nitrification/nitritation and denitritation/denitrification in a single reactor instead of sequential reactors provides significant operating cost reductions (Asadi et al., 2012).

Intermittent aeration is a promising process for effective simultaneous carbon and nitrogen removals from wastewater, particularly by creating sequential aerobic and anoxic/anaerobic conditions in a single reactor (Sahinkaya et al., 2017).

In a study by Hasar et al. (2002), the impact of various aeration strategies (continuous, the aeration on/off time of 30/30 min, 60/120 min, 60/90 min, 60/75 min) on the

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This chapter is based on the paper 'Yilmaz, T., Demir, E. K., Başaran, S. T., Çokgör, E. U., & Sahinkaya, E. (2023). Specific ammonium oxidation and denitrification rates in an MBR treating real textile wastewater for simultaneous carbon and nitrogen removal. Journal of Chemical Technology & Biotechnology. DOI: 10.1002/jctb.7492'.

treatment performance of domestic wastewater in a membrane bioreactor (MBR) system was examined. Chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN) s

Optimization of operating conditions such as DO, hydraulic retention time (HRT), carbon/nitrogen (C/N) ratio, and oxic/anoxic cycle time is necessary for effective nitrification and denitrification processes in a single system (Di Capua et al., 2022). The abundance of nitrifying bacteria and the ammonia oxidation rate are highly affected by environmental parameters such as temperature, ammonium and DO concentrations (Kim, 2013; Lydmark et al., 2007). Especially, the limited DO concentration in the system may affect the reproduction and metabolic activities of nitrifying bacteria (De Morais et al., 2020). In addition, proportional distributions of ammonium oxidizing bacteria (AOB) and nitrite oxidizing bacteria (NOB) in the biological processes having aerobic and anoxic cycles cause significant differences in nitrification rates (Magdalena A Dytczak et al., 2008). Nitrogen oxides are used as electron acceptors by denitrifying bacteria, (Plósz et al., 2003) and therefore, denitrification rates are adversely affected in the presence of oxygen (Sirivedhin and Gray, 2006). In batch studies, a decrease of 35% in the specific denitrification rate was observed even in the presence of 0.09 mg/L oxygen (Oh and Silverstein, 1999). In general, the effects of nitrate and oxygen concentrations and pH on the denitrification rate are investigated for the control of the denitrification process (Wallenstein et al., 2006). In addition, the sizing of the biological pre-denitrification reactor depends on the denitrification rate (Raboni et al., 2014).

Many existing full-scale textile wastewater treatment plants are operated under aerobic conditions with continuous aeration strategy (Jegatheesan et al., 2016). These plants, having low nitrogen removal performances due to the absence of anoxic conditions, should be retrofitted for higher nitrogen removals. Changing the aeration strategy from continuous to the intermittent one may easily be applied, and significantly improve the plant performance in addition to decreased energy demand. Although studies have been conducted to elucidate the impact of aeration on/off time on the specific nitrification/denitrification rates of biomass for domestic wastewater treatment, (Baek and Pagilla, 2008; Lim et al., 2007) there are few, if any, studies on specific nitrification/nitritation/denitrification rates for real-textile wastewater treatment in an intermittently aerated MBR. It would be important for the operators and designers to get knowledge about the effects of intermittent aeration on/off time and nitrite/nitrate accumulation on the specific rates and process efficiency. In the present study, we will provide and discuss the specific ammonium oxidation/denitritation/denitrification rates of the biomass obtained from the intermittently aerated MBR for which long-term performance data was recently published (Yilmaz et al., 2023b).

## 5.2 Materials and Methods

### 5.2.1 Biomass source

Biomass for batch ammonium oxidation/denitrification studies were obtained from a lab-scale MBR treating real textile wastewater under varying aeration strategies. The MBR achieved COD, color and total nitrogen (TN) removal efficiencies of 84-93%, 40-68% and 25-70%, respectively, depending on the operational conditions (Yilmaz et al., 2023b). Biomass obtained from the MBR was directly used for the batch studies without making any concentration adjustments, and hence, the biomass concentrations (760 – 3600 mg VSS/L) at different batch tests were as illustrated in Table 5.1.

### 5.2.2 Batch test for specific ammonium oxidation rates

In order to determine the variations of the specific ammonium oxidation rate, at each tested operational condition (C1-C8) (Table 5.1), one liter of homogeneous sludge sample was taken from the MBR, and continuously aerated in a two-liter glass reactor until a constant DO concentration, which may close to saturation levels, was attained. After reaching stable DO, the tests were started by adding the synthetic concentrated wastewater to obtain initial concentrations of 50 mg $NH_4^+$-N/L, 44.6 mg $KH_2PO_4$/L, and $NaHCO_3$ as 500 mg $CaCO_3$/L (as an alkalinity source) in the aerated batch reactors. The tests were carried out at room temperature (18-25 °C), and variations of $NH_4^+$-N, nitrite-nitrogen ($NO_2^-$-N), nitrate-nitrogen ($NO_3^-$-N), COD, and alkalinity were followed by taking samples regularly.

**Table 5.1 :** The operating conditions and biomass concentrations in the tests.

| Operating Conditions in MBR | Biomass (mg VSS/L) | | |
|---|---|---|---|
| | In the batch tests for specific ammonium oxidation rates | In the respirometric studies for specific ammonium oxidation rates | In the batch tests for denitritation and denitrification and rate |
| **C1 = DO of 6 mg/L** | 3310 | 3480 | 1655 |
| **C2 = DO of 3 mg/L** | 3600 | 2860 | 2210 |
| **C3 = 2 min on/2 min off** | 3550 | 3570 | 1610 |
| **C4 = 15 min on/15 min off** | 3390 | 2980 | 1695 |
| **C5 = 30 min on/30 min off** | 3290 | 3090 | 1690 |
| **C6 = 60 min on/60 min off** | 2270 | 2400 | 1180 |
| **C7 = 90 min on/90 min off** | 2680 | 2850 | 1340 |
| **C8 = 90 min on/360 min off** | 1660 | 1660 | 760 |

In addition to these batch oxidation tests, the specific ammonium oxidation rates were also measured using a respirometric method based on the oxygen consumption rates of bacteria during the oxidation process. The nitrification reaction according to the United States Environmental Protection Agency is given in Reaction 5.1 (Parker, 1975).

$$NH_4^+ + 1.83O_2 + 1.98HCO_3^- \rightarrow 0.021C_5H_7O_2N + 0.98NO_3^- + 1.041H_2O + 1.88H_2CO_3 \quad (5.1)$$

The BM-Advance respirometer (Surcis, Spain) was used for the measurement of dynamic oxygen uptake rate ($R_S$) (mg $O_2$/L-h), specific dynamic oxygen uptake rate ($R_{Sp}$) (mg $O_2$/L-h), and consumed oxygen concentration (CO) (mg/L). The test modes and parameters that can be measured in the BM-Advance respirometer are given by Cristóvão et al., (2016). Sludge taken from each corresponding MBR operating condition (Table 5.1) was placed in a respirometric chamber, and aerated continuously until a stable DO value was obtained. After reaching stable DO concentration, the respirometric test was initiated with the addition of 50 mL of distilled water containing 50 mg $NH_4^+$-N/L to determine the Rs, Rsp, and CO continuously in the R-mode throughout the test. In the calculation of the theoretical oxygen consumption, biomass growth was excluded, and the oxygen requirements for nitrite and nitrate generation from ammonium were considered as 3.42 mg $O_2$/mg $NO_2^-$-N and 4.57 mg $O_2$/mg $NO_3^-$-N, respectively. The possibility of simultaneous nitrification and denitrification in the calculations was neglected.

### 5.2.3 Batch test for specific denitritation/denitrification rates

In order to investigate the specific denitritation and denitrification rates, batch reactors (8 batch reactors for each MBR operational condition C1-C8) with an active volume of 100 mL were set up with sludge taken from the MBR. Before starting the tests, the sludge was rinsed with tap water to remove all organic and inorganic matter attached to biomass, and kept in a sealed bottle for one day to allow the microorganisms to adapt to the anoxic condition.

Batch tests were initiated by adding 50 mL of sludge to the batch reactors. The batch assays between R1 and R4 were operated in parallel at different nitrite or nitrate concentrations (Table 5.2).

**Table 5.2 :** The operating conditions of the batch reactors for determining specific denitritation and denitrification rates.

| Reactor No | $NO_2^-$-N (mg/L) | $NO_3^-$-N (mg/L) | COD (mg/L) | Biomass (mL) |
|---|---|---|---|---|
| **R1** | 0 | 25 | 150 | 50 |
| **R2** | 0 | 50 | 300 | 50 |
| **R3** | 25 | 0 | 150 | 50 |
| **R4** | 50 | 0 | 300 | 50 |
| **R5** | 0 | 25 | 150 | 0 |
| **R6** | 25 | 0 | 150 | 0 |
| **R7** | 0 | 25 | 0 | 50 |
| **R8** | 25 | 0 | 0 | 50 |

No sludge was added to R5 and R6 to investigate whether the nitrogen removal process is biological. In addition, external organic matter was not added to R7 and R8 in order to determine the nitrate and nitrite removal rates due to endogenous respiration of bacteria, and parallel studies for R5, R6, R7 and R8 were not carried out (a total of 12 batch reactors for each condition).

The COD/TN ratio was kept at 6.0 to avoid organic matter limitation in the tests, using acetate as the carbon source in all tests and considering the denitrification/denitritation reactions provided in Reactions 5.2 and 5.3 (Ma et al., 2020).

$$0.25CH_3COO^- + NO_3^- \rightarrow NO_2^- + 0.25HCO_3^- + 0.25H_2O + 0.25CO_2 \qquad (5.2)$$

$$0.375CH_3COO^- + NO_2^- + 0.625H^+ \rightarrow 0.5N_2 + 0.75HCO_3^- + 0.5H_2O \quad (5.3)$$

In addition, inorganic media given by Sahinkaya and Dilek (2005) was also added to the batch reactors in order not to limit the microbial growth.

The denitrification efficiency is affected by many parameters such as nitrate concentration, temperature, type and concentration of carbon source, and the presence of DO in the system (Sirivedhin and Gray, 2006). The sludge was kept sealed for one day before the tests to reduce the DO level. All batch tests were conducted at 25°C in an incubator, and the nitrogen removal rates were determined by monitoring the variations in nitrate, nitrite and COD concentrations with time.

### 5.2.4 Analytical methods

COD, alkalinity, suspended solid (SS), and volatile suspended solid (VSS) concentrations in the batch tests were followed according to Standard Methods (APHA, 2005). $NO_2^-$-N and $NO_3^-$-N were measured with Dionex ICS-5000+ (Thermo Fisher Scientific, USA) ion chromatograph equipped with an AS9-HC column. Multimeter (WTW 3420, Xylem Analytics, Weilheim, Germany) was used for DO measurements in the batch reactor. $NH_4^+$-N concentrations were measured using HACH kits (Hach Company, Loveland, Colorado). Respirometric studies were performed using the BM-Advance Respirometer (Surcis, Spain). Free ammonia (FA), nitrite accumulation rate and ammonium oxidation efficiencies were calculated as described by Zhou et al. (2020). Free nitrous acid (FNA) was determined as given by Asik et al. (2021).

## 5.3 Results and Discussion

### 5.3.1 Specific ammonium oxidation rates

It was aimed to determine the effect of different DO values and the aeration on/off time employed in an MBR (Yilmaz et al., 2023b) on the specific ammonium oxidation rate using batch tests (C1-C8) (Figure 5.1). The ammonium soxidation efficiencies were over 99% in all batch tests at which DO concentrations were mostly >4 mg/L not to limit the nitrification process.

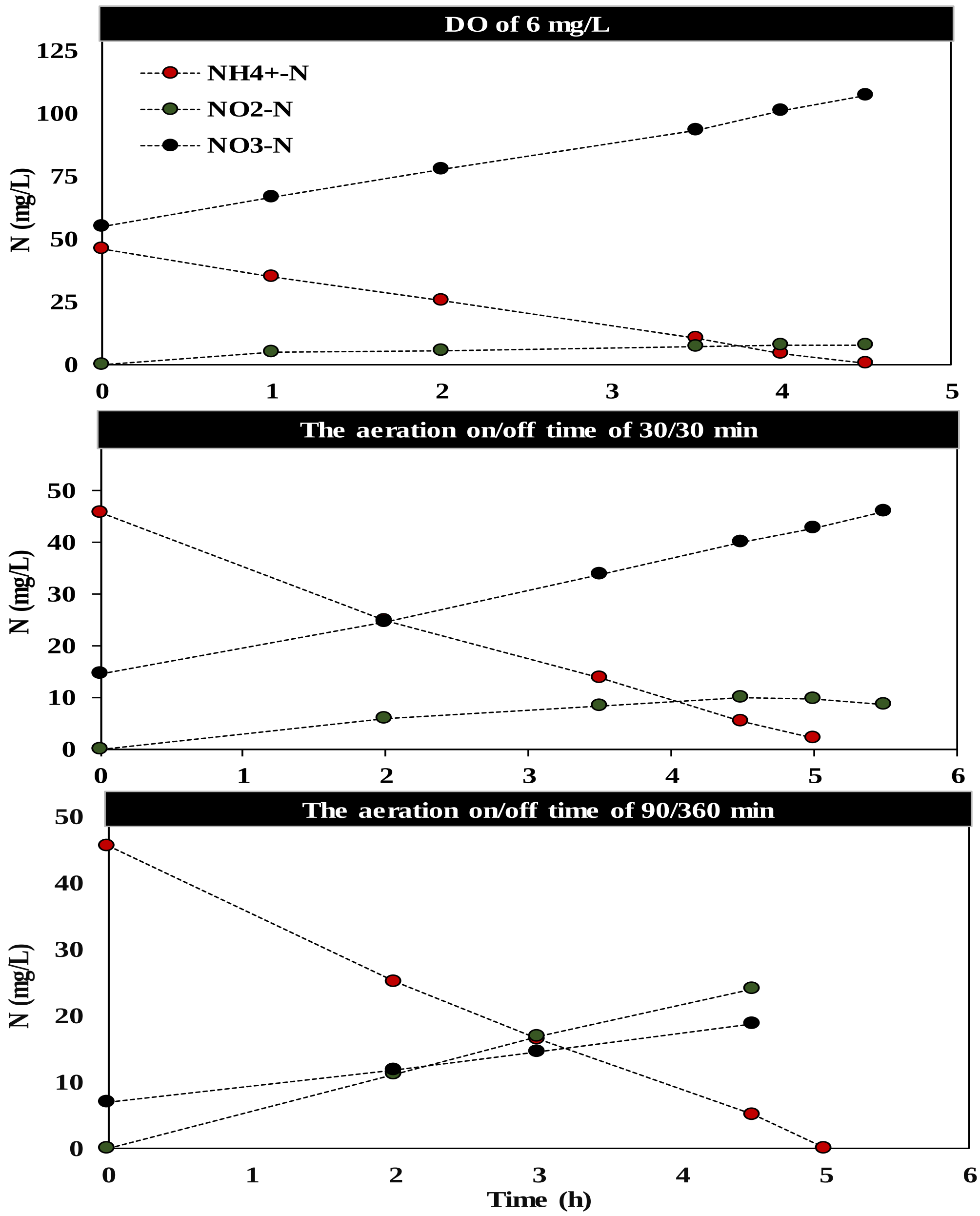


**Figure 5.1 :** Example results from batch tests for specific ammonium oxidation rates. Condition 1 (upper figure), Condition 5 (middle figure), Condition 8 (bottom figure).

In the first batch tests conducted with the sludge from the MBR operated at DO of 6 mg/L DO (C1), the specific ammonium oxidation rate was 3.1 mg N/(g-VSS.h). With the decreasing of DO to 3 mg/L in MBR (C2), the specific ammonium oxidation rate decreased to 2.6 mg N/(g-VSS.h). No significant changes were observed at the aeration on/off time of 2/2 min (C3) compared to C2, and the specific ammonium oxidation rate was determined as 2.3 mg N/(g-VSS.h). However, by increasing the aeration on/off time to 15/15 min (C4), the specific ammonium oxidation rate decreased to 1.3 mg N/(g-VSS.h). In the following conditions, the aeration on/off durations were increased, and the specific ammonium oxidation rates were 2.6, 2.1, and 3.2 mg N/(g-VSS.h) at the aeration on/off time of 30/30 min (C5), 60/60 min (C6), and 90/90 min (C7), respectively. Further increasing the aeration off time to 360 min at the last run (C8), the highest specific ammonium oxidation rate of 5.4 mg N/(g -VSS.h) in the MBR system was obtained. Compared to the batch tests conducted with the sludge obtained from the continuously aerated MBR (conditions: C1 and C2), the batch test carried out with the sludge taken during the condition C8, at which the longest aeration off time was performed in the MBR, resulted in approximately two times higher ammonium oxidation rate. These results agree with literature, reporting that the nitrification rate increases nearly two times in the case of intermittent aeration compared to continuous aeration due to the dominance of rapid-nitrifiers (*Nitrosomonas* and *Nitrobacter*) under intermittent aeration condition rather than the slow-nitrifiers (*Nitrosospira* and *Nitrospira*) under continuous-aeration condition (Miao et al., 2022). In the two different sequencing batch reactors (SBRs) operated under aerobic and alternating anoxic/aerobic conditions, the specific nitrification rates were 2.95 ± 0.26 and 6.16 ± 0.34 mg $NH_4^+$-N/(g-VSS.h) under aerobic and alternating anoxic/aerobic conditions, respectively. One of the most important factors in this increase is that the anoxic cycle and the high nitrite concentrations create favorable conditions for the *Nitrosomonas* and *Nitrobacter*, which rapidly grow at high substrate concentrations due low substrate affinity (high half velocity constant, Ks) (Dytczak et al., 2008).

Szpyrkowicz et al. (1996) reported the nitrification rates as 0.01 and 0.05 mg N/(mg MLSS (mixed liquor suspended solids).d), at the SRT of 75 and 65 d and, DOs of 3.6 and 4.3 mg/L, respectively, during the treatment of textile dyeing wastewater in a full-scale extended aeration process. The ammonium oxidation rates observed in our study are comparable with the rates reported in the literature for different wastewaters. Gao et al., (2004) reported that the specific nitrification rate ranged from 0.27 to 0.56 g $NH_4^+$-N/(g-SS.d) in an aerobic MBR treating synthetic inorganic wastewater. In the other study investigating landfill leachate treatment, the specific nitrification rate was between 0.043 and 0.154 kg $NH_3$-N/(kg-SSV.d) in an SBR (Yabroudi et al., 2013). However, in the study by Ganesh et al. (2015), the specific nitrification rate was calculated as high as 6.9 mg/(g-sMLVSS.h) during tannery wastewater treatment in the aerobic SBR (cycle and react phase durations were 12 and 10 h, respectively).

The specific nitrate production rates also showed similar trends to the specific ammonium oxidation rate, except for aeration on/off time of 90/360 min condition. The nitrate production rates were observed to be higher, 3.5 and 2.3 $NO_3^-$-N/(g-VSS.h) at DO of 6 and 3 mg/L, respectively, for the continuously aerated MBR sludge compared to that of intermittently aerated ones as it decreased significantly with increased off time (C3-C8) down to 1.6 $NO_3^-$-N/(g-VSS.h) at aeration on/off time of 90/360 min. Interestingly, the specific ammonium oxidation rate reached its maximum for the tested sludge obtained at the longest aeration off time (C8). The highest nitrite accumulation ratios observed were 29, 23, 26 and 67% in C4, C5, C6 and C8, respectively, while it was below 1% in C2 with continuous aeration. In the intermittent aeration process, nitrite accumulation occurs commonly due to quickly overcome of ammonium oxidizing bacteria after a longer anoxic condition (Bournazou et al., 2013; Z. Zhou et al., 2020). Turk and Mavinic (1986) observed that with the prolongation of the aeration time, the negative effect on nitrite oxidizers is decreased, and the accumulated nitrite was further oxidized. Hence, intermittent aeration with long non-aeration times may eliminate nitrite-oxidizers, and partial nitrification process may become dominant (Miao et al., 2022).

The nitrite accumulation and activities of AOBs and NOBs are highly influenced by important factors such as pH, temperature, DO levels, presence of FA and FNA (Yang and Yang, 2011; Zhou et al., 2011). The pH between 7.5 and 8.5 was reported as suitable for nitrite accumulation (Sinha and Annachhatre, 2007) and the average pH of sludge used in nitrification tests was 8.4 ± 0.2. The DO varied between 4 and 8.8 mg/L throughout the tests, and nitrite accumulation is generally observed at lower oxygen concentrations (Zhou et al., 2011). The specific growth rates of AOBs and NOBs are temperature-dependent, and usually temperatures of 25°C and above may be favorable conditions for nitrite accumulation due to amplifying the differences in specific growth rates between AOB and NOB (Guo et al., 2010; Yan et al., 2019). AOB and NOB can be affected at a FA range of 10 – 605 mg N/L and 0.1-5 mg N/L, respectively (Abeling and Seyfried, 1992; Liu et al., 2020). The FNA concentration in the range of 0.42–1.72 mg $HNO_2^-$N/L yielded a 50% decrease in the activity of AOB, whereas NOBs are completely inhibited at 0.026–0.22 mg $HNO_2^-$N/L (Zhou et al., 2011). FA concentrations were below 6 mg/L except for C4 and C6, where maximum FA concentrations of 9 and 12.5 mg/L were calculated, respectively. FA concentrations were below 0.06 mg N/L at the end of all the tests. In this study, the FNA ranged from 0 to 0.00029 mg $HNO_2^-$N/L in batch tests for the determination of the ammonium oxidation rate. Hence, in our study, observing partial nitrification after long non-aeration conditions was mostly due to the slower recovery of nitrite-oxidizers from long anoxic conditions compared to ammonium-oxidizers (Miao et al., 2022). After low DO and a long anoxic period, AOB is less affected due to high oxygen affinity, while the activity of NOBs decreases. After the transition from an anoxic condition to an aerobic condition, nitrite accumulation can be observed due to the faster adaptation of AOBs to changing conditions and the recession in the activities of NOBs (Hou et al., 2017).

Theoretically, 7.14 mg of $CaCO_3$ alkalinity is consumed during the oxidation of 1 mg of $NH_4^+$-N (Scearce et al., 1980) and the consumed alkalinity in the batch tests were very close to the theoretical alkalinity requirement. For example, the theoretical and the measured alkalinity consumptions were 330 and 300 mg $CaCO_3$/L, respectively, at DO of 6 mg/L. In the aeration on/off time of 90/90 min, the theoretical and the measured alkalinitiy consumptions were 373 and 400 mg $CaCO_3$/L, respectively. COD removals ranged from 0 to 16% in the batch tests, except for C4 (the aeration on/off time of 15/15 min) at which it was 27%. In the batch test of C4, the nitrification efficiency was the lowest in the MBR (Yilmaz et al., 2023b). The low COD removals in the batch assays are attributed to the non-biodegradable nature of the organics present in the tests as no external organic matter was added in the batch tests, and the soluble COD came from the MBR liquor together with the biomass. It seems that the COD remaining the bioreactor after MBR treatment is not further degraded in the batch tests even after long-term aeration. Hence, the MBR may provide high performance and the remaining COD may be regarded as non-biodegradable (Yilmaz et al., 2023b).

### 5.3.2 Variations of specific ammonium oxidation rates in respirometric studies

The respirometric studies were performed with sludge from the MBR operated under varying conditions (Table 5.1). The results of respirometric studies were quite close to the batch tests (Table 5.3). The maximum Rsp were 13.6 and 11.7 mg $O_2$/(g-VSS.h) for at DO of 6 and 3 mg/L, respectively. In C3 and C4, testing sludge from the intermittent aeration phases, the maximum Rsp decreased to 6.8 and 6.0 mg $O_2$/(g-

VSS.h), respectively. However, with the increase of the aeration/on time, the maximum Rsp also increased, and it was calculated as 11.3, 13.0, and 16.0 mg $O_2$/(g-VSS.h) for C5, C6, and C7, respectively. In the last condition (C8) with the longest aeration off time, the maximum Rsp value decreased compared to previous periods and was 8.1 mg $O_2$/(g-VSS.h).

**Table 5.3 :** The specific ammonium oxidation rates in batch tests and respirometric studies.

| Conditions | Specific ammonium oxidation rate in the batch tests (mg N/(gVSS.h)) | The $R_{sp}$ in the respirometer (mg $O_2$/(gVSS.h)) | Consumed oxygen in the respirometer (mg/L) | Consumed oxygen calculated based on nitrite and nitrate produced (mg/L) |
|---|---|---|---|---|
| **C1 = DO of 6 mg/L** | 3.1 | 13.6 | 363 | 202 |
| **C2 = DO of 3 mg/L** | 2.6 | 11.7 | 340 | 222 |
| **C3 = 2 min on/2 min off** | 2.3 | 6.8 | 219 | 200 |
| **C4 = 15 min on/15 min off** | 1.3 | 6.0 | 295 | 269 |
| **C5 = 30 min on/30 min off** | 2.6 | 11.3 | 234 | 200 |
| **C6 = 60 min on/60 min off** | 2.1 | 13.0 | 237 | 235 |
| **C7 = 90 min on/90 min off** | 3.2 | 16.0 | 226 | 239 |
| **C8 = 90 min on/360 min off** | 5.4 | 8.1 | 170 | 181 |

In a study investigating the treatment of real municipal wastewater in an MBR operated under intermittent aeration, it was suggested that there was no clear relationship between the maximum specific oxygen uptake rate (SOUR) (5.7 – 26.57 mg $O_2$/(g-VSS.h) and the ratio of aerobic to anoxic time for heterotrophic biomass, and a reduction in SOUR was reported even at the highest aeration on/off ratio of 0.8 (Capodici et al., 2016). In the study investigating the treatment of simulated textile wastewater containing 1000 mg COD/L (COD:N:P mass ratio of 100:3.7:30) in aerobic granular sludge-SBR with intermittent aeration, SOUR was measured 40 and 16 mg $O_2$/(g-MLVSS.h) in the aeration start and end phase, respectively, at the aeration on/off time of 20/30 min (total 6 cycles) (without dye feeding). It was reported that SOURs were considerably lower than the ones measured in previous periods without intermittent aeration (Mata et al., 2015).

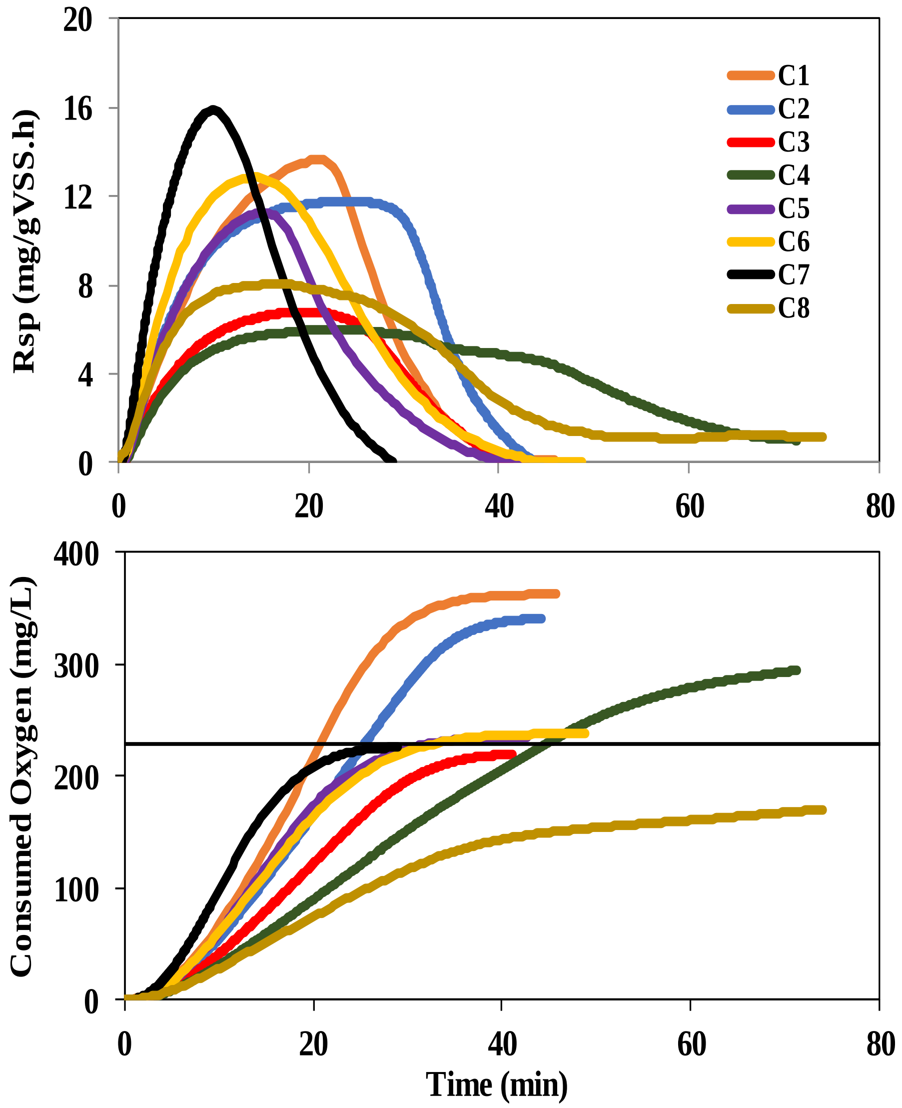


**Figure 5.2 :** $R_{sp}$ and CO variations in the respirometric studies for specific ammonium oxidation rates (The straight line in the bottom figure shows the theoretical oxygen consumption of 228.5 mg/L $O_2$).

Theoretically, 228.5 mg oxygen is needed for complete oxidation of 50 mg $NH_4^+$-N. A small part of the consumed ammonium is used for the synthesis of AOBs and NOBs, but this part was generally not taken into account in the calculations (Liu and Wang, 2012). However, there is a higher consumption due to endogenous respiration. The amounts of consumed oxygen in our study were approximately 363 mg/L and 340 mg/L, respectively, at DO of 6 and 3 mg/L. Higher consumption in these periods may be due to the higher endogenous respiration rates of aerobic heterotrophic bacteria. In C3 (the aeration on/off time of 2/2 min), the amount of oxygen consumed was measured as 219 mg/L, very close to the theoretical value. However, at 15/15 min, the CO increased again, and was measured as 295 mg/L. COs were 234, 237, and 226 mg/L in the C5, C6, and C7, respectively. However, CO decreased to 170 mg/L at 90/360 min (C8) (Table 5.3). The reduced oxygen consumption was due to nitrite accumulation, which was evidenced by the batch assays (Figure 5.2). The theoretical CO values calculated considering the nitrite and nitrate values produced at end of the batch tests are given in Table 5.3. These values were quite close to the data obtained in the respirometric tests especially when intermittent aeration was applied.

### 5.3.3 Variations of specific denitritation and denitrification rates

In all batch experiments, it was aimed to determine the effects of intermittent aeration on denitritation and denitrification rates. Selected results from the batch tests were presented in Figure 5.3 and 5.4. In addition, all the rates calculated for the batch test are given in detail in Table 5.4. The specific denitritation and denitrification rates were quite low in the tests conducted with continuously aerated sludge at DOs of 6 and 3 mg/L. The denitrification rate partially increased with increasing the nitrate from 25 to 50 mg $NO_3^-$-N/L (in R2), and it was 1.2 mg N/(g-VSS.h) at DO of 6 mg/L. However, the specific denitrification rate was 0.9 mg N/(g-VSS.h) at DO of 3 mg/L (in R2). Theoretically, the specific denitrification rate in sludge is expected to increase with decreasing DO concentration, although a partial decrease in the denitrification rate was observed. At the aeration on/off time of 2/2 min, the specific denitrification rate partially increased and reached approximately 1.1 mg N/(g-VSS.h) (in R2). By increasing the on/off time to 15/15 min, the specific denitrification rate rose up 2 mg N/(g-VSS.h) with the corresponding increase of 46% compared to the highest rate obtained in the previous period. Similar trends were observed in the nitrite reduction rate, and in the batch reactors operated with 50 mg/L $NO_2^-$-N, the specific denitritation rates were 1, 0.8, 0.6 and 1.7 mg N/(g-VSS.h) in C1, C2, C3, and C4, respectively. By increasing the aeration on/off time to 30/30 min, no significant change in the specific denitrification rates was observed. However, the specific denitritation rate increased to 2 and 2.1 mg N/(g-VSS.h) in R3 and R4 tests, respectively, for on/off time of 30/30 min (C5).

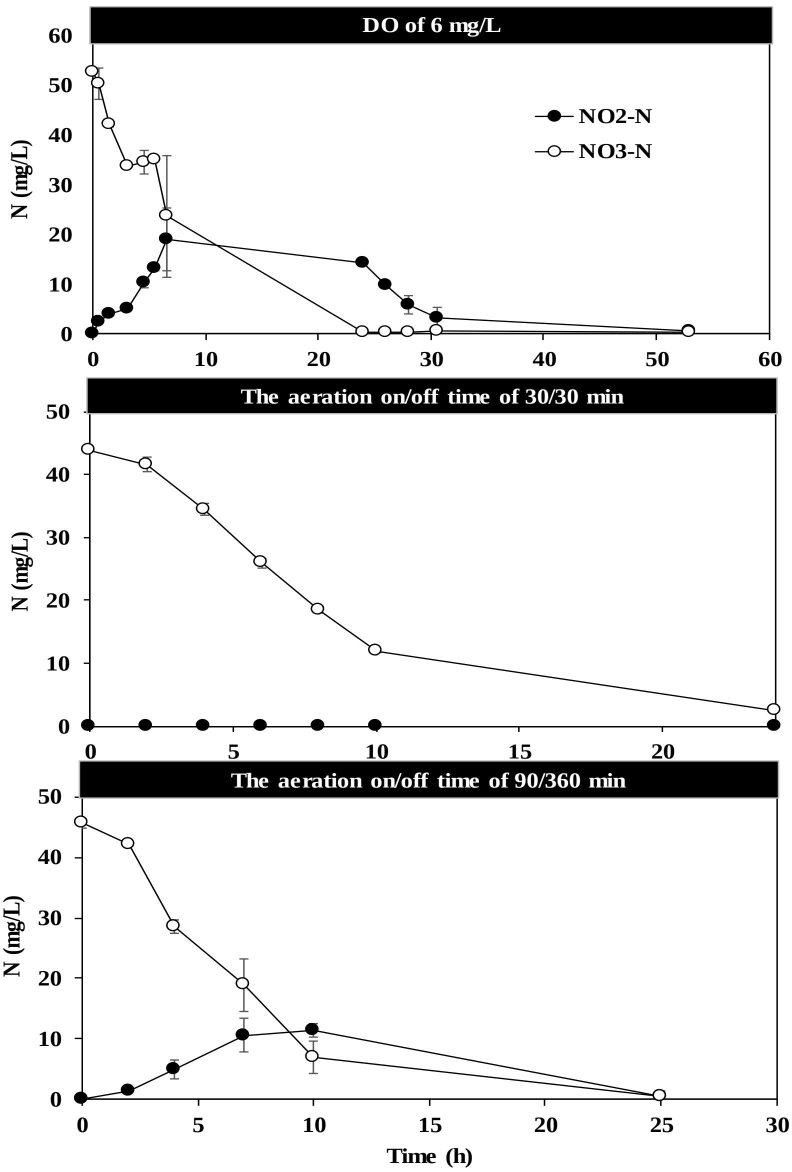


**Figure 5.3 :** Example results from batch tests for specific denitrification rates for R2. Condition 1 (upper figure), Condition 5 (middle figure), Condition 8 (bottom figure).

At the aeration on/off time of 60/60 min, the specific denitrification rates increased to 1.5 and 3.3 mg N/(g-VSS.h) in R1 and R2, respectively, while the specific denitritation rates were 2.1 and 3.2 mg N/(g-VSS.h) in R3 and R4. Although the increase in all rates was expected to continue by increasing the aeration off time to 90 min, the specific denitrification rates decreased partially, while the specific denitritation rates were similar to the previous tests. Similarly, the TN removal performance also decreased by increasing the on/off time to 90/90 min in the MBR, which was reported in our previous study (Yilmaz et al., 2023b). The specific denitrification rates increased significantly at the on/off time of 90/360 min, and they were 2.5 and 5.3 mg N/(g-

VSS.h) in R1 and R2, respectively. However, specific denitritation rates remained unchanged in R3 (50 mg $NO_2^-$-N/L), while it increased to 3.8 mg N/(g-VSS.h) in R4 (50 mg $NO_2^-$-N/L).

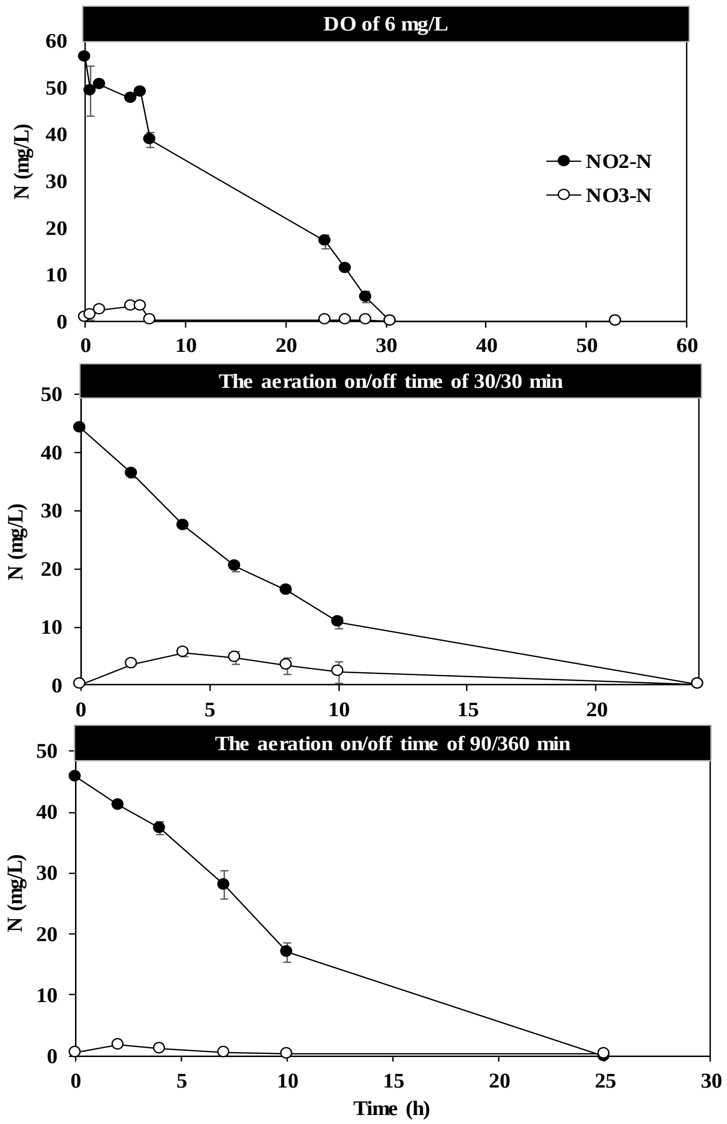


**Figure 5.4 :** Example results from batch tests for specific denitritation rates for R4. Condition 1 (upper figure), Condition 5 (middle figure), Condition 8 (bottom figure).

In a study investigating the treatment of textile wastewater in combined anaerobic and aerobic reactors, the specific denitrification rate in the system ranged from 0.4 to 1.2 mg $NO_3^-$/(mg-VSS.d) (Ahmed et al., 2005). In the study conducted by Cheng and Liu (2001) for anaerobically digested swine wastewater treatment with intermittent aeration, specific nitrification and denitrification rates were 2.79–3.70 mg $NO_3^-$-N/(g-VSS.h) and 0.59–1.03 mg $NO_3^-$-N/(g-VSS.h), respectively, at the aeration on/off time of 60/60 min. In an MBR system treating domestic wastewater, the highest denitrification rate was obtained as 2.68 mg $NO_3^-$-N/g-MLVSS/h at the aeration on/off time of 40/80 min, and it was higher than the relatively shorter aeration off times (Lim et al., 2007). In our study, the highest denitrification rate was 5.3 mg N/(g-VSS.h), and it was obtained for the sludge obtained from the longest aeration/off time.

In the experiments conducted with acetate as a carbon source in the literature, the denitrification rate generally varied between 3.09 mg N/(g-VSS.h) to 10.6 mg N/(g-VSS.h) depending on the operational conditions (reactor type, environmental factors and sludge properties) (Cherchi et al., 2009). The denitrification rate ranged from 0 to 0.7 mg N/(g-VSS.h) in batch tests conducted in the absence of organic matter, i.e. acetate. The specific denitrification rate of activated sludge by internal respiration has been reported as 0.2–0.6 mg $NO_3^-$-N/(g-VSS.h) in the literature (Collivignarelli et al., 2017; Foladori et al., 2010).

The highest denitritation rates were 2.4 and 3.8 mg N/(g-VSS.h) for R3 and R4 at the aeration on/off time of 90/360 min, respectively. In a study investigating the denitritation performance with different organic carbon sources, the maximum specific nitrite reduction rate were 0.25 g N/(g-VSS.d) for glycerol, while it decreased to between 0.13 and 0.17 g N/(g-VSS.d) for ethanol, landfill leachate and fermented primary sludge centrate in the SBR (Torà et al., 2011).

In the nitrite-based nitrogen removing processes, as the nitrite concentration increases, the denitritation rate increases, but FNA may form, which inhibits the process (Miao et al., 2017). FNA is a product that causes mutagenic effects by reacting with substrates, and its amount in the system increases with the decrease of pH (Zhou et al., 2010). Abeling and Seyfried (1992) reported that the inhibition threshold in the denitrification process of FNA was 0.13 mg $HNO_2^-$N/L (Abeling and Seyfried, 1992). Ma et al. (2010) observed that the nitrate reducing activity of biomass was limited by approximately 60% in an FNA of 0.01–0.025 mg $HNO_2^-$N/L, while completely ceased at over 0.2 mg $HNO_2^-$N/L (Ma et al., 2010). In this study, the highest FNA value was 0.00076 mg $HNO_2^-$N/L, which was considerably lower than the inhibition thresholds given in the literature.

**Table 5.4 :** The specific denitrification and denitritation rates in batch tests.

| Condition | Reactor No | $NO_3^-$-N (mg/L) | $NO_2^-$-N (mg/L) | Specific denitrification rate (mg N/(gVSS.h)) | Specific denitritation rate (mg N/(gVSS.h)) |
|---|---|---|---|---|---|
| C1 | **R1** | 25 | - | 1.1 ± 0.03 | - |
| | **R2** | 50 | - | 1.2 ± 0.01 | - |
| | **R3** | - | 25 | - | 0.5 ± 0.03 |
| | **R4** | - | 50 | - | 1 ± 0.04 |
| C2 | **R1** | 25 | - | 0.8 ± 0.04 | - |
| | **R2** | 50 | - | 0.9 ± 0.01 | - |
| | **R3** | - | 25 | - | 0.4 ± 0.001 |
| | **R4** | - | 50 | - | 0.8 ± 0.02 |
| C3 | **R1** | 25 | - | 0.5 ± 0.02 | - |
| | **R2** | 50 | - | 1.1 ± 0.02 | - |
| | **R3** | - | 25 | - | 0.5 ± 0.01 |
| | **R4** | - | 50 | - | 0.6 ± 0.003 |
| C4 | **R1** | 25 | - | 0.8 ± 0.01 | - |
| | **R2** | 50 | - | 2 ± 0.02 | - |
| | **R3** | - | 25 | - | 1.5 ± 0.01 |
| | **R4** | - | 50 | - | 1.7 ± 0.05 |
| C5 | **R1** | 25 | - | 1 ± 0.1 | - |
| | **R2** | 50 | - | 2 ± 0.02 | - |
| | **R3** | - | 25 | - | 2 ± 0.05 |
| | **R4** | - | 50 | - | 2.1 ± 0.06 |
| C6 | **R1** | 25 | - | 1.5 ± 0.07 | - |
| | **R2** | 50 | - | 3.3 ± 0.1 | - |
| | **R3** | - | 25 | -- | 2.1 ± 0.04 |
| | **R4** | - | 50 | - | 3.2 ± 0.004 |
| C7 | **R1** | 25 | - | 1.2 ± 0.1 | - |
| | **R2** | 50 | - | 2.8 ± 0.1 | - |
| | **R3** | - | 25 | - | 2.4 ± 0.2 |
| | **R4** | - | 50 | - | 3.2 ± 0.2 |
| C8 | **R1** | 25 | - | 2.5 ±0.02 | - |
| | **R2** | 50 | - | 5.3 ± 0.46 | - |
| | **R3** | - | 25 | - | 2.4 ± 0.01 |
| | **R4** | - | 50 | - | 3.8 ± 0.21 |

## 5.4 Conclusion

In this study, specific ammonium oxidation, denitritation and denitrification rates were investigated in batch assays for which sludge samples were obtained from a lab scale MBR treating textile wastewater at different dissolved oxygen concentrations and aeration on/off time. In the batch tests, the highest specific ammonium oxidation and denitrification rates were obtained at the aeration on/off time of 90/360 min. Compared to the period with continuous aeration, the specific ammonium oxidation, denitritation, and denitrification rates increased by approximately 1.8, 3.7 and 4.3 times, respectively, in the aeration on/off time of 90/360 min. Hence, the specific ammonium oxidation, denitritation and denitrification rates depend significantly on the aeration cycle on/off time, which should be optimized for the bioreactors operated under intermittent aeration conditions for the highest TN removal performance.

# 6. CONCLUSIONS AND RECOMMENDATIONS

Textile industry wastewater, which is produced in large amounts and has serious negative effects on human health and the ecosystem, needs to be treated with an economical and environmentally friendly approach before being discharged into the receiving environment. Within the scope of this thesis, two different process configurations were investigated in order to contribute to the circular economy during the treatment of a real textile wastewater and to minimize the negative environmental effects of the treated textile wastewater to be discharged. For this purpose, firstly, the treatment performance and organic matter recovery of textile wastewater in a high-rate MBR (including HF-UF membrane) operated at extremely low SRTs (5, 3, 2, 1, and 0.5 d) were examined (Chapter 3) in comparison with an MBR operated at conventional SRTs (30, 20, and 10 d) (Chapter 2). In the second process, the optimization of aeration on/off time for simultaneous nitrification-denitrification process in an intermittently aerated MBR system including an HF-UF membrane was investigated for nitrogen removal from textile wastewater (Chapter 4). Additionally, variations in the specific ammonium oxidation and denitrification rates of the MBR operated under different aeration patterns were examined using batch reactors and respirometry in detail (Chapter 5). Finally, the effects of changing operating conditions on the energy requirement for aeration, the amount of sludge produced, membrane filtration performances, and characterization of membrane foulants in both processes were also examined. The results obtained from this thesis study can be listed as follows:

- **Chapter 2:** While reducing SRT did not have a negative effect on COD and ammonium oxidation efficiencies, color removal efficiency decreased to 50% by reducing SRT to 10 d in a conventional MBR. Reducing the SRT resulted in a significant increase in $Y_{obs}$ values, a decrease in the energy requirement for aeration, and a limited impact on membrane fouling.
- **Chapter 3:** COD removal in the high-rate MBR was higher than 77% even at an SRT of 0.5 d and an HRT of 1.2 h. By reducing SRT to ≤ 1 d, slowly

biodegradable COD accumulated in the bulk. No correlation was observed between color removal and the tested SRTs. Nitrification performance stopped completely by reducing SRT and HRT to 2 d and 4.8 h, respectively. SS and VSS concentrations were quite close at all SRT values (except SRT of 1 d), where the SRT/HRT ratio was the same. The highest $Y_{obs}$ value was obtained at a SRT of 1 d (0.46 mg SS/mg COD). In addition, the SRT/HRT ratio significantly affected the amount of energy requirement for aeration, and as it was increased at the same SRT, the amount of energy requirement for aeration also increased. Membrane fouling profiles occurred more rapidly at SRTs of ≤ 1 d.

- **Chapter 4:** COD removal efficiencies remained above ≥84 at all aeration on/off times, but color removal did not show any correlation with all the conditions tested. Complete ammonium oxidation was achieved with at least aeration-on times of 30 min in the system. As aeration-off time increased in the system, higher TIN removal efficiencies were achieved. With intermittent aeration, sludge production and energy requirements for aeration were significantly reduced. However, long aeration-off times accelerated membrane fouling.
- **Chapter 5:** Until the aeration on/off times of 90/180 min and 90/360 min, the specific ammonium oxidation rate was significantly lower than that of the continuous aeration period. However, the specific ammonium oxidation rate increased significantly at the aeration on/off times of 90/360 min compared to the continuous aeration period (from 3.1 to 5.4 mg N/(gVSS.h)). Nitrite accumulation was observed in batch tests performed during long aeration-off periods. Through respirometric studies, oxygen consumption amounts close to theoretical values in ammonium oxidation were obtained, especially during periods of intermittent aeration. As the aeration off time increased, specific denitritation and denitrification rates increased, reaching 5.3 ± 0.46 and 3.8 ± 0.21 mg N/(gVSS.h). In addition, no inhibition of FA and FNA was observed during the all batch tests.

A general conclusion, considering all the studies conducted in the thesis, may also be summarized as follows:

- For the treatment of textile wastewater, the energy requirement for aeration was significantly reduced by decreasing the SRT from 30 d to 10 d, but the effect of the SRT reduction on membrane fouling profiles could not be fully revealed due to the low flux values in the conventional aerobic MBRs.
- Under high-rate conditions, especially at low SRT (0.5, 1 and 2 d) and HRT (1.2 and 2.4 h) combinations, the energy requirement for aeration decreased and COD accumulation occurred in the supernatant of the MBR. It is predicted that especially the accumulation of COD and the presence of fresh sludge in the system will increase the biomethane production potential. However, accelerated membrane fouling, especially at low SRT (1 and 0.5 d) and HRT (2.4 and 1.2 h) combinations, is the bottleneck of the process.
- For energy-neutral and energy-positive MBR treatment of textile wastewater, SRT should be ≤ 2 d.
- If energy consumption is not a concern, long SRTs (20-30 d) provide high performance with low membrane fouling.
- The intermittent aeration process is a very compact and energy efficient system for carbon and nitrogen removal from textile wastewater. The existing aerobic biological processes can be easily retrofitted to an intermittent aeration process, which can decrease energy consumption and increase nitrogen removal.

The following recommendations for future studies can be summarized as follows:

- New studies are needed to reveal the causes of membrane fouling and reduce membrane fouling in high-rate MBRs for energy-efficient textile wastewater treatment.
- It should be recommended to conduct pilot-scale studies in both processes in order to better observe operational outcomes.
- To complete the economic evaluation of high-rate MBR, the bioenergy production potential of the resulting mixed liquid containing high organic matter can be investigated in pilot-scale studies.

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# APPENDICES

**APPENDIX A:** Supplementary Materials for Effect of sludge retention time on the performance and sludge filtration characteristics of an aerobic membrane bioreactor treating textile wastewater (Chapter 2)

**APPENDIX B:** Supplementary Materials for Performances of a high-rate membrane bioreactor for energy-efficient treatment of textile wastewater (Chapter 3)

**APPENDIX C:** Supplementary Materials for Impact of aeration on/off duration on the performance of an intermittently aerated MBR treating real textile wastewater (Chapter 4)

**APPENDIX A :** Supplementary Materials for Effect of sludge retention time on the performance and sludge filtration characteristics of an aerobic membrane bioreactor treating textile wastewater (Chapter 2)

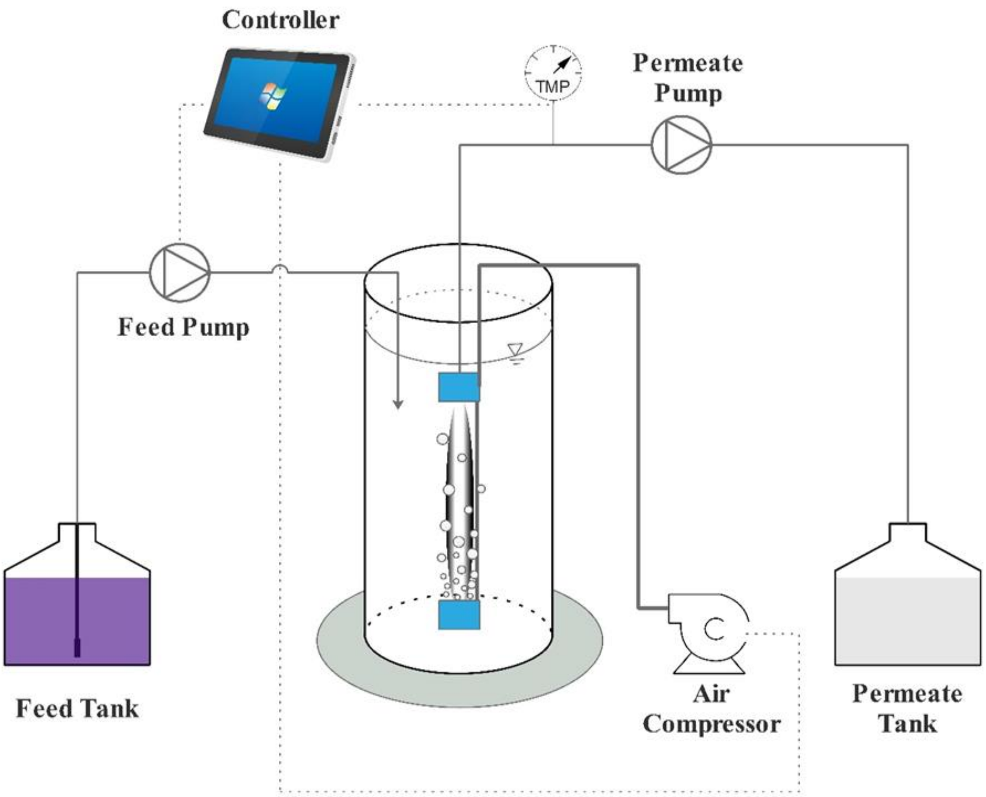


**Figure A.1 :** The schematic diagram of the aerobic MBRs.

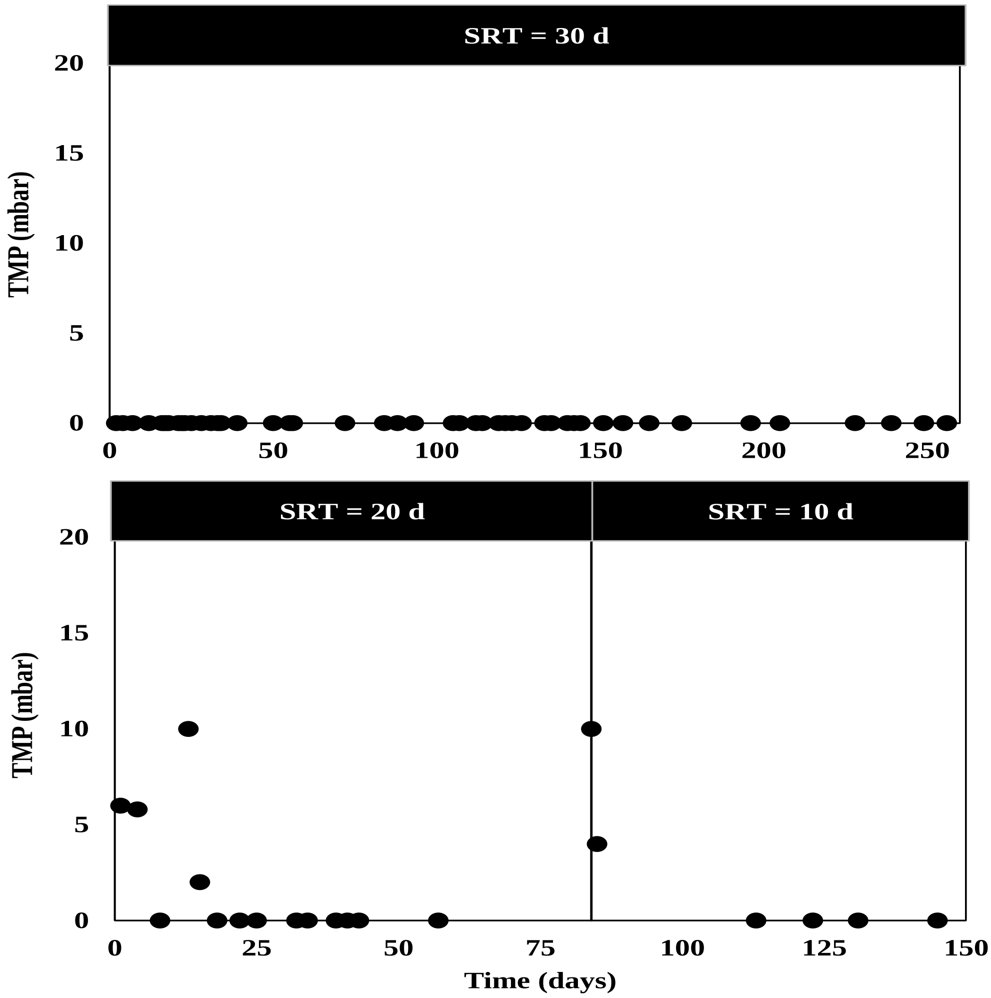


**Figure A.2 :** Variations of TMP in the aerobic MBRs throughout the study.

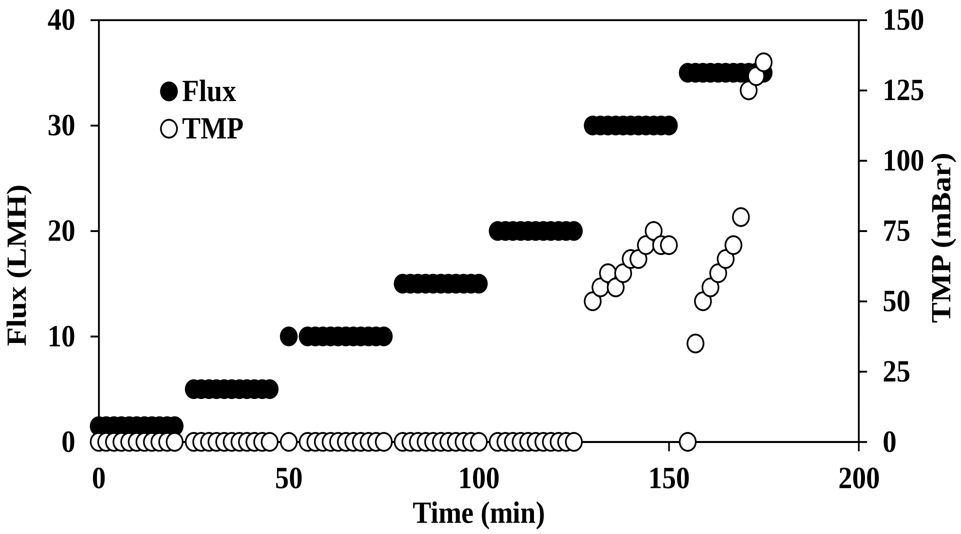


**Figure A.3 :** Variations of flux and TMP in batch test to determine critical flux.

**APPENDIX B :** Supplementary Materials for Performances of a high-rate membrane bioreactor for energy-efficient treatment of textile wastewater (Chapter 3)

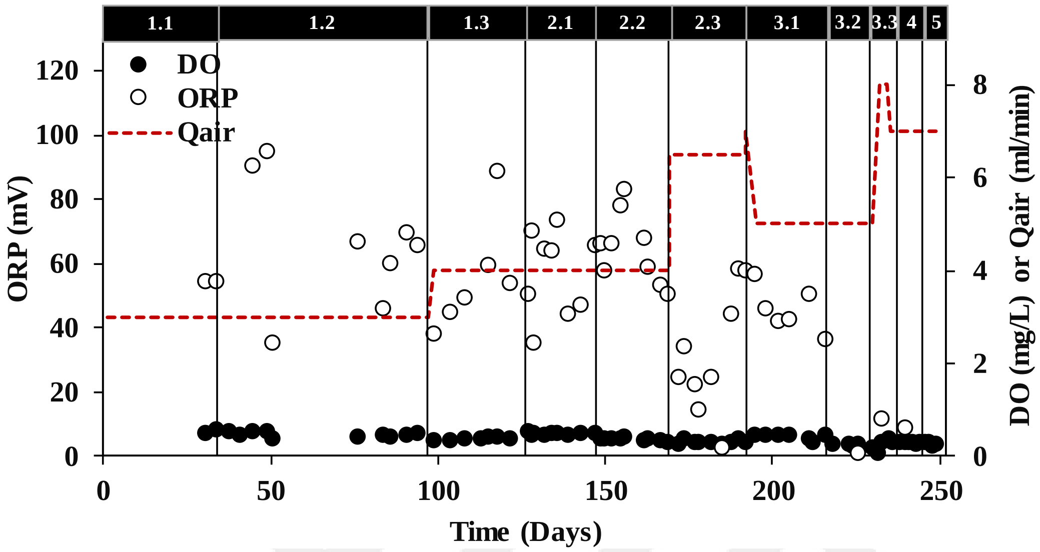


**Figure B.1 :** Variations of ORP, DO and airflow rate in the MBR at all SRTs.

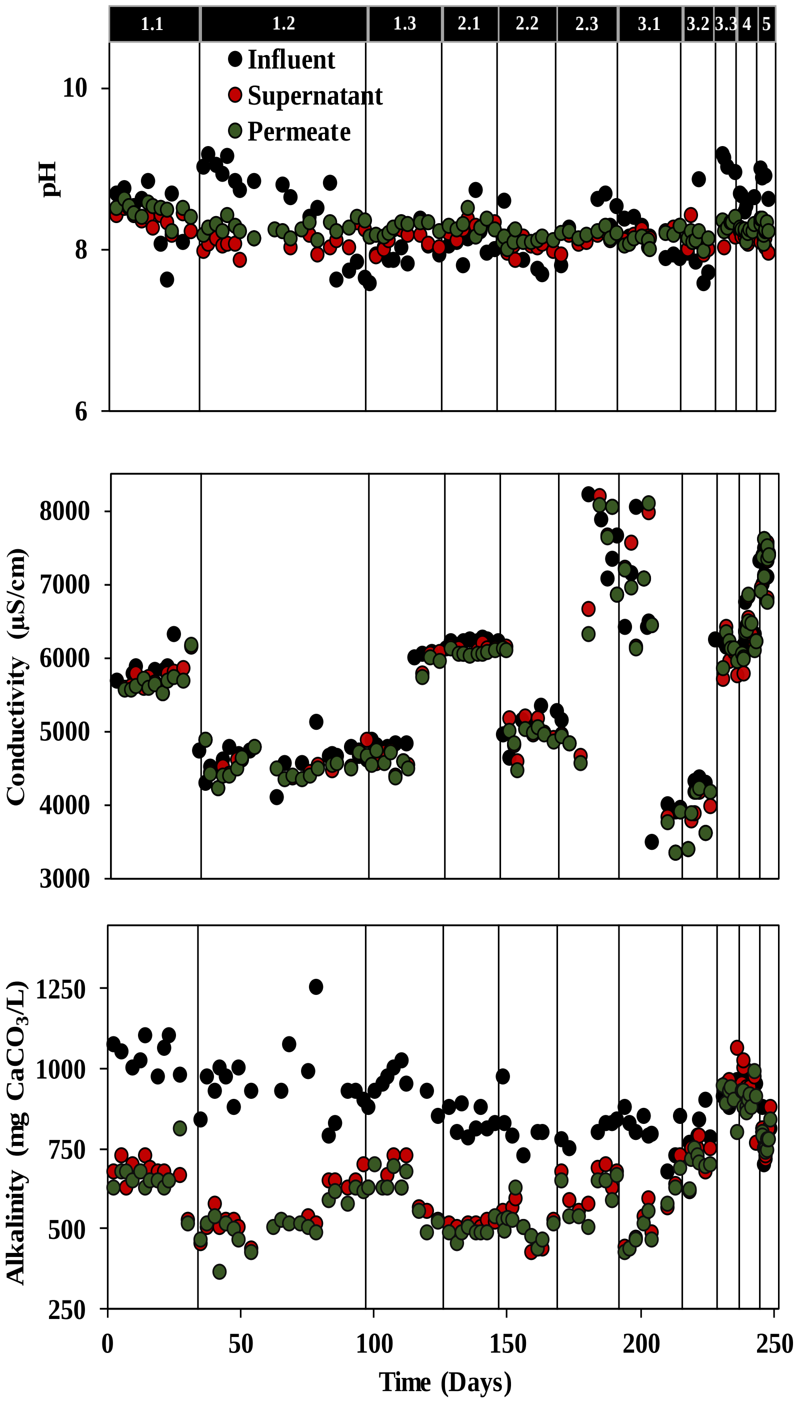


**Figure B.2 :** Variations of pH, conductivity and alkalinity in the MBR throughout the study.

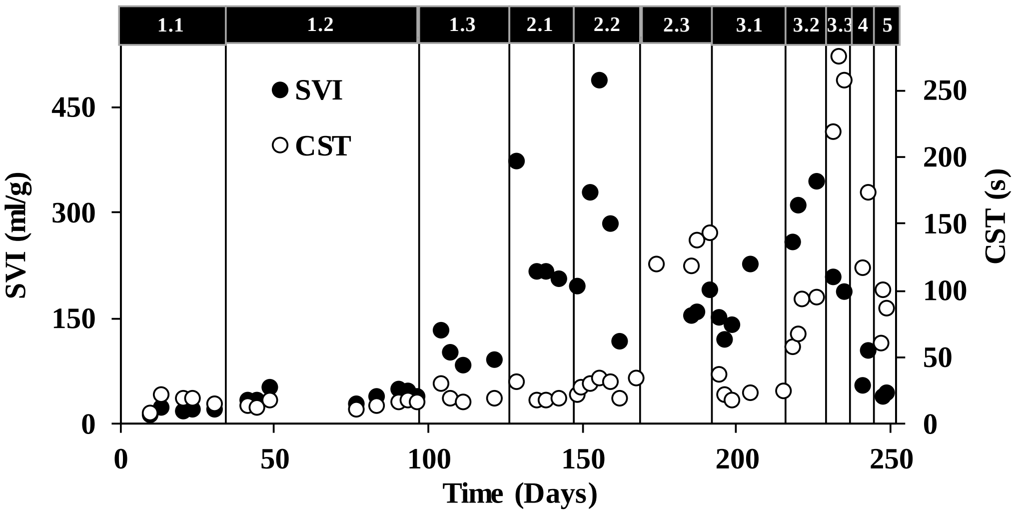


**Figure B.3 :** Variations of SVI and CST in the MBR throughout the study.

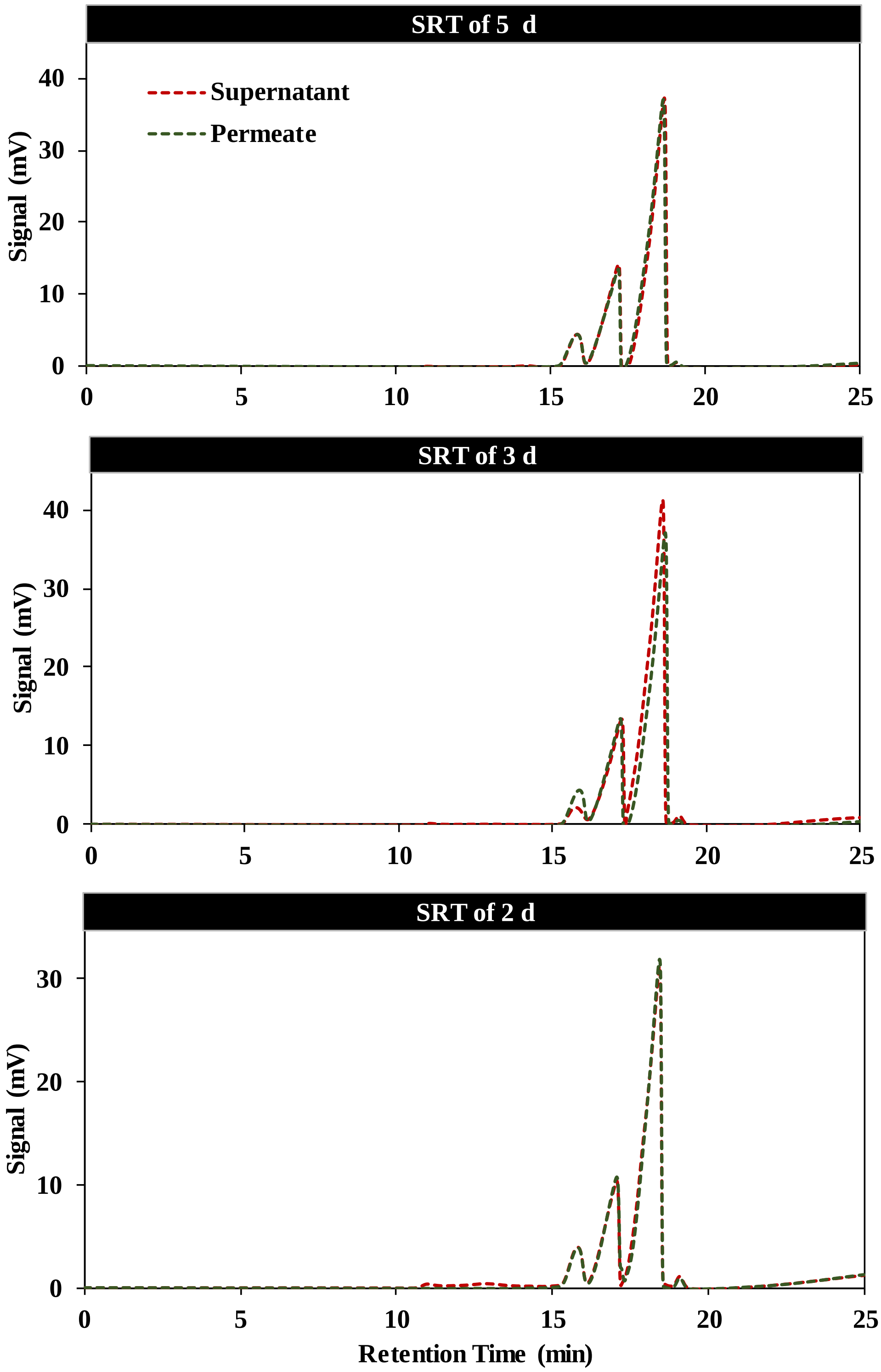


**Figure B.4 :** Impact of the low SRTs on the presence of organic molecules.

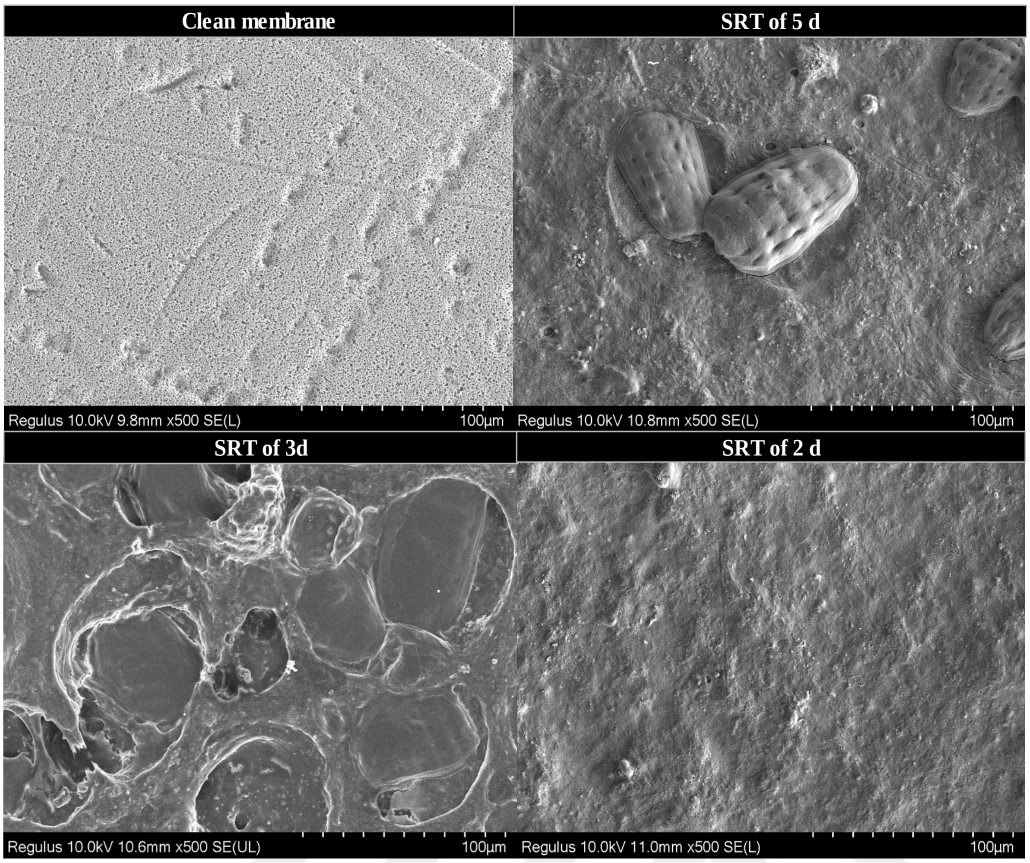


**Figure B.5 :** SEM photographs of cleaned membrane surface and fouled membrane surface at SRT of 5, 3 and 2 d (SRT/HRT ratio of 10).

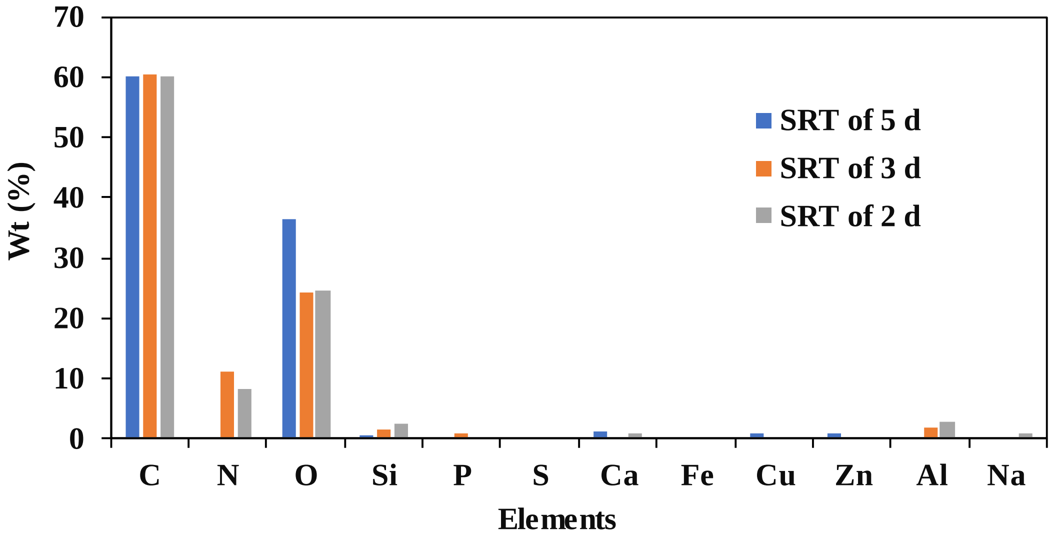


**Figure B.6 :** EDS results of fouled membrane surface at SRT of 5, 3 and 2 d (SRT/HRT ratio of 10).

**Table B.1 :** Characterization of the wastewater fed to the MBR throughout the study.

| Parameter | Unit | Average ± stdv |
|---|---|---|
| **pH** | **-** | 8.3 ± 0.5 |
| **Total COD** | **mg/L** | 834 ± 143 |
| **Soluble COD** | **mg/L** | 543 ± 133 |
| **Color** | **Pt-Co** | 1037± 407 |
| **TN** | **mg/L** | 60 ± 11 |
| **$N\text{-}NH_4^+$** | **mg/L** | 51 ± 11 |
| **TP** | **mg/L** | 3.7 ± 1 |
| **Alkalinity** | **mg $CaCO_3$/L** | 893 ± 105 |
| **Conductivity** | **µS/cm** | 5588 ± 1084 |
| **$N\text{-}NO_2^-$** | **mg/L** | ≤1 |
| **$N\text{-}NO_3^-$** | **mg/L** | ≤1 |

**Table B.2 :** Variations of the SS, VSS, CST, SVI, SRF, SF and viscosity in the high rate MBR throughout the study.

| SRT (d) | SRT/HRT ratio | SS (mg/L) | VSS (mg/L) | CST (s) | SVI (ml/g) | SRF (mg/kg) | SF (ml/min) | Viscosity (cP) |
|---|---|---|---|---|---|---|---|---|
| 5 | 5 | 1256 ± 370 | 1128 ± 270 | 16 ± 4 | 82 ± 39 | $7.6 \times 10^{14} \pm 1.4 \times 10^{14}$ | 1 ± 0.12 | 2 ± 0.05 |
| | 10 | 2729 ± 259 | 2484 ± 257 | 38 ± 8 | 207 ± 44 | $4.7 \times 10^{14} \pm 1.0 \times 10^{14}$ | 1 ± 0.13 | 2.6 ± 0.2 |
| | 20 | 5051 ± 388 | 4404 ± 257 | 100 ± 22 | 197 ± 19 | $5.9 \times 10^{14} \pm 1.1 \times 10^{14}$ | 0.6 ± 0.01 | 7.2 ± 0.3 |
| 3 | 5 | 1019 ± 68 | 771 ± 85 | 17 ±1 | 211 ± 5 | $5.4 \times 10^{14} \pm 1.7 \times 10^{14}$ | 1.8 ± 0.5 | 1.9 ± 0.1 |
| | 10 | 2131 ± 452 | 1937 ± 411 | 29 ±7 | 303 ± 153 | $6.1 \times 10^{14} \pm 2.3 \times 10^{14}$ | 1.1 ± 0.5 | 2.2 ± 0.3 |
| | 20 | 5916 ± 532 | 5144 ± 256 | 128 ±13 | 166 ± 20 | $5.2 \times 10^{14} \pm 5.9 \times 10^{13}$ | 0.7 ± 0.2 | 9.9 ± 1.6 |
| 2 | 5 | 1286 ± 283 | 1145 ± 216 | 21 ± 3 | 161 ± 58 | $9.1 \times 10^{14} \pm 5.9 \times 10^{13}$ | 1.2 ± 0.1 | 2.1 ± 0.002 |
| | 10 | 2942 ± 156 | 2682 ± 208 | 77 ± 19 | 303 ± 44 | $7.2 \times 10^{14} \pm 9.4 \times 10^{13}$ | 0.9 ± 0.01 | 3.2 ± 0.3 |
| | 20 | 5364 ± 678 | 4640 ± 598 | 250 ± 29 | 196 ± 15 | $1.3 \times 10^{15} \pm 1.7 \times 10^{14}$ | 0.6 ± 0.1 | 4.2 ± 0.6 |
| 1 | 10 | 2968 ± 586 | 2538 ± 481 | 144 ± 41 | 78 ± 35 | $3.0 \times 10^{15} \pm 5.2 \times 10^{14}$ | 0.44 ± 0.06 | 2.2 ± 0.1 |
| 0.5 | 10 | 1980 ± 607 | 1585 ± 327 | 82 ± 21 | 39 ± 4 | $1.15 \times 10^{16} \pm 4.9 \times 10^{15}$ | 0.33 ± 0.07 | 2.1 ± 0.1 |

**APPENDIX C : ** Supplementary Materials for Impact of aeration on/off duration on the performance of an intermittently aerated MBR treating real textile wastewater (Chapter 4)

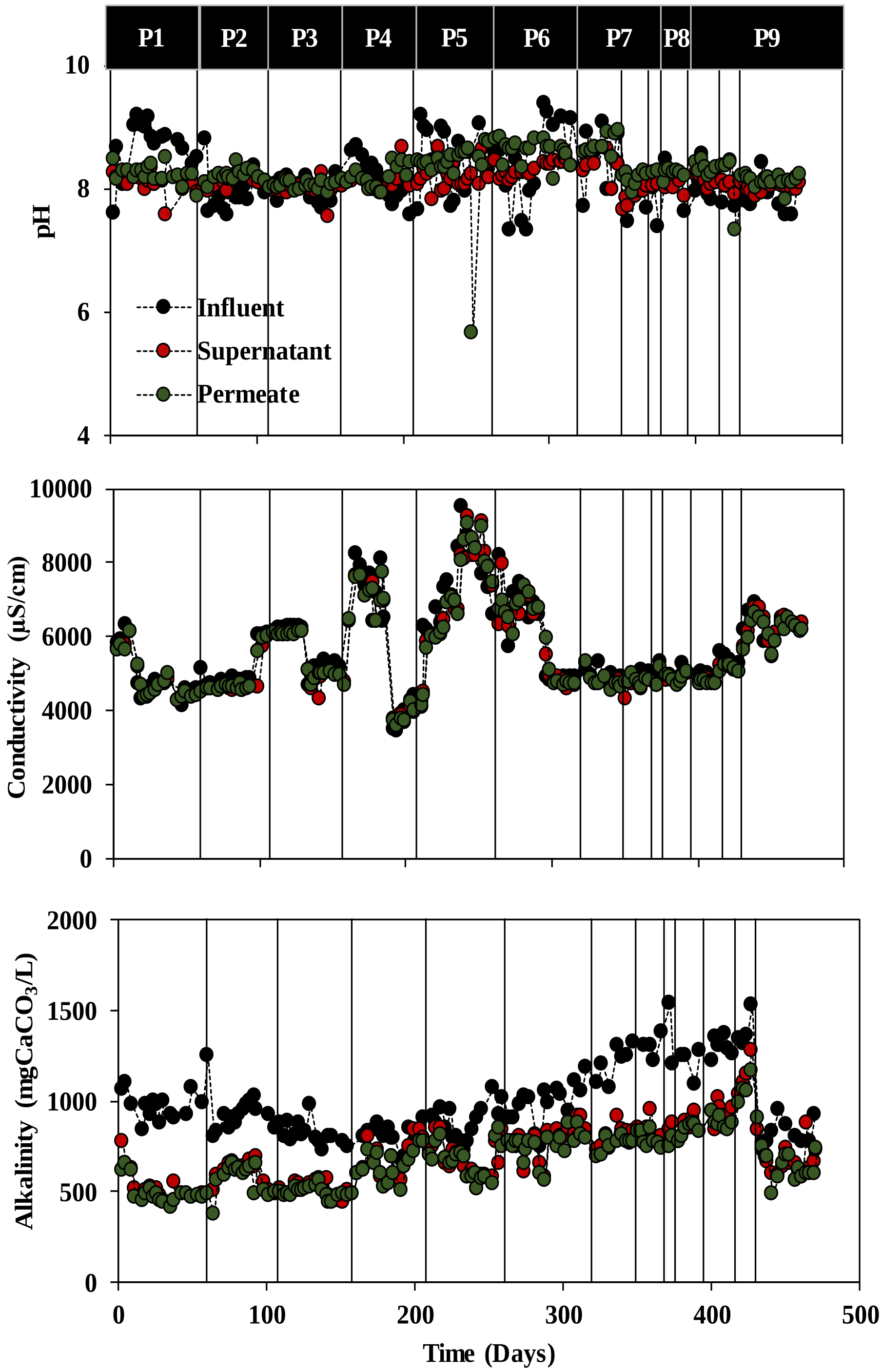


**Figure C.1 :** Variations of pH, conductivity and alkalinity in the MBR throughout the study.

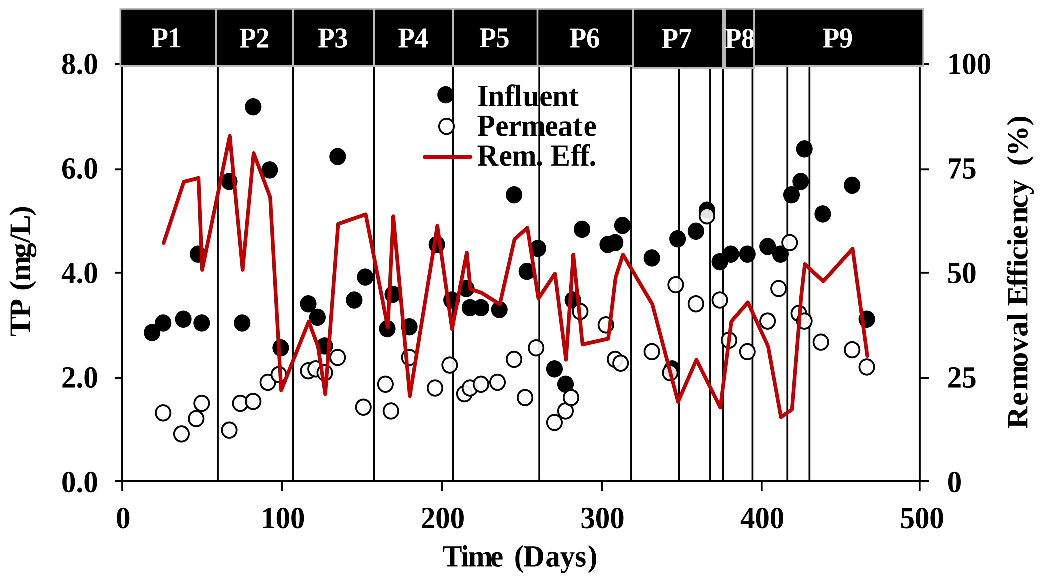


**Figure C.2 :** Variations of TP in the MBR throughout the study.

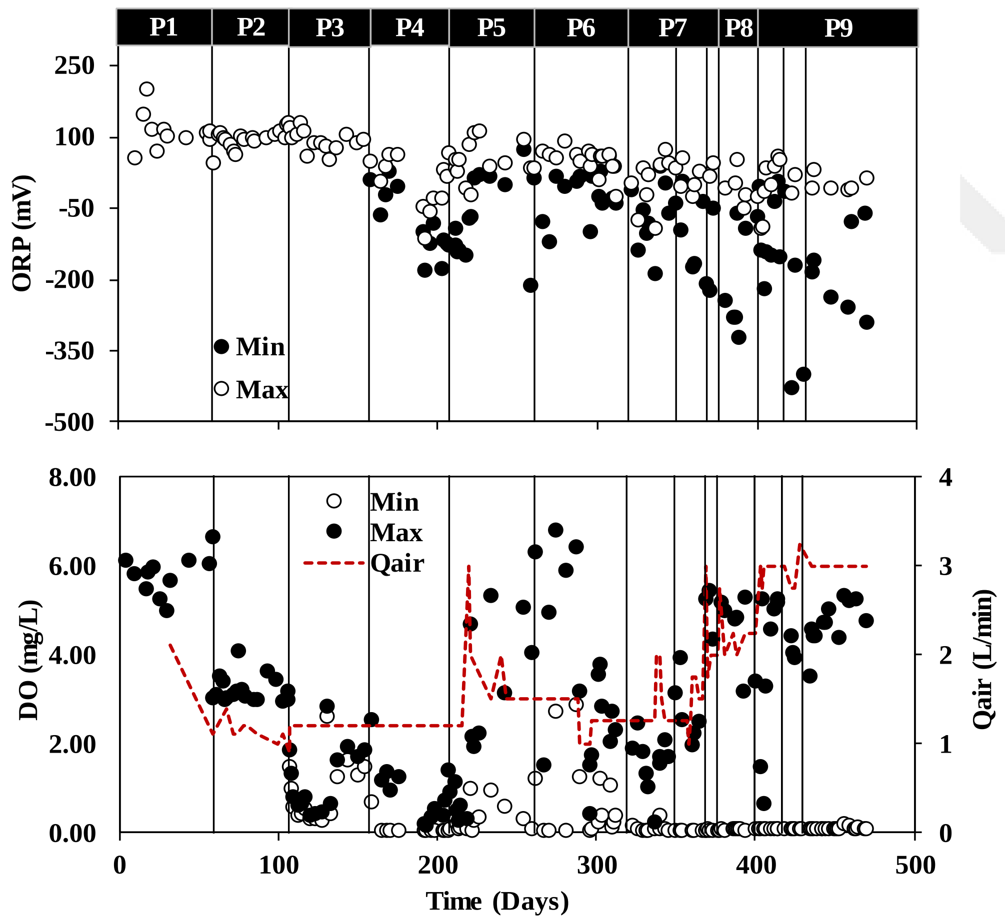


**Figure C.3 :** Variations of DO, ORP and airflow rate in the MBR throughout the study.

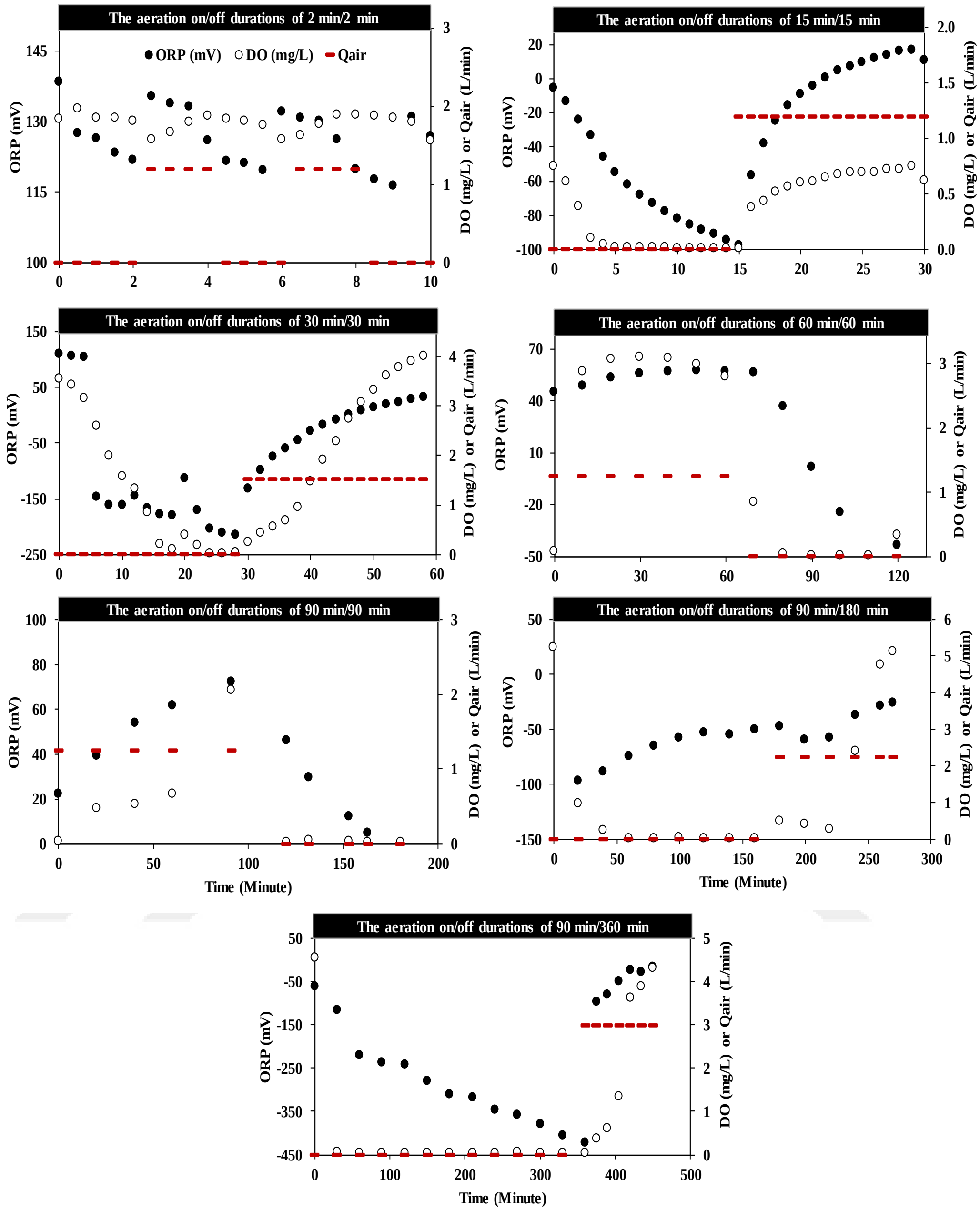


**Figure C.4 :** Variations of ORP, DO and airflow rate in the MBR for each period operated under intermittent aeration.

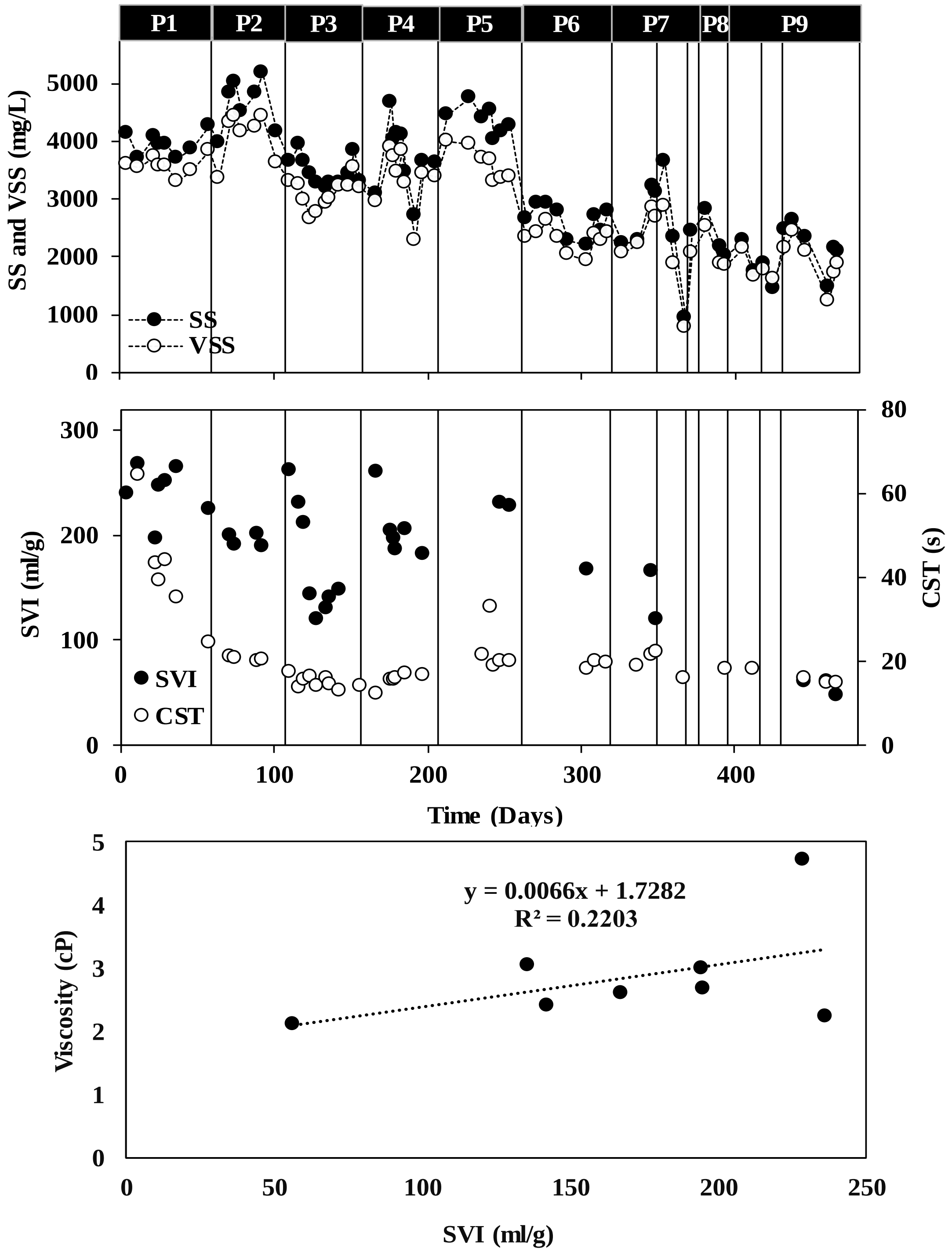


**Figure C.5 :** Variations of SS, VSS, SVI and CST in the MBR throughout the study.

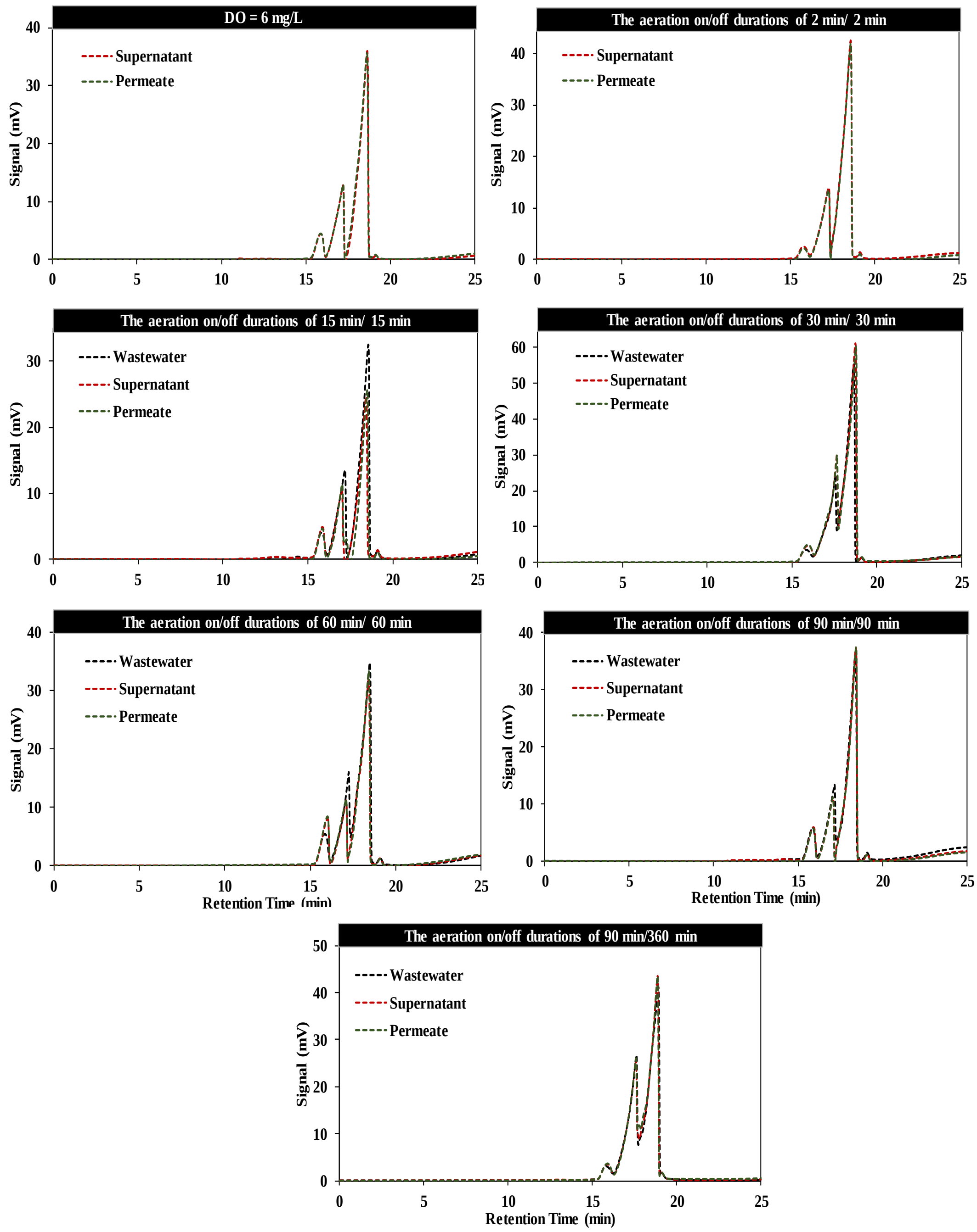


**Figure C.6 :** Impact of the different aeration on/off durations on the presence of organic molecules in the wastewater, supernatant and permeate.

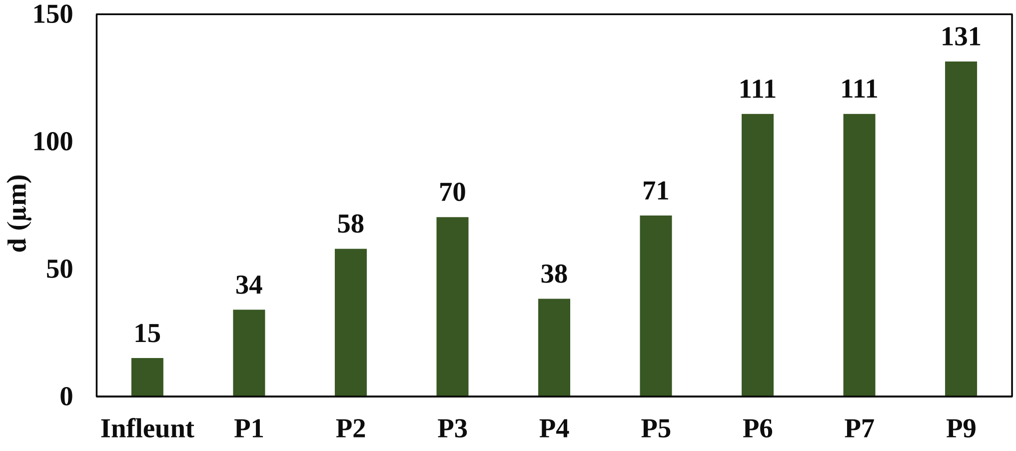


**Figure C.7 :** Impact of the different aeration on/off durations on the particle size distribution in the bioreactor.

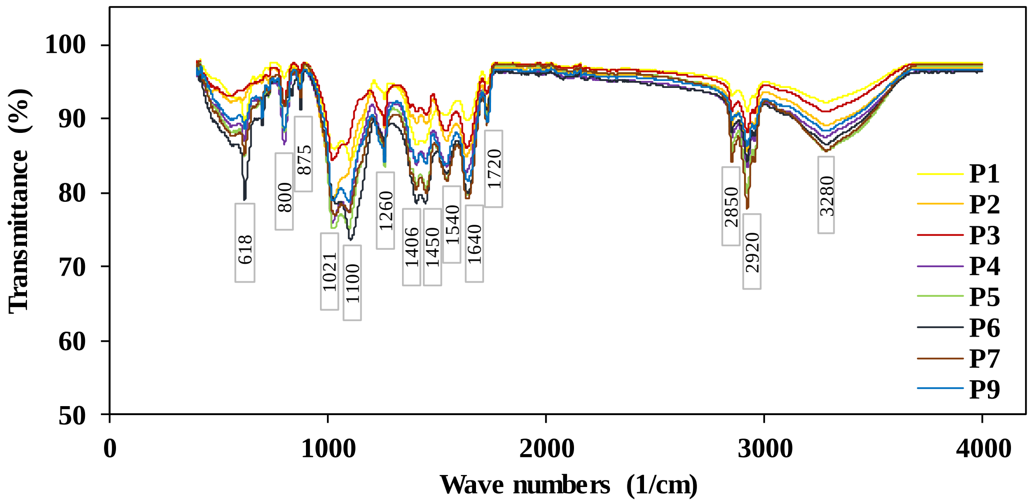


**Figure C.8 :** FT-IR spectra of the compounds in the bioreactor under the different aeration on/off durations.

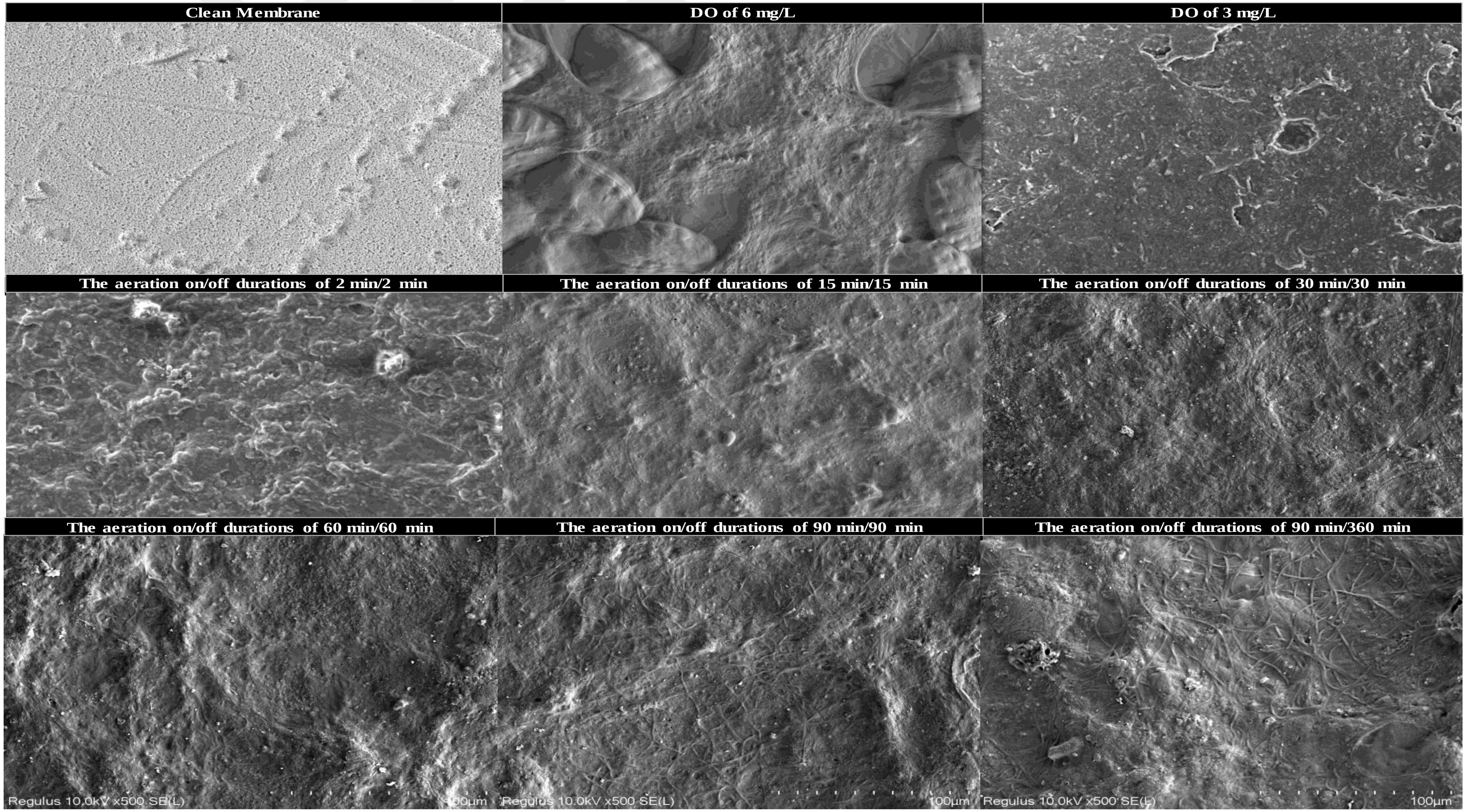


**Figure C.9 :** SEM photographs of clean and cake-deposited membrane surfaces in the each period.

**Table C.1 :** Characterization of the wastewater used for feeding the MBR throughout the study.

| Parameter | Unit | Average ± stdv |
|---|---|---|
| **pH** | **-** | 8.2 ± 0.5 |
| **Total COD** | **mg/L** | 793 ± 173 |
| **Soluble COD** | **mg/L** | 532 ± 144 |
| **Color** | **Pt-Co** | 1171 ± 458 |
| **TN** | **mg/L** | 65 ± 15 |
| **$N\text{-}NH_4^+$** | **mg/L** | 51 ± 15 |
| **TP** | **mg/L** | 4 ± 1 |
| **Alkalinity** | **mg $CaCO_3$/L** | 974 ± 195 |
| **Conductivity** | **µS/cm** | 5673 ± 1168 |
| **$N\text{-}NO_2^-$** | **mg/L** | ≤1 |
| **$N\text{-}NO_3^-$** | **mg/L** | ≤1 |

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**PUBLICATIONS, PRESENTATIONS AND PATENTS ON THE THESIS:**

**Articles**

- **Yilmaz, T., Demir, E. K., Başaran, S. T., Çokgör, E. U., and Sahinkaya, E.** 2023. Impact of aeration on/off duration on the performance of an intermittently aerated MBR treating real textile wastewater. *Journal of Water Process Engineering*, 54, 103886.
- **Yilmaz, T., Demir, E. K., Asik, G., Başaran, S. T., Çokgör, E. U., Sözen, S., and Sahinkaya, E.** 2023. Effect of sludge retention time on the performance and sludge filtration characteristics of an aerobic membrane bioreactor treating textile wastewater. *Journal of Water Process Engineering*, 51, 103390.

- **Yilmaz, T., Demir, E. K., Başaran, S. T., Çokgör, E. U., and Sahinkaya, E. 2023.** Specific ammonium oxidation and denitrification rates in an MBR treating real textile wastewater for simultaneous carbon and nitrogen removal. *Journal of Chemical Technology & Biotechnology.*

**Presentations**

- **Yilmaz, T., Demir, E. K., Başaran, S. T., Çokgör, E. U., and Sahinkaya, E.** 2022. Determination of specific nitrification rate with batch reactors and respirometric studies for an MBR operated under intermittent aeration conditions**,** 6th International Conference on Advances in Natural and Applied Sciences, October 11-13, 2022 Ağrı/Turkey.
- **Yilmaz, T., Demir, E. K., Başaran, S. T., Çokgör, E. U., and Sahinkaya, E.** 2022. Determination of specific denitrification and denitritation with batch reactors for an MBR operated under intermittent aeration conditions nitrate reduction in autotrophic and heterotrophic processes**,** 6th International Conference on Advances in Natural and Applied Sciences, October 11-13, 2022 Ağrı/Turkey.

**OTHER PUBLICATIONS, PRESENTATIONS AND PATENTS:**

**Articles**

- **Yilmaz, T., and Uçar, D.** (2023). Utilization of excess microorganisms as carbon and electron sources in the sulfate reduction process. *Journal of Chemical Technology & Biotechnology*.
- **Yenilmez, A. E., Ertul, S., Yilmaz, T., Ucar, D., Di Capua, F., and Sahinkaya, E.** (2023). Co-removal of P-nitrophenol and nitrate in sulfur-based autotrophic and methanol-fed heterotrophic denitrification bioreactors. *Journal of Environmental Chemical Engineering*, 111325.
- **Yilmaz, T., Yurtsever, A., Sahinkaya, E., and Ucar, D.** 2023. Perchlorate reduction in a thiosulfate-based denitrifying membrane bioreactor. *Biochemical Engineering Journal*, 109100.
- **Toprak, D., Yilmaz, T., and Uçar, D.** 2023. Effects of trace elements (Fe, Cu, Ni, Co and Mg) on biomethane production from paper mill wastewater. *Nordic Pulp & Paper Research Journal.*
- **Kılıç, M., Yılmaz, T., Partal, R., Şık, E., Doğan, Ö., Kitis, M., and Sahinkaya, E.** 2023. Nutrient recovery from anaerobic digester supernatant using a fluidized-bed reactor. *Journal of Water Process Engineering*, 54, 103950.
- **Yilmaz, T., Yildiz, M., Arzum Yapici, C. Ş., Annak, H. U., and Uçar, D**. 2023. Direct and sulfide mediated treatment of textile effluents in acetate and ethanol-fed upflow sulfidogenic bioreactors. *International Journal of Environmental Science and Technology*, 1-12.
- **Yılmaz, T., and Sahinkaya, E.** 2023. Performance of sulfur-based autotrophic denitrification process for nitrate removal from permeate of an MBR treating textile wastewater and concentrate of a real scale reverse osmosis process. *Journal of Environmental Management*, 326, 116827.

- **Toprak, D., Yilmaz, T., and Uçar, D.** 2023. Increasing biomethane production from paper industry wastewater with optimum trace element supplementation. *International Journal of Environmental Science and Technology*, 1-14.
- **Asik, G., Yilmaz, T., Di Capua, F., Ucar, D., Esposito, G., and Sahinkaya, E.** 2021. Sequential sulfur-based denitrification/denitritation and nanofiltration processes for drinking water treatment. *Journal of Environmental Management*, 295, 113083.
- **Ucar, D., Yilmaz, T., Di Capua, F., Esposito, G., and Sahinkaya, E.** 2020. Comparison of biogenic and chemical sulfur as electron donors for autotrophic denitrification in sulfur-fed membrane bioreactor (SMBR). *Bioresource Technology*, 299, 122574.
- **Yildiz, M., Yilmaz, T., Arzum, C. S., Yurtsever, A., Kaksonen, A. H., and Ucar, D**. 2019. Sulfate reduction in acetate-and ethanol-fed bioreactors: Acidic mine drainage treatment and selective metal recovery. *Minerals Engineering*, 133, 52-59.
- **Ucar, D., Sahinkaya, E., Yilmaz, T., and Cakmak, Y.** 2019. Simultaneous nitrate and perchlorate reduction in an elemental sulfur-based denitrifying membrane bioreactor. *International Biodeterioration & Biodegradation*, 144, 104741.
- **Yilmaz, T., Yucel, A., Cakmak, Y., Uyanik, S., Yurtsever, A., and Ucar, D.** 2019. Treatment of acidic mine drainage in up-flow sulfidogenic reactor: Metal recovery and the pH neutralization. *Journal of Water Process Engineering*, 32, 100916.
- **Toprak, D., Yılmaz, G., Yılmaz, T., Gülpınar, K., Çakmak, Y., Yucel, A., and Ucar, D.** 2019. Process optimization with multiple reactor systems of paper ındustry wastewater's biological treatability and biogas production. *Harran University Journal of Engineering*, 4(3), 29-37.
- **Erkan, D., Yılmaz, T., Yucel, A., Yılmaz, A., Tel, A. and Ucar D.** (2018). Applicability of micro scale hydroelectric power plants for energy recovery in wastewater treatment plants. *Harran University Journal of Engineering*, 3(1), 1-6.

**Presentations**

- **Yilmaz, T., Yucel, A., Sahinkaya, E. and Ucar, D.,** Bioreduction of nitrate and perchlorate in thiosulfate-based batch bioreactor, 5th International Conference on Engineering and Natural Sciences, June 12-16, 2019 Prague/ Czechia.
- **ucel, A., Yilmaz, T., Çakmak, Y and Ucar, D.,** Utilization of excess activated sludge and yeast residues as alternative substrate for sulfate reduction, 5th International Conference on Engineering and Natural Sciences, June 12-16, 2019 Prague/ Czechia.
- **Ucar, D., Yilmaz, T., Sahinkaya, E. and Jarmer, K.,** Simultaneous nitrate and perchlorate reduction in elemental sulfur fed autotrophic batch reactors, VI. International Gap Engineering Congress, October 23-25, 2018 Şanlıurfa/Turkey.
- **Ucar, D., Yilmaz, T., Yucel, A., and Sahinkaya, E.,** Perchlorate existence in the tap water collected from several cities in south east anatolia region, VI. International Gap Engineering Congress, October 23-25, 2018 Şanlıurfa/Turkey.

- **Ucar, D., Yilmaz, T., Erkan, D. Yildiz, M. and Arzum, C. S.,** Micro scale hydroelectric power plants for energy recovery in wastewater treatment plants, International Gap Renewable Energy and Energy Efficiency Congress, May 10-12, 2018 Şanlıurfa/Turkey.
- **Arzum, C. S., Yildiz, M., Erkan, D., Yilmaz, T., Bayar, S., Sahinkaya, E. and Ucar, D.,** Nitrate reduction in autotrophic and heterotrophic processes, 4th International Conference on Engineering and Natural Sciences, March 2-6, 2018 Kiev/Ukraine.
- **Erkan, D., Yilmaz, T., Yildiz, M., Arzum, C. S. and Ucar, D.,** The effect of sludge age on the operating parameters of activated sludge systems and the fate of daily produced sludge, 4th International Conference on Engineering and Natural Sciences, March 2-6, 2018 Kiev/Ukraine.
- **Yildiz, M., Arzum, C. S., Yilmaz, T., Erkan, D. and Ucar, D.,** Treatment of domestic wastewater by dynamic membrane bioreactors, 4th International Conference on Engineering and Natural Sciences, March 2-6, 2018 Kiev/Ukraine.
- **Ucar, D., Yilmaz, T., Yildiz, M., Yurtsever, A. and Arzum, C. S.,** Sulfate reduction in upflow anaerobic reactors and metal recovery, 3rd International Conference on Environmental Science and Technology, October 19-23, 2017 Budapest/Hungary.