MINIMIZATION OF EXCESS SLUDGE PRODUCTION IN THE TREATMENT OF MUNICIPAL WASTEWATERS BY USING MEMBRANE BIOREACTOR-ULTRASOUND COMBINED SYSTEMS

by

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To my precious family,

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ABSTRACT

The treatment and disposal of excess sludge has become the most challenging problem in wastewater treatment plants due to economic, environmental and legislative factors. Therefore, to develop strategies and technologies in order to minimize excess sludge production has become unavoidable. The purpose of the study was to investigate the effect of ultrasonic sludge disintegration on the minimization of excess sludge production in the treatment of municipal wastewater by using membrane bioreactors (MBRs). In the first stage of the study, in order to evaluate the effect of sonication on sludge properties, a series of batch experiments were performed. Ultrasonic disintegration increased the solubility of the organic matter and so the soluble chemical oxygen demand (SCOD), disintegration degree (DD_{COD}) and supernatant turbidity in sludge while it deteriorated the dewaterability and the rheological properties of sludge. In the second stage, membrane bioreactor-ultrasound (MBR-US) combined systems with different sludge sonication conditions were studied. In order to minimize the production of excess sludge in the MBR, ultrasonic disintegration was applied to a part of excess sludge and this sludge returned back into the MBR. When sonication was applied to a part of excess sludge daily, the effluent quality of MBR-US system was deteriorated. However, it was observed that the MBR-US system can tolerate the negative effects of returned disintegrated excess sludge when the sonication time interval increases. The results indicated that minimization of excess sludge production can be achieved in MBR-US system without any deterioration in the effluent quality. Furthermore the enhancement of nutrient removal is possible in MBR-US systems.

ÖZET

Atık çamurların arıtılması ve bertarafı, ekonomik, çevresel ve yasal faktörlerden dolayı, atıksu artıma tesislerinde en önemli problem haline gelmiştir. Bu yüzden, atık çamur oluşumunu minimize etmek amacıyla stratejiler ve teknolojiler geliştirmek kaçınılmaz olmuştur. Bu çalışmanın amacı, evsel atıksuların membran biyoreaktörler (MBR) ile arıtılması esnasında oluşan atık çamurların minimizasyonunda, ultrasonik çamur dezentegrasyonunun etkisini incelemektir. Çalışmanın ilk aşamasında, sonikasyonun çamur özellikleri üzerindeki etkisini değerlendirmek amacıyla, kesikli deneyler yürütülmüştür. Ultrasonik dezentegrasyon, organik maddelerin çözünürlüğünü ve böylece çamurun çözünebilir kimyasal oksijen ihtiyacını (ÇKOİ), dezentegrasyon derecesini ($DD_{KOİ}$) ve süpernatant (üst faz) bulanıklığını arttırken, çamurun reolojik özelliklerini ve susuzlaştırılabilirliğini olumsuz olarak etkilemiştir. İkinci aşamada, membran biyoreaktör-ultrases (MBR-US) birleşik sistemi farklı çamur sonikasyon koşullarıyla çalışılmıştır. MBR'daki atık çamur oluşumunu azaltmak için, reaktördeki çamurun bir kısmına ultrasonik dezentegrasyon uygulanmış ve bu çamur MBR sistemine geri konmuştur. Reaktördeki atık çamurun bir kısmına günlük olarak sonikasyon uygulandığında, MBR-US sisteminin çıkış kalitesi bozulmuştur. Ancak, sonikasyon süresi aralıkları arttırıldığında, MBR-US sisteminin, sisteme geri verilen dezentegre atık çamurun olumsuz etkilerini tolere edebileceği gözlenmiştir. Çalışmanın sonuçları göstermiştir ki, çıkış kalitesinde bir bozulma olmadan, MBR-US sisteminde atık çamur minimizasyonu sağlanabilir. Ayrıca, MBR-US sistemlerinde, nütrient gideriminin arttırılması mümkündür.

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LIST OF SYMBOLS/ABBREVIATIONS

Symbol	Explanation	Units used
BOD_5	5-day Biochemical Oxygen Demand	(mg/L)
COD	Chemical Oxygen Demand	(mg/L)
CST	Capillary suction time	(s)
DD	Disintegration Degree	(%)
DO	Dissolved Oxygen	(mg/L)
HRT	Hydraulic Retention Time	(day)
MLSS	Mixed Liquor Suspended Solids	(mg/L)
MLVSS	Mixed Liquor Volatile Suspended Solids	(mg/L)
NH_3-N	Ammonia Nitrogen	(mg/L)
NO_2^--N	Nitrite Nitrogen	(mg/L)
NO_3^--N	Nitrate Nitrogen	(mg/L)
SCOD	Soluble Chemical Oxygen Demand	(mg/L)
SE	Specific Energy	(kJ/kg TS)
SRT	Solids Retention Time	(day)
SV	Settling Velocity	(mm/h)
SVI	Sludge Volume Index	(mL/g)
TCOD	Total Chemical Oxygen Demand	(mg/L)
TKN	Total Kjeldahl Nitrogen	(mg/L)
TP	Total Phosphorus	(mg/L)
TS	Total Solids	(mg/L)
TVS	Total Volatile Solids	(mg/L)

Abbreviations	Explanation
CMBR	Control Membrane Bioreactor
CR	Control Reactor

MBRs	Membrane Bioreactors
MBR-SD	Membrane Bioreactor -Sludge Disintegration Combined System
MBR-US	Membrane Bioreactor-Ultrasound Combined System
R1	Reactor 1 (Sequential Batch Reactor-Ultrasound Combined System)
R2	Reactor 2 (Sequential Batch Reactor-Ultrasound Combined System)
SBRs	Sequential Batch Reactors
SBR-SD	Sequential Batch Reactor- Sludge Disintegration Combined System
SBR-US	Sequential Batch Reactor-Ultrasound Combined System
WAS	Waste Activated Sludge
WWTPs	Wastewater Treatment Plants

1. INTRODUCTION

The amount of wastewater and the treatment rate has been increasing worldwide causing a considerable increase in the excess sludge production. A large amount of sludge has been produced by aerobic biological wastewater treatment processes, including the widely used activated sludge process (Liu, 2003; Wei et al., 2003; Pérez-Elvira et al., 2006; Chu et al., 2009). Currently, the production of excess sludge from activated sludge process is one of the most important problems faced in wastewater treatment plants (Liu and Tay, 2001) because of economic, environmental and legislative factors.

The treatment and disposal of excess sludge in a biological wastewater treatment plant accounts for about 25-65% of the total operating cost of treatment plant (Pérez-Elvira et al., 2006; Mahmood and Elliot, 2006; Zhang et al., 2009; Chu et al., 2009; Uggetti et al., 2010). The main conventional methods for ultimate disposal of excess sludge (like land-filling, incineration etc.) may cause secondary pollution problems. For this reason, in many countries, legislation concerning land application of sludge is being tightened in order to prevent secondary pollution and health risks to man and livestock (Pérez-Elvira et al., 2006). Thereby, the problem of wastewater converts into a sludge disposal problem (Pérez-Elvira et al., 2006). All these factors are the considerable driving force to develop strategies and technologies in order to minimize excess sludge production.

In recent years, for the purpose of excess sludge minimization on biological wastewater treatment plants, several strategies and technologies have been developed (Pérez-Elvira et al., 2006). All of the technologies utilize one or more of three basic approaches to reduce excess sludge production: cell lysis, cyclic oxic environments, and long solids retention time (Roxburgh et al., 2006). Cell lysis (by disintegration methods using as pretreatments) and long solids retention time (provided by membrane bioreactors) are the mainly used approaches in minimization of excess sludge production.

The combination of the different processes can produce novel and more efficient methods for the minimization of excess sludge production (Liu and Tay, 2001). Excess

sludge production can be greatly minimized by partial disintegration of the returned sludge in an aerobic biological wastewater treatment process (Ramakrishna and Viraraghavan, 2005; Chu et al., 2009).

Recently, various disintegration methods have been investigated and developed as pretreatment processes for the purpose of excess sludge minimization. Mechanical, thermal, chemical and biological pretreatment techniques are mainly used in order to improve biological sludge degradation (Winter, 2002; Bougrier et al., 2005; Filibeli and Kaynak, 2006; Chu et al., 2009). These pretreatments cause the lysis or disintegration of sludge cells (Bougrier et al., 2005). Ozone (Yasui et al., 1996; Sakai et al., 1997; Moussavi et al., 2008), alkali (Li et al., 2008), fenton (Dewil et al., 2005; Tokumura et al., 2007; Tokumura et al., 2008), thermal hydrolysis (Bougrier et al., 2008), microwave (Wojciechowska, 2005) and ultrasound (Zhang et al., 2007; Zhang et al., 2009; Cao et al., 2006; Yin et al., 2006; Pham et al., 2009; Tiehm et al., 2001; Salsabil et al., 2009; He et al., 2011) are some of the successfully applied sludge disintegration methods. The effective physical disintegration of sludge can decrease the disadvantages of chemical oxidations; thereby ultrasonic technology has been preferred to use in many studies based on its distinct advantage, such as safe, clean and effective (Zhang et al., 2007; Cao et al., 2006).

In order to prevent excess sludge production during biological wastewater treatment, membrane bioreactor-sludge disintegration (MBR-SD) combined system has been introduced, where the disintegrated sludge is recycled to the bioreactor as a feed solution (Yoon, 2003). In the application of combined systems, together with the use of MBR, ozone (Song et al., 2003; He et al., 2006; Wang et al., 2008), ozone and alkali (Oh et al., 2007), fenton (Ming-he and Chao-hai, 2010) and ultrasound (Yoon et al., 2004) are some of the widely used sludge disintegration methods.

This study investigated the effect of ultrasonic sludge disintegration on the minimization of excess sludge production in the treatment of municipal wastewater by using membrane bioreactors (MBRs). First, the effect of ultrasonic disintegration on sludge properties was investigated. Then, MBR systems with different amounts of ultrasonically disintegrated excess sludge were operated in two cases in order to evaluate the effect of sludge sonication conditions on the sludge minimization and effluent quality.

2. THEORETICAL BACKGROUND

2.1. Sludge Production

The amount of wastewater being treated has been increasing worldwide as a result of gradually increasing world population. The activated sludge process is the most widely used biological wastewater treatment method in the world (Pérez-Elvira et al., 2006; Wei et al., 2003), e.g. over 90% of the municipal wastewater treatment plants use it as the core part of the treatment process (Liu, 2003). However, the activated sludge process results in the generation of a considerable amount of excess sludge that has to be treated and disposed of. Treatment and disposal of excess sludge in a biological wastewater treatment plant accounts for about 25-65% of the total operating cost of treatment plant (Pérez-Elvira et al., 2006; Mahmood and Elliot, 2006; Zhang et al., 2009; Uggetti et al., 2010).

The main conventional methods for ultimate disposal of excess sludge (like land-filling, incineration etc.) may cause secondary pollution problems. For this reason, in many countries, legislation concerning land application of sludge is being tightened in order to prevent secondary pollution and health risks to man and livestock (Pérez-Elvira et al., 2006). Thereby, the problem of wastewater converts into a sludge disposal problem (Pérez-Elvira et al., 2006).

Currently, the production of excess sludge from activated sludge process is one of the serious problems encountered in aerobic wastewater treatment (Liu and Tay, 2001) because of economic, environmental and legislative factors. Hence, the treatment and disposal of excess sludge has become the most challenging problem in wastewater treatment plants (WWTPs). Therefore, to develop strategies and technologies for minimization of excess sludge production in WWTPs has received considerable attention and interest from both academic and engineering fields.

2.1.1. The Types of Sludge Produced in WWTPs

The type of sludge is important to correctly evaluate the potential of a sludge reduction route. To obtain high sludge reduction, it is necessary to act on inert fractions which often account for the majority of sludge mass, especially in the case of biological processes with long SRT. The conversion of inert particulates to a biodegradable substrate significantly affects sludge reduction efficiency. If this conversion process is zero in a certain technique, complete sludge reduction is obviously not possible.

The types of sludge produced in a WWTP (in the wastewater handling units) are primary, secondary and chemical sludge (Turovskiy and Mathai, 2006).

2.1.1.1. Primary Sludge. This type of sludge is produced by settleable solids removed from raw wastewater in primary settling; characterized by high putrescibility and good dewaterability when compared to biological sludge. TS content in primary sludge is in the range 2-7% (Turovskiy and Mathai, 2006). Since primary sludge is already readily degradable, pretreatment may be less effective (Carrère et al., 2010).

2.1.1.2. Secondary Sludge. This type of sludge is also called biological sludge. It is produced by biological processes such as activated sludge or biofilm systems. It contains microorganisms grown on biodegradable matter (either soluble or particulate), endogenous residue and inert solids not removed in the primary settling (where a primary settler is present) or entering with the raw wastewater (where no primary settler is present). TS content in secondary sludge is in the range 0.5-1.5% (Turovskiy and Mathai, 2006).

Biological sludge (waste activated sludge) has relatively low degradability, especially at long sludge ages. In addition, waste activated sludge is generally hydrolysis limited, and can be enhanced by improved rates. Thus, biological sludge pretreatment is often used in preference to primary sludge pretreatment, except for specific circumstances (Carrère et al., 2010).

2.1.1.3. Chemical Sludge. This type of sludge is produced by precipitation of specific substances (i.e. phosphorus) or suspended solids (Turovskiy and Mathai, 2006).

2.1.2. The Processes Leading to Sludge Production

Organic matter is oxidized by heterotrophic microorganisms to produce H_2O and CO_2 in the process known as c*atabolism*. This process requires the availability of an electron acceptor -which may be oxygen or nitrate- and lead to the production of energy as ATP. This energy is then used by microorganisms to grow forming new cellular biomass and to guarantee maintenance functions (such as the renewal of cellular constituents, maintenance of osmotic pressure, nutrient transport etc.) in the process called *anabolism*.

Simultaneously biological decay of cellular biomass occurs, which creates two fractions: biodegradable particulate COD and endogenous residue considered as inert particulate COD which accumulates in the system. The biodegradable particulate COD fraction is subjected to hydrolysis process and is further oxidized to generate new cellular biomass (*cryptic growth*), while the endogenous residue (8-20%) remains and accumulates in the sludge. A simplified scheme of these processes leading to sludge production in the biological treatment of influent wastewater is indicated in Figure 2.1.

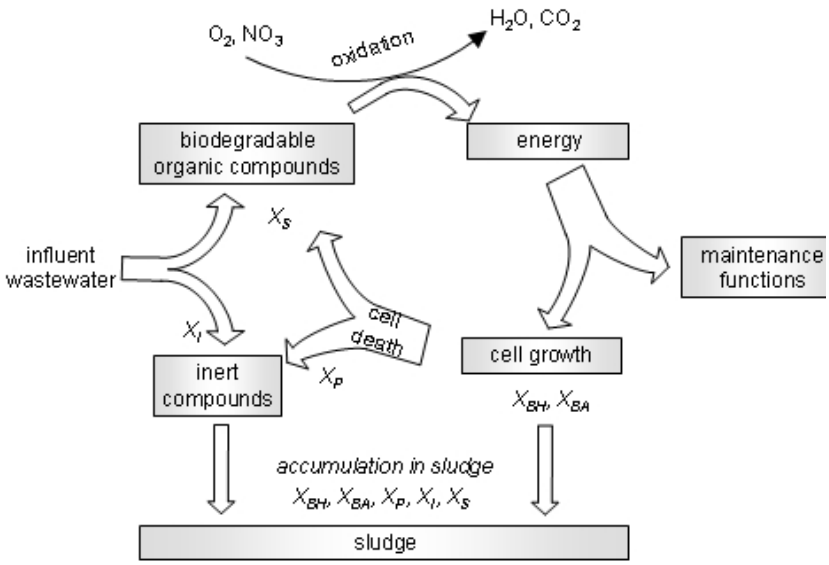


Figure 2.1. The scheme of the processes leading to sludge production in the biological treatment of wastewater.
(http://www.iwawaterwiki.org/xwiki/bin/view/Articles/SludgeProduction)

Daily production of excess sludge from conventional activated sludge process is approximately 15-100 L/kg BOD_5 removed, in which over 95% is water (Liu and Tay, 2001). The volume of sludge produced in a WWTP is only about 1% (dewatered sludge is 0.5‰) of the volume of influent wastewater to be treated. To manage WWTPs effectively and efficiently, it is absolutely necessary to extract waste sludge, including inert solids and excess biomass, in order to prevent their accumulation within the system (http://www.iwawaterwiki.org/xwiki/bin/view/Articles/SludgeProduction).

2.2. Sludge Minimization Strategies and Technologies

Sludge minimization technologies have been available for several decades; however recent developments have brought some sludge minimization technologies to the forefront. All of the technologies utilize one or more of three basic approaches to minimize the amount of waste activated sludge produced by an activated sludge process: cell lysis, cyclic oxic environments, and long solids retention time (Roxburgh et al., 2006).

Sludge minimization refers generally to the optimum reduction of the mass of sludge or biosolids produced at a wastewater treatment facility. A fundamental understanding of the basic mechanisms available to minimize sludge production is needed prior to evaluating the various sludge minimization technologies (Roxburgh et al., 2006).

In recent years, for the purpose of excess sludge minimization on biological wastewater treatment plants, several strategies have been developed. Pérez-Elvira et al. (2006) preferred to categorize the different processes according to the place of the plant where the minimization takes place. Three main strategies are identified as followings (Pérez-Elvira, 2006);

(1) Processes in the water line
   a. Processes that reduce the yield coefficient
      - Lysis cryptic growth (Chemical oxidation such as Ozonation and Chlorination, Integration of chemical and heat treatment, High purity oxygen process, Enzymatic reactions)
      - Maintenance metabolism (**Membrane bioreactor**)

- Uncoupling metabolism (Chemical uncoupler, Oxic-settling-anaerobic process)
- Predation on bacteria (Two-stage system, Oligochaetes)

b. Processes with low yield coefficient
- Anaerobic/aerobic systems

(2) Processes in the sludge line

a. Pre-treatment processes
- Physical pre-treatments (*Cavitation* such as High pressure homogenizer and **Ultrasonic homogenizers**, *Thermal* such as Thermal hydrolysis, Freezing and thawing, *Mechanical* such as Impact grinding, Stirred ball mills and High performance pulse technique, *Radiation*)
- Chemical pre-treatments (Acid or alkaline hydrolysis, Pre-treatment using ozone)
- Biological pre-treatments
- Combined pre-treatments (Combination of thermal, decompression and shear forces and Chemically enhanced thermal hydrolysis)

b. Modified anaerobic digestion processes
- Two-stage anaerobic digestion
- Temperature phased anaerobic digestion
- Anoxic Gas Flotation (AGF)

(3) Sludge removal processes

a. Incineration
b. Gasification and pyrolysis
c. Wet Air Oxidation (WAO)
d. Supercritical water oxidation (SCWO)

The strategies for sludge minimization should be evaluated and chosen for practical application using costs analysis and assessment of environmental impact. Employing any strategy for reducing sludge production may have an impact on microbial community in biological wastewater treatment processes. This impact may influence the sludge characteristics and the quality of effluent (Wei et al., 2003).

Pérez-Elvira (2006) noted that the idea in the first strategy (processes in the water line) is to reduce sludge production in the wastewater treatment rather than the post-treatment of sludge after generation. The aim in the other processes which are in the sludge line is to reduce the final stream of sludge to be disposed of. In these type processes the reduction of excess sludge production is occurred by enhanced treatment of the sludge. The last technologies (processes in the final waste line) aim to treat the sludge produced to get a final stable, dewatered and pathogen free residue. They do not represent a minimization strategy, but a post-treatment to dispose of the sewage solids.

The ideal way to solve the sludge-associated problems is to reduce sludge production in the wastewater treatment process rather than the post-treatment of the sludge after generation (Liu and Tay, 2001; Liu, 2003; Wei et al., 2003; Ramakrishna and Viraraghavan, 2005).

The various mechanisms exploited in the sludge minimization techniques are identified and schematically indicated in the Figure 2.2.

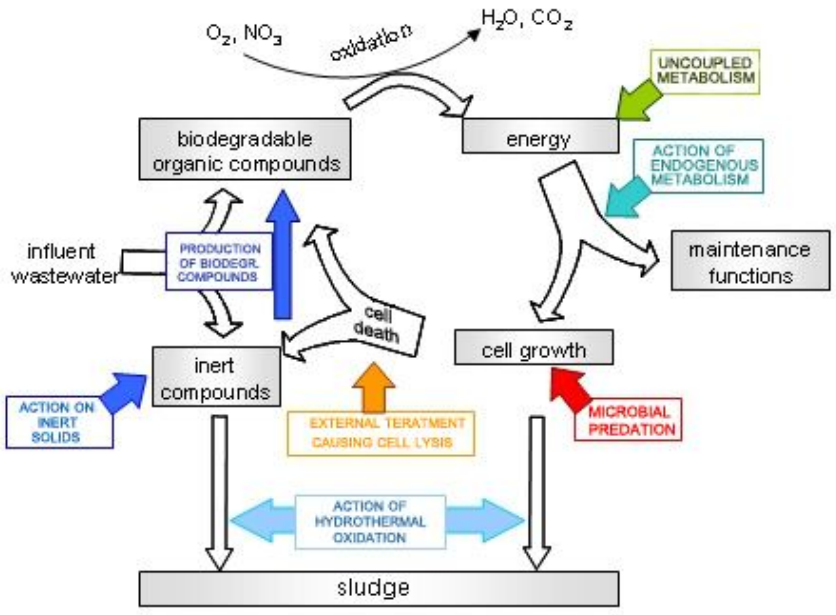


Figure 2.2. Mechanisms in the sludge minimization techniques.
(http://www.iwawaterwiki.org/xwiki/bin/view/Articles/SludgeProduction)

2.3. Membrane Bioreactors (MBRs)

Membrane bioreactors (MBRs) have become a more and more attractive technology in the wastewater treatment field in consequence of the development of membrane technology and the decrease of the membrane cost in the competitive environment. Therefore, the membrane bioreactor (MBR) can no longer be considered as a novel process. The MBR process was introduced by the late 1960s, as soon as commercial scale ultrafiltration (UF) and microfiltration (MF) membranes were available (Le-Clech et al., 2006).

These first type MBRs were designed with the separation device located external to the reactor and they had high operating cost due to power consumption of recirculation pumps. The lower operating cost obtained with the submerged configuration along with the steady decrease in the membrane cost encouraged an exponential increase in MBR plant installations from the mid 1990s (Le-Clech et al., 2006; Judd, 2008). Thereby, worldwide MBR inventory in centralized municipal wastewater applications has increased considerably since 1995, and the volume of wastewater treated by MBR plants is estimated to be growing by 20% per year (Le-Clech et al., 2005).

A recent review indicated the market value of MBR technology to be approximately US$217 million in 2005, rising at an average annual growth rate of 10.9% – significantly faster than other advanced wastewater treatment technologies (Judd, 2008; Drews, 2010).

The technology is becoming more cost-effective as membrane and membrane process costs continue to fall and environmental regulations become increasingly more stringent. It is estimated that the market is currently doubling every seven years (Judd, 2008; Drews, 2010), and will be worth a projected US$360 million by 2010 (Judd, 2008).

Membrane bioreactors are composed of two primary parts: the biological unit responsible for the biodegradation of the waste compounds and the membrane module for the physical separation of the treated water from mixed liquor (Cicek, 2003; Judd, 2006).

2.3.1. Membrane Separation Processes

A membrane as applied to water and wastewater treatment is simply a material that allows some physical or chemical components to pass more readily through it than others. The four key membrane separation processes in which water forms the permeate product are reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF) and microfiltration (MF) (Judd, 2006). Figure 2.3 illustrates the ranges of membrane based on separations.

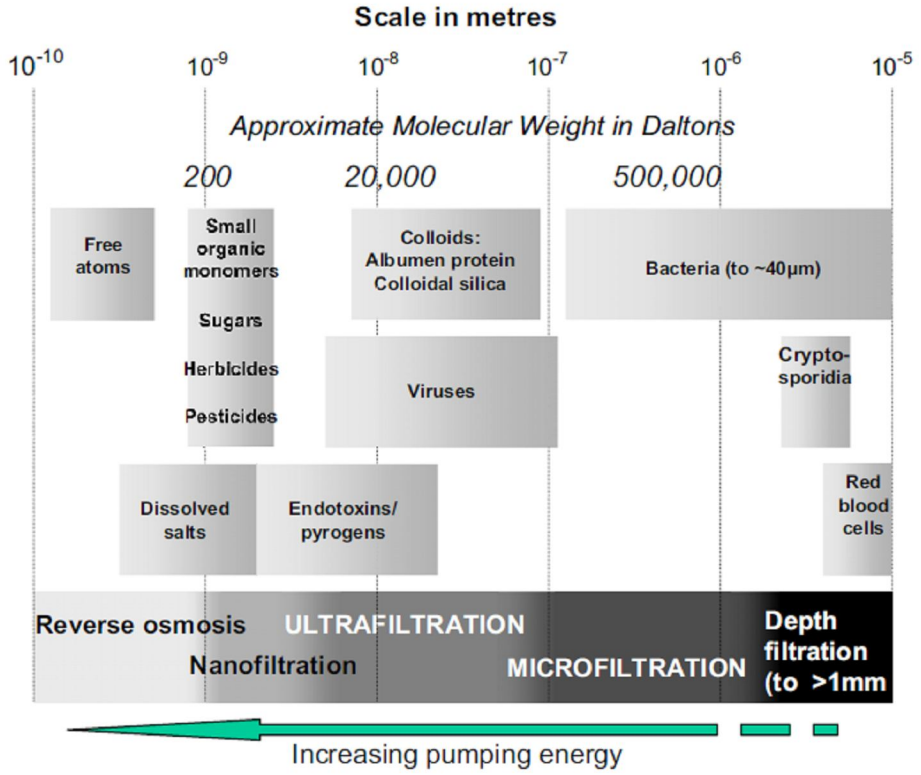


Figure 2.3. Membrane separation processes overview (Judd, 2006).

Among the membrane processes, membrane bioreactor (MBR) technologies are becoming an innovative and promising option for wastewater treatment and reuse (Guo et al., 2008). Microfiltration and ultrafiltration is widely used in MBR wastewater treatment field. Several researchers have used microfiltration (e.g. Yoon et al. 2004; Shim et al., 2002; Ferraris et al., 2009; Guo et al., 2008; Kawasaki et al., 2007) and ultrafiltration (e.g. Hasar and Kinaci, 2004) for wastewater treatment in their studies.

2.3.2. Membrane Materials

There are mainly two different types of membrane material, these being polymeric and ceramic. A number of different polymeric and ceramic materials are used to form membranes. The MBR market is now supplied with products mainly based on polyethersulfone (PES), polyvinylidene difluoride (PVDF) or on derivatives of polyethylene (PE). Membranes are usually fabricated both to have a high surface porosity (or per cent total surface pore cross-sectional area) and narrow pore size distribution to provide as high a throughput and selectivity as possible (Judd, 2006).

2.3.3. Membrane Element Configurations

The configuration of the membrane element, that is, its geometry and the way it is mounted and oriented in relation to the flow of water, is crucial in determining the overall process performance (Judd, 2006).

There are six principal configurations currently employed in membrane processes, which all have various practical benefits and limitations. The configurations are based on either a planar or cylindrical geometry and comprise (Judd, 2006):

- Plate-and-frame/flat sheet (FS)
- Hollow fiber (HF)
- (Multi) tubular (MT)
- Capillary tube (CT)
- Pleated filter cartridge (FC)
- Spiral-wound (SW)

Of the above configurations, only the first three are suited to MBR technologies. Membrane configurations and schematics showing flow through membrane (configured as FS, CT or MT and HF) are shown in Figure 2.4 and Figure 2.5, respectively.

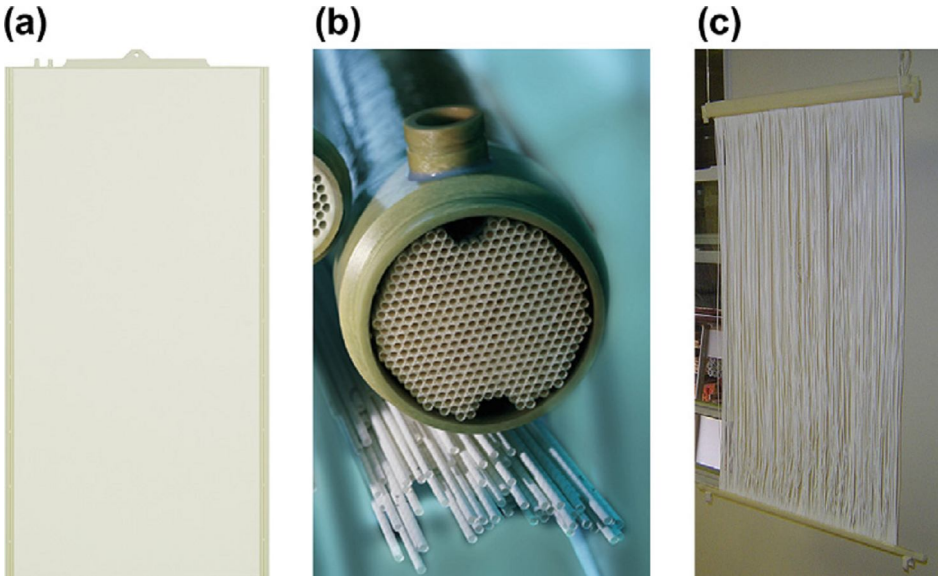


Figure 2.4. Membrane configurations (a) FS, (b) MT, (c) HF modules (Judd, 2006).

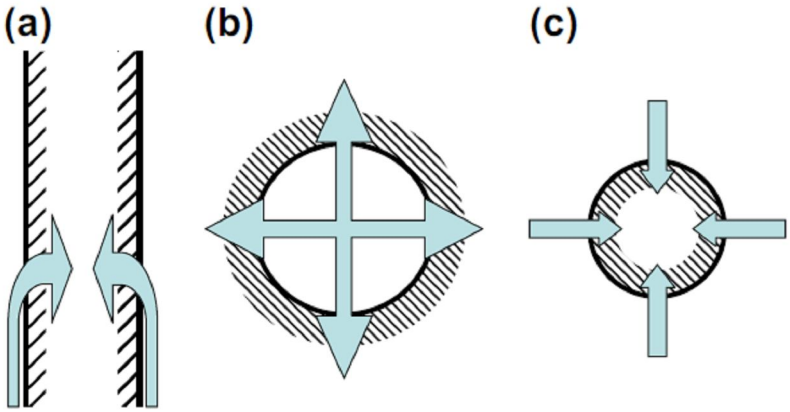


Figure 2.5. Schematics showing flow through membrane configured as (a) FS, (b) CT or MT, (c) HF (Judd, 2006).

Although there are several types and configurations of membranes, the widely and mainly used membranes for a submerged type are hollow fibers and flat sheet membranes (Le-Clech et al., 2005; Shim et al., 2002).

2.3.4. Membrane Process Configurations

The word ‘configuration’ for the MBR process is used to refer specifically how the membrane is integrated with the bioreactor (Judd, 2006). According to their configuration, MBR processes are classified as submerged MBR (immersed) which is integrated to the bioreactor and sidestream MBR (crossflow) which is recirculated or external (Cicek, 2003; Melin et al., 2006; Ueda et al., 1997; Judd, 2008). Schematic representation of MBR process configurations is given in Figure 2.6.

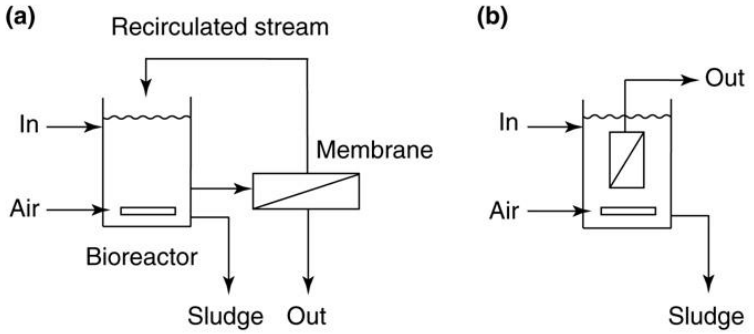


Figure 2.6. MBR process configurations (a) sidestream (b) submerged (Judd, 2008).

There are also two modes of hydraulic operation for MBR process: pumped and air-lift. Principle configurations of MBR technologies are summarized in Figure 2.7.

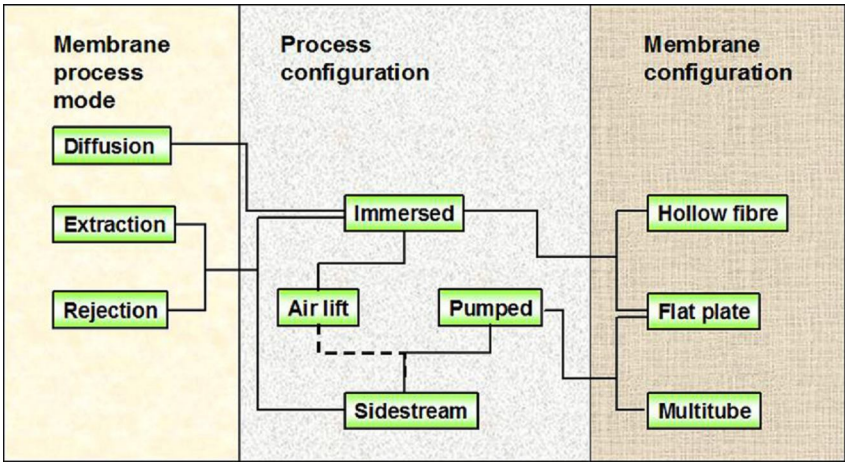


Figure 2.7. Principle configurations of MBR technologies (Judd, 2006).

The two MBR configurations (external/immersed) are characterized by different operating conditions such as membrane material, filtration mode, shear stress etc. and they have some effects on sludge structure and microbial activity (Clouzot et al., 2011).

2.3.5. Membrane Process Operation

The key elements of any membrane process relate to the influence of the following parameters on the overall permeate flux (Judd, 2006).

- the membrane resistance,
- the operational driving force per unit membrane area,
- the hydrodynamic conditions at the membrane-liquid interface and
- the fouling and subsequent cleaning of the membrane surface.

2.3.5.1. Fouling Mechanisms. Fouling is the restriction, occlusion or blocking of membrane pores at the surface of the membrane, reducing the flow of permeate water through the membrane material (Judd, 2008). Filtration proceeds according to a number of widely recognized mechanisms comprising (Figure 2.8):

- complete blocking,
- standard blocking,
- intermediate blocking and
- cake filtration.

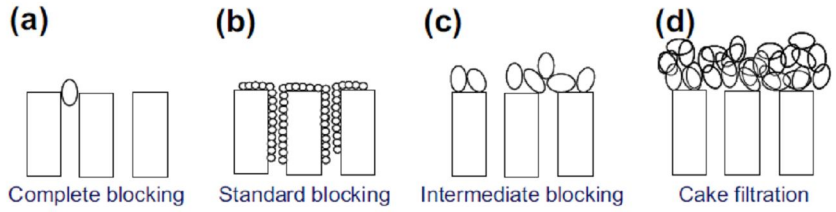


Figure 2.8. Fouling mechanisms (Judd, 2006).

The standard blocking and cake filtration models appear most suited to predicting initial flux decline during colloid filtration or protein filtration (Judd, 2006).

Fouling can be classified into different categories. Traditionally, the term *reversible fouling* refers to fouling that can be removed by physical means such as backflushing or relaxation under crossflow conditions, while *irreversible fouling* refers to fouling which can only be removed by chemical cleaning (Drews, 2010).

2.3.5.2. Membrane Cleaning Methods. The main impact of the operating flux is on the period between cleaning, which may be by physical or chemical means (Figure 2.9), and usually both.

In MBRs, physical cleaning is normally achieved either by backflushing, i.e. reversing the flow, or relaxation, which is simply ceasing permeation whilst continuing to scour the membrane with air bubbles. These two techniques may be used in combination, and backflushing may be enhanced by combination with air. Chemical cleaning is carried out with mineral or organic acids, caustic soda or, more usually in MBRs, sodium hypochlorite, and can be performed either in situ ('cleaning in place' or CIP) or ex situ. Alternatively, a lower concentration of chemical cleaning agent can be added to the backflush water to produce a 'chemically enhanced backflush' (CEB), usually performed only periodically (Judd, 2006).

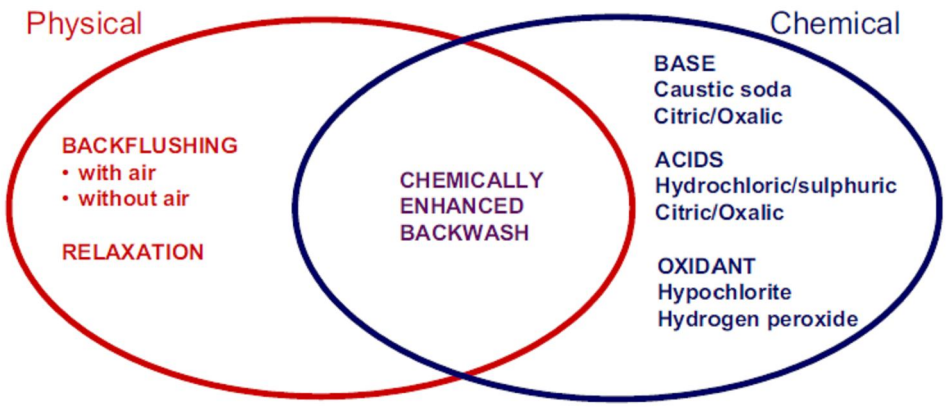


Figure 2.9. Membrane cleaning methods (Judd, 2006).

2.3.6. Advantages of MBRs

In literature, several advantages associated with the MBR which make it a valuable alternative over other treatment techniques have been introduced by many researchers (Pérez-Elvira et al., 2006; Melin et al., 2006; Ferraris et al., 2009; Yang and Cicek, 2008; Howell et al., 2003; Tian et al., 2009; Trussell, 2005; Le-Clech, 2010). The main advantages of MBR technology can be summarized as followings:

- Lower sludge production (option for high sludge age),
- Enhanced nutrient removal stability,
- Smaller foot print and smaller reactor volume as a consequence of higher MLSS concentration and loading rate (option for low to moderate sludge age),
- Higher and more consistent effluent quality as a result of membrane filtration,
- High treated water quality suitable for re-use,
- Complete suspended solids retention and consequently easy control of biomass concentration and operation at higher biomass concentrations,
- Operational reliability and stability,
- Lower sensitivity to contaminant peaks so easier to treat difficult wastes,
- The flexibility of operation,
- Complete solids/liquid separation and total solids retention at all biomass concentrations,
- High intensity of biochemical oxygen demand (BOD) removal,
- Lower sludge yields,
- Sludge is less fouling,
- Effluent is disinfected and almost virus free from UF.

Pérez-Elvira et al. (2006) stated that in a MBR, solids retention time (SRT) can be controlled independently from hydraulic retention time (HRT), which will result in a higher sludge concentration (typically 15-20 g/L), and subsequently in a lower sludge loading rate. When this sludge loading rate becomes low enough, little or no excess sludge can be produced.

2.3.7. Disadvantages of MBRs

Besides its advantages over conventional methods, MBR have also some disadvantages. The main disadvantages of MBR documented in the literature studies (Pérez-Elvira et al. 2006; Cicek, 2003; Melin et al., 2006; Kawasaki et al., 2007; Drews, 2010) are:

- Sludge settling and dewatering is more difficult because of the characteristics of the sludge; open flocs , high viscosity and high SVI,
- Membrane fouling, which requires frequent cleaning and replacement (high cost),
- Energy requirements,
- Relatively expensive to install and operate,
- Frequent membrane monitoring and maintenance,
- The adhering of sludge solids onto the membrane surface and the clogging of membrane pore,
- Less efficient oxygen transfer caused by high MLSS concentrations,
- Treatability of surplus sludge is questionable.

The disadvantages associated with the MBR are mainly cost related. As a natural result of the development in the membrane technology field the installation cost of membrane decreases.

Also, the membrane fouling considered as a phenomena and the biggest problem related to MBR can be prevented in many situations. It was reported that the membrane fouling might be reduced when adopting appropriate strategies and operating parameters (Tian et al., 2009).

2.4. Sludge Disintegration

In recent years, several disintegration methods have been investigated and developed as pretreatment processes for the purpose of excess sludge minimization. Mechanical, thermal, chemical and biological pretreatment techniques are mainly used in order to improve biological sludge degradation (Winter, 2002; Bougrier et al., 2005; Filibeli and Kaynak, 2006; Chu et al., 2009). These pretreatments cause the lysis or disintegration of sludge cells (Bougrier et al., 2005).

The mechanisms and objectives of disintegration process were briefly reviewed here.

2.4.1. Sludge Disintegration Mechanisms

The strategies of excess sludge minimization (mechanical, chemical, thermal and biological methods) are generally based on lysis-cryptic growth. The biomass growth on the lysates (that subsequently occurs on the autochthonous substrate) is termed *cryptic growth*, to distinguish from growth on the original organic substances (Wei et al., 2003; Chu et al., 2009).

When disintegration methods are applied, microbial cells undergo lysis or death during which cell contents (substrate and nutrients) are released. The organic autochthonous substrate is reused in microbial metabolism and a portion of the carbon is liberated as respiration products. This results in a reduction in the overall biomass production. As shown in Figure 2.10, there are two stages in cryptic growth: lysis and biodegradation.

The rate-limiting step of lysis-cryptic growth is the lysis stage (Wei et al., 2003; Chu et al., 2009), and an increase of the lysis efficiency can therefore lead to an overall reduction of sludge production (Wei et al., 2003).

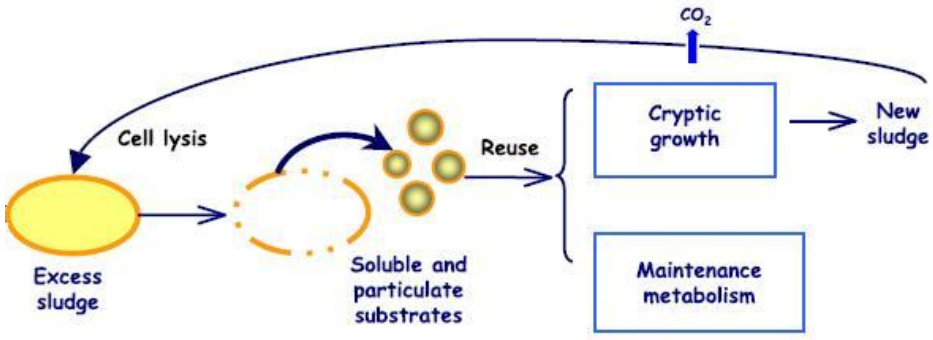


Figure 2.10. Schematic diagram of cryptic growth (Chu et al., 2009).

Sewage sludge disintegration can be defined as the destruction of sludge by external forces. These forces can be of physical, chemical or biological nature. A result of the disintegration process is numerous changes of sludge properties, which can be grouped in three main categories (Müller et al., 2004):

- destruction of floc structures and disruption of cells,
- release of soluble substances and fine particles,
- biochemical processes.

2.4.1.1. Floc Destruction and Cell Disintegration. The applied stress during the disintegration causes the destruction of floc structures within the sludge and/or leads to the break-up of micro-organisms. If the energy input is increased, the first result is a drastic decrease in particle size within the sludge. The destruction of floc structures is the main reason for this behavior. The disruption of microorganisms is not as easily determined by the analysis of particle size because disrupted cell walls and the original cells are of similar size. Floc destruction and cell disruption will lead to some changes in sludge characteristics such as hydrolysis (disintegrated micro-organisms are much more easily hydrolyzed than undisrupted ones), settling and dewatering, flocculation, viscosity and foaming (Müller et al., 2004).

2.4.1.2. Release of Soluble Substances and Small Particles. The destruction of floc structures and disruption of cells result in the release of organic sludge components into the liquid phase. These components exist in a dissolved phase already, e.g. components of the intracellular water, or can be liquefied. Particle size or colloidal components may still be present within the solution because they cannot be separated from the liquid phase. Their minute particle size and only a slight difference in density of particle and surrounding water are the cause. But the components are easily biodegradable on the other hand. Since they are already liquefied or offer a large surface in comparison to their volume, the hydrolyzing process is simple. The influence of the released amounts of carbon, nitrogen and phosphorus compounds on sludge characteristics are degradation, return-flow-pollution, recycling. After disintegration, the liquid phase has to be cleaned from released organic (such as nitrogen and phosphorus) compounds before leaving the treatment plant. Disintegration within the sludge pre-treatment has advantages in combination with selective recycling processes due to the increased nitrogen and phosphorus concentrations (Müller et al., 2004).

2.4.1.3. Biochemical Reactions. During or immediately after the disintegration, biochemical reactions may appear. The influence of these reactions on the degradability of the sludge is contrary:

- Continuing formation or release of easily degradable compounds
- Formation of hardly degradable compounds

Biochemical reactions following the disintegration that is catalyzed by released enzymes are seen controversially by experts. While some authors see great potential, others have not been able to prove any kind of effect (Müller et al., 2004).

The disintegration of micro-organisms leads to a release of organic substances which are easily accessible to a subsequent biological degradation process. The disintegrated sludges can be used as a substrate either in aerobic or anaerobic processes. This leads to an improvement of the biological degradation processes in wastewater and sludge treatment. The application of disintegration is especially useful for excess sludge because of its high content of micro-organisms (Müller, 2000).

2.4.2. Sludge Disintegration Methods

In recent years, several methods have been applied for the purpose of sludge disintegration and excess sludge minimization. Mechanical, thermal, chemical and biological pretreatment techniques are mainly used in order to improve biological sludge degradation (Winter, 2002; Bougrier et al.; 2005; Filibeli and Kaynak; 2006; Chu et al., 2009). Chemical disintegration of sludge typically includes the use of ozone, alkali and Fenton. Thermal treatment commonly used in the studies involves thermal hydrolysis and microwave (MW) irradiation. Ultrasonication is widely used for mechanical disintegration. The three types of pretreatments (chemical, thermal and mechanical disintegration) and combined pretreatments commonly discussed in the literature are given with some case studies as following:

2.4.2.1. Ozone. Ozone is a powerful oxidant and can oxidize a wide range of organic and inorganic compounds. The process of sludge ozonation is generally described by the sequential decomposition reactions of floc disintegration, solubilization, and the subsequent oxidation of the released organics intocarbon dioxide (mineralization). Following ozonation, the characteristics of the sludge are greatly changed. The flocs are broken down into fine, dispersed particles. Floc disintegration and solubilization generates a large number of micro particles dispersed in the supernatant in addition to soluble organic substances (Chu et al., 2009). Sludge disintegration by ozonation has been studied by several researchers.

Yasui et al. (1996) proposed and developed an activated sludge process coupled with ozonation for sludge reduction. They reported that In a full-scale operational experiment lasting 10 months under 550 kg/d of BOD loading, no excess sludge was needed to be withdrawn and no significant accumulation of inorganic solids occurred in the aeration tank. Material balance indicated that one-third of ozonated sludge was mineralized via the recirculation treatment, and thereby the requirement of sludge mass to be treated was 3.3 times as much as sludge to be eliminated. The operation costs associated with the process were estimated to be lower than those of conventional dewatering and disposal.

In a similar study, Sakai et al. (1997) developed an activated sludge process which produces no excess sludge. A full-scale plant for treating 450m^3/d of municipal wastewater was constructed and has been operated successfully for 9 months. At the ozone dosing rate of 0.034 kg/kg-SS, complete elimination of excess sludge has been achieved when 4 times more amount of sludge is ozonated than that of the excess sludge expected in the treatment without ozonation. As for effluent quality, BOD and nitrogen were kept good. Although effluent SS was 2-15 mg/L higher compared to a control without ozonation, it has been well below the discharge limit.

Moussavi et al. (2008) investigated the effect of ozonation on sludge treatment. Their results indicated that the ozone at doses between 0.125 to 2 gO_3 g^{-1} TS reduced TS, TSS and VS between 15.4% to 80.7%, 8.3% to 47.9% and 5.8% to 45.9%, respectively. Ozonation also improved the settlabilty of the sludge which resulted in reducing the sludge volume.

2.4.2.2. Alkaline. Alkali treatment is relatively effective in sludge solubilization, with in order of efficacy being (NaOH > KOH > $Mg(OH)_2$ and $Ca(OH)_2$) (Carrère et al., 2010). Li et al. (2008) investigated alkaline sludge treatment to know the effects of different doses (0-0.5 mol/L) of sodium hydroxide (NaOH) and calcium hydroxide [$Ca(OH)_2$] at 0-40 ^oC. Their results showed that NaOH was more suitable than $Ca(OH)_2$ for sludge disintegration. For NaOH treatment, most efficient dose was about 0.05 mol/L (0.16 g/g dry solid), and 60-71% solubilization of organic matters was achieved in first 30 min during its treatment of 24 h.

Most of the literature pertaining to alkaline treatment describes how alkali addition enhances other sludge disintegration techniques by making alkali-treated sludge more amenable to hydrolysis (Elliott and Mahmood, 2007).

2.4.2.3. Fenton. Fenton reactions are responsible for the degradation of organic pollutants. Dewil et al. (2005) studied the influence of the Fenton peroxidation on the thermal conductivity of the sludge. Their results demonstrated that the Fenton's peroxidation positively influences the sludge cake consistency and hence enhances the mechanical dewaterability and the drying characteristics of the dewatered sludge.

Tokumura et al. (2007) applied the photo-Fenton reaction as a novel technique for the minimization of excess sludge in activated sludge process and they examined experimentally its effectiveness. They found that the chemical oxidation sludge disintegration by photo-Fenton reaction could be divided into two phases. At the beginning of the photo-Fenton process the dissolved chemical oxygen demand (COD) increased. The COD reached the maximum and then decreased. They noted that the Fe dosage enhanced the sludge disintegration by the photo-Fenton reaction as well as the H_2O_2 dosage. They concluded that photo-Fenton reaction is one of feasible processes for disintegration of excess activated sludge.

Similarly, Tokumura et al. (2009) investigated the solubilization of excess sludge by the solar photo-Fenton reaction for the reduction of excess sludge in the activated sludge process. They found that the solubilization kinetics depended on the dosages of the Fenton reagents, Fe and H_2O_2. About 40% reduction of mixed-liquor suspended solids (MLSS) by the solar photo-Fenton reaction was found. Additionally, they found that solar light used as a light energy source instead of costly and hazardous artificial UV light was very effective.

2.4.2.4. Thermal Hydrolysis. Thermal pre-treatments can be used in order to enhance the efficiency of anaerobic digestion of waste activated sludge (Bougrier et al., 2007; Bougrier et al., 2008). In their study, Bougrier et al. (2008) found that COD solubilization increased linearly with treatment temperature for temperatures lower than 200 ^{o}C, and all the different sludge samples behaved in the same way. For temperatures lower than 150 ^{o}C, carbohydrates solubilization was more important than proteins solubilization. They also reported that thermal treatments up to 190 ^{o}C allowed the biogas production to increase during batch anaerobic digestion of sludge.

2.4.2.5. Microwave Irradiation. Recent years have seen growing interest in microwave irradiation as a promising technique for sludge treatment, because the use of microwave energy may lead to significant waste volume reduction, processing time reduction, and savings in energy consumption (Yu et al., 2010). The mechanism of microwave irradiation includes a thermal effect and an athermal effect. The term “athermal effect”, for microwave, generally relates to an effect that is not associated with an increased temperature, while “thermal effect” relates to the process that generates heat as a result of

the absorption of the microwave energy by water or organic complexes marked by either constant or induced polarization. The microwave energy is transformed into heat derived from the internal resistance of rotation (Yu et al., 2010).

Wojciechowska (2005) studied the effect of microwave treatment on the dewaterability of sewage sludge. In this study, the specific resistance to filtration, capillary suction time, and dry matter content in centrifuged sludge cake were measured. It was found out that microwaves improve sludge dewaterability. The quantitative improvement depends on sludge type: better effect of microwave processing was observed in the primary sludge compared to mixed or digested sludges.

2.4.2.6. Combined Disintegration Studies. The combined pretreatment methods studied in the literature and their effects on sludge disintegration were briefly reviewed here.

Doğan and Sanin (2009) studied the combination of alkaline solubilization (using NaOH) and microwave (MW) irradiation (160 ^{o}C) as a pretreatment method of waste activated sludge (WAS). First alkaline and MW pretreatment methods were examined separately, then their combination for different conditions was investigated in terms of their effect on COD solubilization, turbidity and capillary suction time (CST). For combined pretreatments, soluble COD to total COD ratio (SCOD/TCOD) of WAS increased from 0.005 (control) to 0.18, 0.27, 0.34 and 0.37 for combined methods of MW and pH-10, 11, 12 and 12.5, respectively. Deteriorated dewaterability due to alkaline pretreatment was also improved due to the incorporation of MW irradiation. Further, with small scale batch anaerobic reactors, pH-10, pH-12, MW (alone), MW+pH-10 and MW+pH-12 pretreated WAS samples were anaerobically digested. Highest total gas and methane productions were achieved with MW+pH-12 pretreatment with 16.3% and 18.9% improvements over control reactor, respectively. Finally the performance of MW+pH-12 pretreatment was examined with 2 L anaerobic semi-continuous reactors for 92 days and compared to that of the control reactors. After steady state, 43.5% and 55% improvements were obtained in respective daily total gas and methane productions. TS, VS and TCOD reductions were improved by 24.9%, 35.4% and 30.3%, respectively based on a relative calculation with respect to control reactors. This way combined alkaline-microwave treatment proved to be an effective sludge minimization method.

Kim et al. (2010) tested the individual effects of alkaline (pH 8-13) and ultrasonic (3750-45000 kJ/kg TS) pretreatments on the disintegration of sewage sludge separately and then investigated the effect of combining these two methods at different intensity levels. In the combined pretreatment, ultrasonic treatment was applied to the alkali-pretreated sludge. While the solubilization (SCOD/TCOD) increase was limited to 50% in individual pretreatments, it reached 70% in combined pretreatment, and the results clearly showed that preconditioning of sludge at high pH levels played a crucial role in enhancing the disintegration efficiency of the subsequent ultrasonic pretreatment. The results demonstrated that DD increased as pH increased, but it seemed that DD decreased when the specific energy input exceeded about 20000 kJ/kg TS. This phenomenon showed that there exists a certain point where additional energy input is ineffective in achieving further disintegration. A synergetic disintegration effect was also found in the combined pretreatment, with lower specific energy input in ultrasonic pretreatment yielding higher synergetic effect.

2.5. Ultrasonic Sludge Disintegration

Ultrasonic cell lysis was first developed through laboratory-scale research in the 1960s, but was initially uneconomical due to limitations of the ultrasound equipment available at that time. Advances in ultrasound technology in the last decade have enabled commercial application of the technology for wastewater applications. Ultrasound can be used for sludge minimization in the activated sludge process or in digestion (Roxburgh et al., 2006).The mechanisms and effects of ultrasonic treatment on sludge disintegration were briefly reviewed here.

2.5.1. Mechanisms of Ultrasonic Disintegration

Ultrasounds include a wide range of frequencies between 20 kHz and 10 MHz (Dewil et al., 2006a; Dewil et al., 2006b). It is the term which is used to describe sound energy at frequencies above 20 kHz, i.e. above the range which is normally audible to human beings (Clark and Nujjoo, 1998; Khanal et al., 2007). The nomenclature of sound waves at different frequencies is given in Figure 2.11.

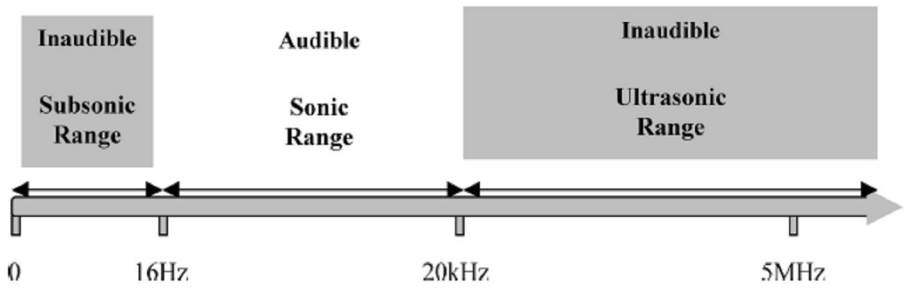


Figure 2.11. Nomenclature of sound waves at different frequencies (Khanal et al., 2007).

In sludge treatment, it has been shown that degradation of excess sludge is more efficient using low frequencies (20-40 kHz) (Carrère et al., 2010; Bougrier et al., 2005).

Ultrasonic treatment acts to mechanically disrupt the cell structure and floc matrix. There are two key mechanisms associated with ultrasonic treatment; *cavitation*, which is favored at low frequencies, and *chemical reactions* due to the formation of OH•, HO_2•, H• radicals at high frequencies (Carrère et al., 2010).

2.5.1.1. Cavitation Phenomena. High-power ultrasound is usually generated by a transducer (which converts mechanical or electrical energy into high-frequency vibrations), and is delivered into a fluid via a horn or probe (Clark and Nujjoo, 1998).

When the ultrasound wave propagates in sludge medium, it generates compressions and rarefactions, the compression cycles exert a positive pressure on the liquid by pushing the molecules together and the rarefaction cycle exerts a negative pressure by pulling the molecules from one another. Because of this excessively large negative pressure, microbubbles (cavitation bubbles) are formed in the rarefaction regions. These microbubbles grow in successive cycles and reaches to an unstable diameter that collapse violently producing shock waves (temperature of around 5000 ^{o}C and pressure of 500 atmospheres at a lifetime of few microseconds) (Zhang et al., 2007; Bougrier et al., 2005; Cao et al., 2006; Pilli et al., 2011;). This process by which the bubbles form, grow and undergo violent collapse is known as *cavitation* (Pilli et al., 2011) and it is only encountered at frequencies below 1 MHz (Dewil et al., 2006a). Representation of development and collapse of the cavitation bubble is shown in Figure 2.12.

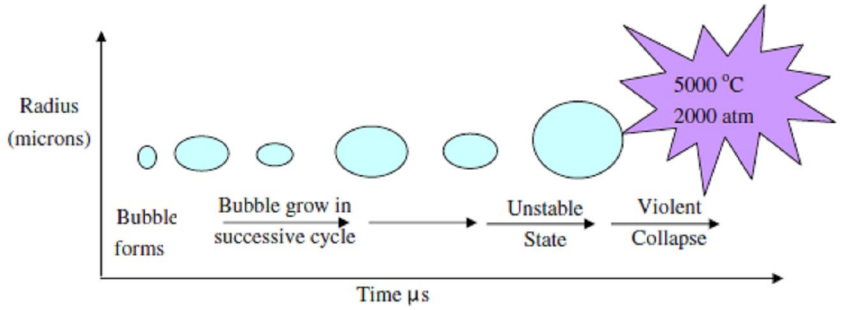


Figure 2.12. Development and collapse of the cavitation bubble (Pilli et al., 2011).

Because of the extreme local conditions, OH•, HO_2•, H• radicals and hydrogen peroxide can be formed during cavitation phenomena (Bougrier et al., 2005). Therefore, cavitation will result in (Dewil et al., 2006a; Dewil et al., 2006b):

- the promotion of chemical reactions as a result of a locally high temperature and pressure,
- extreme shear forces in the liquid, thereby mechanically attacking components,
- the formation of highly reactive radicals (H• and OH•) which can again facilitate chemical reactions to take place and
- the additional destruction of specific compounds since cavitation bubbles are surrounded by a liquid hydrophobic boundary layer which preferentially permeates volatile and hydrophobic substances, subsequently reacting in the gas bubble.

The sludge disintegration efficiency is essentially based on the cavitation phenomena. The mechanism of cavitation plays an important role when applying ultrasounds to waste activated sludge. The factors influencing the mechanism of cavitation are (Clark and Nujjoo, 1998; Bougrier et al., 2005; Pilli et al., 2011);

- Liquid temperature (it is more likely to occur at higher temperatures),
- Viscosity,
- Surface tension,
- Ultrasonic intensity (often referred to as the acoustic energy density),

- Duration and,
- Frequency of ultrasonic vibration (usually set at 20 kHz or 35 kHz).

2.5.1.2. Chemical Reactions. There are four paths, which are shown as following, responsible for the ultrasonic activated sludge disintegration (Wang et al., 2005):

- hydro-mechanical shear forces,
- oxidizing effect of •OH, •H, •N and •O produced under the ultrasonic radiation,
- thermal decomposition of volatile hydrophobic substances in the sludge,
- increase of temperature during ultrasonic activated sludge disintegration.

Disintegration by ultrasonic treatment causes disruption of microbial cells in the sludge, thereby destroying the cell walls. The destruction of floc structures and disruption of cells result in the release of organic sludge components (extracellular and intracellular matter) into the liquid phase. Released carbon compounds after disintegration are easily accessible and can be digested much faster in later biological process than sludge in a particular phase. The results are shorter degradation times and higher degrees of degradation during the aerobic stabilization. Hence, ultrasound can be used and integrated with biological wastewater treatment systems for enhancing sludge decomposition.

Mechanisms of the ultrasonic process are influenced by some factors (Bougrier et al., 2005; Carrère et al., 2010):

- supplied energy,
- ultrasonic frequency and
- nature of the influent.
- the treatment time and power

Cell disintegration is proportional to supplied energy. High frequencies promote oxidation by radicals, whereas low frequencies promote mechanical and physical phenomena like pressure waves. With complex influents, radical performance decreases (Bougrier et al., 2005).

2.5.2. Applications of Ultrasonic Sludge Disintegration

Many researchers have investigated the effect of ultrasonic treatment on sludge disintegration (e.g. Zhang et al., 2007; Zhang et al., 2009; Cao et al., 2006; Yin et al., 2006; Pham et al., 2009; Tiehm et al., 2001; Salsabil et al., 2009; He et al., 2011) and their results are given following:

Zhang et al. (2007) found that the most effective conditions for sludge reduction were as following: sludge sonication ratio of 3/14, ultrasound intensity of 120 kW/kgDS, and sonication duration of 15 min., while the researches Cao et al. (2006) showed that suitable treatment condition by ultrasound might be kept with power density 0.25 W/mL, treatment ratio 50%, and sonication time prolonged in the suitability of economy for their study. Zhang et al. (2007) and Cao et al. (2006) mainly agreed that effective physical disintegration of sludge can decrease the disadvantages of chemical oxidations and therefore they applied ultrasonic technology in their study which is based on its distinct advantage, such as safe, clean and effective.

Zhang et al. (2009) evaluated the performance of previous study (Zhang et al., 2007) in practical wastewater treatment using urban sewage without temperature control. In their previous study they reported that sonication is effective to reduce WAS using artificial wastewater in the conditions of 3/14 (0.21) sonication ratio and 120 kW/kgDS ultrasound intensity. The results of their latter study showed that the optimal specific energy for sludge lysis was 20 kWh/kgDS. When the specific energy was 20 kWh/kgDS and the sludge recycle ratio was 0.007, the WAS decreased by 54%, the biomass synthesis abated by 59%, and the sewage mineralization ratio increased from 31% to 58%.

Yin et al. (2006) reported the influences of low frequency ultrasound (20 kHz) on dewater ability and anaerobic digestion behaviors of activated sewage sludge. The results demonstrated that together with flocculant, the ultrasound pretreatment decreased the specific filtration resistance (SFR) of the sludge from 3.59×10^{12} m/kg to 1.18×10^{12} m/kg and saved about 25–50% of the flocculant dosage. They also noted that ultrasound pretreatment could enhance digestion and reduce digestion time.

Pham et al. (2009) studied the pretreatment of wastewater sludge by ultrasonic waves at frequency of 20 kHz using fully automated lab-scale ultrasonication equipment. Different wastewater sludge solids concentrations, ultrasonication intensities, and exposure times of pre-treatment were investigated for the optimization of ultrasonication treatment process. The effect of ultrasonication treatment was assessed in terms of increase in soluble solids and the biodegradability of the wastewater sludge. In addition, rheological parameter of wastewater sludge, namely, viscosity was also measured. Their studies revealed that the optimal conditions of ultrasonic pre-treatment were 0.75W/cm^2 ultrasonication intensity, 60 min exposure time, and 23 g/L total solids concentration.

Tiehm et al. (2001) investigated the pretreatment of waste activated sludge by ultrasonic disintegration in order to improve the anaerobic sludge stabilization. The results showed that short sonication times resulted in sludge floc deagglomeration without the destruction of bacteria cells. Longer sonication brought about the break-up of cell walls, the sludge solids were disintegrated and dissolved organic compounds were released. The anaerobic digestion of waste activated sludge following ultrasonic pretreatment causing microbial cell lysis was significantly improved. There was an increase in the volatile solids degradation as well as an increase in the biogas production.

Salsabil et al. (2009) compared aerobic and anaerobic digestions in reactors fed with sonicated activated sludge. They observed an important improvement of aerobic and anaerobic biodegradability for a sonication treatment of 108000 kJ/kgTS due to the increase of the instantaneous specific soluble COD uptake rate. Sonication also led to an increase of biogas production due to the increase of available soluble COD.

More recently, He et al. (2011) studied the influences of operational parameters to improve the energy efficiency during ‘ultrasonic lysis–cryptic growth’ sludge reduction. They defined a new index, EE, to evaluate the energy efficiency in ultrasonic lysis–cryptic growth sludge reduction system. Results showed that the most important operational parameter was the proportion of sonicated sludge (SP) (with upper limit of SP: 30%). In summary, the recommended conditions for ‘ultrasonic lysis–cryptic growth’ sludge reduction were SP of 15%, lysis frequency of 1 time/day. Under these conditions, the highest energy efficiency of 0.012 kg TS/kWh was achieved.

2.6. Sequential Batch Reactors (SBRs)

Sequential batch reactors or sequencing batch reactors (SBR) are processing tanks for the treatment of wastewater. SBR reactors treat wastewater such as sewage or output from anaerobic digesters or mechanical biological treatment facilities in batches. Oxygen is bubbled through the wastewater to reduce biochemical oxygen demand (BOD) and chemical oxygen demand (COD) to make suitable for discharge into sewers or for use on land. While there are several configurations of SBRs the basic process is similar. In its most basic form, the SBR system is a set of tanks that operate on a fill-and draw basis. Each tank in the SBR system is filled during a discrete period of time and then operated as a batch reactor. After desired treatment, the mixed liquor is allowed to settle and the clarified supernatant is then drawn from the tank. The cycle for each tank in a typical SBR is divided into five discrete periods: Fill, React, Settle, Draw and Idle.
(http://www.iwawaterwiki.org/xwiki/bin/view/Articles/SequencingBatchReactor_R)

2.7. Literature Review of Combined Systems for Sludge Minimization

Liu and Tay (2001) reported that the combination of the different processes would generate novel and more efficient techniques for the minimization of excess sludge production. It is well documented that excess sludge production can be greatly reduced by partial disintegration of the returned sludge in an activated sludge process (Ramakrishna and Viraraghavan, 2005; Chu et al., 2009).

In a biological wastewater treatment system, new sludge is continuously generated with the consumption of feed organic materials while some sludge mass is decayed by endogenous respiration. Due to the slower sludge decay rate, these sludge generation and reduction cannot reach an equilibrium state. As a result, some part of excess sludge should be removed to maintain proper MLSS level. If sludge decay rate can be boosted by some sort of sludge disintegration, sludge generation and decay rates will reach equilibrium (Yoon, 2003).

According to the type of the biological reactors, different combined systems with sludge disintegration to reduce excess sludge production have been reported.

2.7.1. Membrane Bioreactor-Sludge Disintegration (MBR-SD) Systems

In order to prevent excess sludge production during wastewater treatment, a membrane bioreactor-sludge disintegration (MBR-SD) system has been introduced, where the disintegrated sludge is recycled to the bioreactor as a feed solution (Yoon, 2003).

Figure 2.13 shows a schematic representation of the MBR process equipped with a sludge disintegration device. In this system, sludge decay is promoted by an artificial sludge disintegration process such as ozone, alkaline treatment, Fenton, ultrasound etc. The final steady-state MLSS will be achieved when sludge generation and decay rates are equivalent.

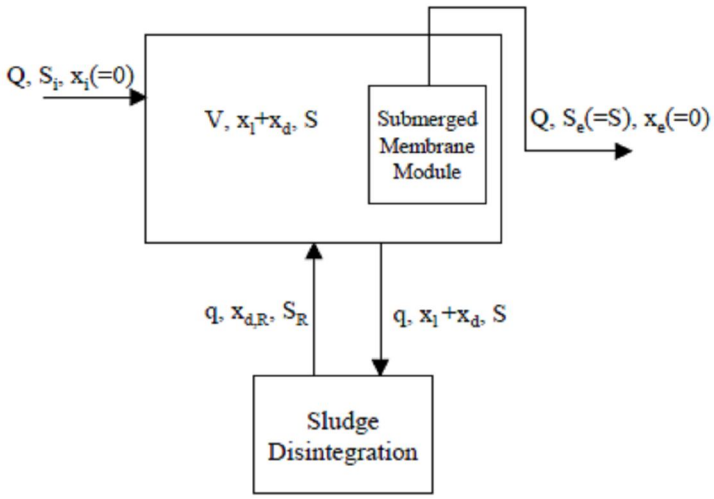


Figure 2.13. Schematic representation of MBR-SD system for zero excess sludge production (Yoon, 2003).

Song et al. (2003) investigated the effects of sludge ozonation on excess sludge minimization and enhancement of nutrient removal in membrane bioreactor (MBR). Two MBR system were operated in parallel with or without a batch-type sludge ozonation process. The specific ozone dosage was set at 0.1 gO_3/gSS. In the control run (without sludge ozonation), the daily sludge production was about 1.04 g/d. However, in the ozone run (with sludge ozonation), the daily sludge production was negligible. In addition, the MLSS concentrations and the volatile fraction of MLSS in the reactor maintained constant

around 8000 mg/L and 0.75, respectively, without excess sludge. The concentration of effluent was maintained at a satisfactory level in both runs. Furthermore, the MBR system with sludge ozonation process showed relatively better nutrient removal than without sludge ozonation. It revealed that ozonated sludge might be completely degraded in the reactor and effectively used as a carbon source. They concluded that application of sludge ozonation to the MBR system was significantly effective for minimization of excess sludge production as well as for enhancement of nutrient removal.

He et al. (2006) studied activated sludge ozonation to reduce sludge production in MBR. Total experimental period was divided into two stages. At first stage, a series of batch studies were carried out to get an understanding of the effect of ozonation on sludge properties. At the following stages, three membrane bioreactors (MBRs) with different amounts of activated sludge to be ozonated were run in parallel. On the basis of batch study, a suitable ozone dosage of 0.16 kgO_3/kg MLSS was determined. Three systems were run in parallel for a total period of 120 days; it was demonstrated that a part of activated sludge ozonation could reduce sludge production significantly. Experimental results proved that the combination of ozonation unit with MBR unit could achieve an excellent quality of permeate as well as a small quantity of sludge production, and economic analysis indicated that an additional ozonation operating cost for treatment of both wastewater and sludge was only US$ $0.0115/m^3$ wastewater.

In a similar study, MBR and sludge ozonation (SO) were coupled into the MBR-SO process to treat domestic wastewater for 80 days as a bench-scale study (Wang et al., 2008). To keep the MLSS concentration in MBR around 8000 mg/L, the ratio of flow-rate draining to ozonation unit (q) to influent wastewater flow-rate (Q) (q/Q) for each batch ozonation was set at 0.0067. The generated excess activated sludge was continuously drained into ozonation unit at a frequency of 2 batch/d for lysing cells. Almost constant MLSS concentration with zero observed sludge yield coefficient (Y_{obs}) and excellent effluent quality could be achieved in MBR except for TP concentration. The low input ozone gas concentration and high flow-rate could enhance the sludge lysing effects at same ozone dosage, and therefore lower energy consumption of US$$0.0053/m^3$ wastewater was obtained.

Oh et al. (2007) tested a new wastewater treatment process combining a MBR with chemical sludge disintegration in bench scale experiments. In the study, the effects of the disintegration treatment on the excess sludge production in MBR were investigated. Two MBRs were operated. In one reactor, a part of the mixed liquor was treated with NaOH and ozone gas consecutively and was returned to the bioreactor. The flow rate of the sludge disintegration stream was 1.5% of the influent flow rate. During the 200 days of operation, the MLSS level in the bioreactor with the disintegration treatment was maintained relatively constant at the range of 10000-11000 mg/L while it increased steadily up to 25000 mg/L in the absence of the treatment. They concluded that a complete control of excess sludge production in the membrane-coupled bioreactor was possible without significant deterioration of the treated water quality and membrane performances.

Ming-he and Chao-hai (2010) reported the minimization of excess sludge produced in the MBR coupled with a sludge Fenton oxidation (oxidation using H_2O_2 with an iron catalyst) process. Two MBRs with and without the Fenton process were operated to evaluate the influence of sludge Fenton oxidation on the sludge yield and water quality. Their study demonstrated that the incorporation of Fenton process can significantly reduce sludge production, as evidenced from the decrease in the value of the average sludge yield from 0.15 to 0.006 g MLSS/g COD. The water quality of effluent in both systems was maintained at a satisfactory level. Furthermore, the study revealed that the MBR system with the sludge Fenton oxidation process showed relatively better performance for TN removal than that without it.

Yoon et al. (2004) studied the incorporation of ultrasonic cell disintegration into a MBR for zero sludge production. The results of this study showed that excess sludge production could be prevented using an MBR-US hybrid system at an organic loading of around 0.91 kg BOD_5/m^3 per day. Under the same organic loading rate, the MLSS of MBR-US system was maintained at 7000-8000 mg/L while the MLSS of a conventional MBR increased from 7000 to 13700 mg/L during the experimental period. While sludge production was completely prevented, the effluent quality of the MBR-US system slightly deteriorated. With sonication the volume of the average particle size of the sludge in the aeration tank decreased from 132 to 95 μm. In the MBR-US system, around 25-30% of TP removal was achieved without sludge removal from the aeration tank.

2.7.2. Activated Sludge Process-Sludge Disintegration (ASP-SD) Systems

Lee et al. (2005) operated a pilot-scale activated sludge system coupled with sludge ozonation process for 112 days of a winter season without excess sludge wasting. The basis of operation was to determine either the optimal amount of sludge in kg SS ozonated each day (SO) or the optimal ozonation frequency (n) under the variable influent COD loading and temperature conditions. The optimal "n" was observed between 2.5 and 2.7 at around 15 ^{o}C, but it was doubled at 10 ^{o}C. MLSS concentration was leveled off at around 5000 mg/L in bioreactor at 15 ^{o}C, but the volatile fraction of MLSS was fixed around 0.7 indicating that there was no significant inorganic accumulation. Suspended solids (SS) and soluble COD in effluents kept always a satisfactory level of 10 and 15 mg/L with sufficient biodegradation.

2.7.3. Sequential Batch Reactor-Sludge Disintegration (SBR-SD) Systems

Huysmans et al. (2001) studied lab-scale sequencing batch reactor (SBR) combined with ozone pretreatment for sludge minimization. Intermittent ozonation of a part of the recycle sludge was performed two times a week for lab-scale reactor at an average ozone dose of 0.019 $gO_3/gSS_{ozonated}$. Under these conditions a decrease of about 50% in sludge growth was obtained for the ozone-treated system, in comparison with the control. Only a slight decrease in quality of effluent was noticed.

3. MATERIALS AND METHODS

3.1. Materials

3.1.1. Sludge

The activated sludge samples were supplied from Paşaköy Advanced Biological Wastewater Treatment Plant which is located in Istanbul. The plant, which has the wastewater treatment capacity of 200.000 m^3/day, treats the wastewater generated by a population equivalent of 1.000.000 PE.

The activated sludge was collected from aeration tank and recirculation unit of the plant. When the MLSS concentration of the sludge was not high enough, the sludge sample was concentrated by gravitational settling before starting the experiments. The sludge samples used for optimization of ultrasound disintegration was stored at 4 ^oC and brought to room temperature before using for analysis. The other sludge samples taken for sequential batch reactors (SBRs) and membrane bioreactors (MBRs) were not stored; they were directly poured into the reactors as soon as brought to the laboratory.

3.1.2. Feedwater

The wastewaster of Paşaköy Advanced Biological Wastewater Treatment Plant was used as feedwater. It was collected from the effluent of aerated grit chamber of the plant and was stored at 4 ^oC before use. It was brought to room temperature and aerated before feeding to reactors. The wastewater characteristics were analyzed periodically. The all information about characteristics of the feedwater used in the experiments is given in the Section 4.3.2.

3.1.3. Chemicals

All chemicals used in this study were of analytical grade. Chemicals and the experiments of their used are listed in Table 3.1.

Table 3.1. Chemicals used in the experiments.

Chemical Name	**Formula**	**Experiment**	**Supplier**
Potassium Dichromate	$K_2Cr_2O_7$	COD Analysis	Riedel-de Haën
Mercury (II) Sulfate	$HgSO_4$	COD Analysis	Riedel -de Haën
Silver Sulfate	Ag_2SO_4	COD Analysis	Fluka Analytical
Potassium Hydrogen Phthalate	KHP	COD Calibration	Merck
Sulfuric Acid	H_2SO_4	COD Analysis	Merck
		TP (Digestion)	
		TKN (Digestion)	
Hydrogen Peroxide	H_2O_2	TP (Digestion)	Sigma-Aldrich
		TKN (Digestion)	
Potassium Hydroxide	KOH	TP Analysis	Hach
		TKN Analysis	
Sodium Hydroxide	NaOH	Alkaline Digestion	Sigma-Aldrich

3.1.4. Instrumental Equipments

Some instrumental equipments used in this study and the experiments of their used are listed in Table 3.2.

Table 3.2. Instrumental equipments used in the experiments.

Instrumental Equipment	**Experiment**	**Trademark/Model**
COD Reactor	COD	Hach
Spectrophotometer	COD, TP, TKN, NH_3-N, NO_2^--N, NO_3^--N	Hach (DR/2010)
Digesdahl Digestion Apparatus	TP, TKN	Hach
Analytical Balance	MLSS, MLVSS, TS, TVS	Scaltec (SBA 31)
Filtration Apparatus	MLSS, MLVSS, TS, TVS	Schott Duran
Drying Oven	MLSS, MLVSS, TS, TVS	NÜVE (FN 500)
Muffle Furnace	MLSS, MLVSS, TS, TVS	Protherm
Dissolved Oxygen (DO) Meter	DO	WTW Oxi 330 Portable Oximeter
pH Meter	pH	WTW
Centrifuge	SCOD	Hettich Zentrifugen
Capillary Suction Time (CST) Instrument	CST	Venture Innovation
Viscometer	Viscosity	Brookfield
Particle Size Analyzer	Particle Size	Malvern Mastersizer 2000 (with dispersion unit of Hydro2000MU)
Ultrasonic Homogenizer	Digestion of sludge samples	Bandelin Sonopuls
Turbidimeter	Turbidity	Hach (2100P)

3.2. Methods

3.2.1. Experimental Set-up and Procedure

Experimental set-up mainly consists of three processes: ultrasound applications, sequential batch reactors (SBRs) and membrane bioreactors (MBRs). Each process used in the study is described in the following sections.

3.2.1.1. Ultrasound Applications. Ultrasonic experiments were performed by using an ultrasonic homogenizer (Bandelin-Sonopuls HD 3400) shown in Figure 3.1. The ultrasonic homogenizer used in the study is equipped with a generator (GM 3400), an ultrasonic converter (UW 3400), a booster horn (SH 3425) and a probe (VS 200 T). The generator converts the received power (50 or 60 Hz) into high-frequency power at a frequency of 20 kHz. The ultrasonic converter connected to the generator transforms the high-frequency power from the generator to ultrasound, converting it to a form of mechanical energy. This is achieved through an efficient and robust PZT ultrasonic transducer system. Hence, the tip of the probe also vibrates at a frequency of 20 kHz and transfers these vibrations with high power density to the sonicated sample (Bandelin, 2009). The ultrasonic unit used in this study has a constant frequency of 20 kHz. The amplitude and therefore the intensity of the ultrasound waves can be varied. The maximum energy-output is 400 W. The full specifications of the used ultrasonic equipment are listed in Table 3.3.

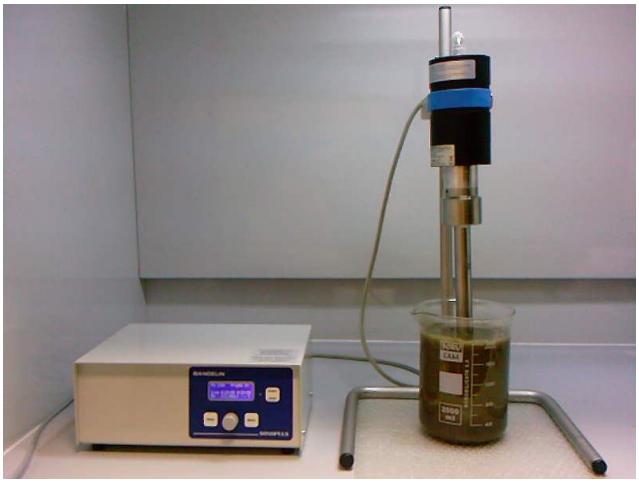


Figure 3.1. The ultrasonic homogenizer used in the study (Bandelin-Sonopuls HD 3400).

Table 3.3. Characteristics of the ultrasonic equipment.

GM 3400 Generator	
Power supply	230V~50/60Hz (alternatively 115V~50/60Hz)
Ultrasonic frequency	20 kHz
Maximum power	400 W
Power setting range	60 – 300 W
Weight	3.1 kg
Dimensions (l × w × h)	324 × 230 × 131 mm
Time setting range	0:00:01-9:59:59 (h:mm:ss) or continuous operation
Amplitude setting range	10 – 100 % in 1 % increments
UW 3400 Ultrasonic Converter	
Frequency	20 kHz
Weight	2.2 kg
Dimensions	Ø 90 ×180 mm
Degree of protection	IP 20
VS 200 T Probe	
Diameter	Ø 25 mm
Connection to standard horn	SH 3425
Volume range	100 – 2500 mL
Maximum admissible amplitude setting	100%
Immersion depth (recommended)	10 – 20 mm

In this study, batch experiments were carried out in beakers for ultrasonic applications. Schematic representation of an experimental set-up for the ultrasonic homogenizer is depicted in Figure 3.2.

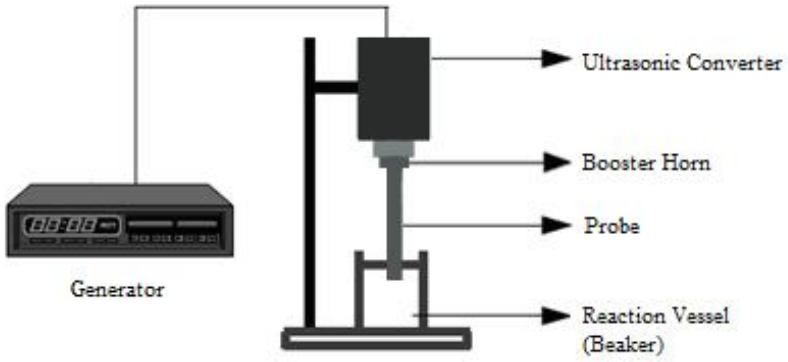


Figure 3.2. Schematic representation of an experimental set-up for the ultrasonic homogenizer (edited from Prasad et al., 2010).

For all sonochemically aided reactions, there exists optimum conditions (power density, power intensity, amplitude etc.) at which a given reaction can be performed optimally in order to avoid unnecessary loss of energy (Sivakumar and Pandit, 2001; Prasad et al., 2010). Thus, before applying ultrasound in reactors, all sonication conditions should be found out and optimized. In this frame, the overall experimental study was planned as following:

First, the ultrasonicator was calibrated to find optimum conditions (power output, amplitude etc.) of ultrasonic power, power density and power intensity for desired sample volumes (500 mL, 1000 mL and 2000 mL). Then, the experiments of ultrasonic sludge disintegration were carried out using ultrasonic conditions determined in the previous study. Subsequently, to minimize excess sludge production, ulrasonication was applied to sequential batch reactors (SBRs) and membrane bioreactors (MBRs) as a pretreatment technique.

(1) The Calibration of Ultrasonicator

Ultrasonic systems are usually calibrated and controlled with procedures that just give an indication about performances (the acoustical power). Basically, there are four different procedures to determine the acoustic power: electrical power measurement (Boucaud et al., 1999), acoustical dosimetry (Mason and Peters, 2002), chemical dosimetry (Koda et al., 2003) and calorimetric dosimetry (Teo et al., 2001). In this study, calorimetric dosimetry was performed to measure the ultrasonic power dissipated into system. This method assumes that almost all the mechanical energy produces heat and so, via calorimetry, some estimate of the acoustic power entering the system can be obtained in a pure liquid (Mason and Peters, 2002). Therefore, deionized water was used as a solvent for the calorimetric dosimetry experiments.

Batch experiments were carried out in glass beakers with the volumes of 500 mL, 1000 mL and 2000 mL. Deionized water of 500 mL at the ambient temperature was placed in a 600 mL low form griffin beaker. The other experiments were carried out as 1000 mL and 2000 mL of deionized water were filling in a 1000 mL and 2000 mL of glass beaker, respectively. Ultrasonic probe was immersed 20 mm into the deionized water containing beaker.

For the each system under study the temperature (T) is recorded against time (t), at 30 second intervals, using a thermocouple placed in the reaction itself. From the T vs. t data, the temperature rise at zero time, (dT/dt), was determined by curve-fitting the data to a polynomial in t. Then, the ultrasonic power actually entering the system was obtained from this by substituting the value of (dT/dt), at time zero into following Equation (Mason and Peters, 2002; Koda et al., 2003; Teo et al., 2001):

$$Power = \left(\frac{dT}{dt} \right) \cdot c_p \cdot M \tag{3.1}$$

where,

$\left(\frac{dT}{dt}\right)$: temperature rise per second

c_p : the heat capacity of water (4.184 J/g)

M : the mass of water (g)

The Power (in Watts) was dissipated into the system from a probe tip with an area of 4.91 cm^2. Therefore, the intensity of power (in W/cm^2) and the density of power (in W/mL) produced by the source of ultrasound was calculated by Equation 3.2 and Equation 3.3, respectively (Mason and Peters, 2002):

$$Intensity = \frac{Power}{Area} \tag{3.2}$$

$$Density = \frac{Power}{Volume} \tag{3.3}$$

Ultrasonic system was calibrated by calorimetry to find the optimum power output and amplitude values for the volumes of 500 mL, 1000 mL and 2000 mL. In the calorimetric dosimetry experiments, the power output values (from the generator of ultrasonic homogenizer) of 100 W, 150 W, 200 W, 225 W, 250 W and 275 W; and the amplitude values of 20%, 40%, 50%, 60%, 70%, 80% and 90% were studied for volumes of 500 mL, 1000 mL and 2000 mL separately.

The experimental conditions matrix for calorimetric dosimetry experiments are shown in Table 3.4.

Table 3.4. The experiment conditions matrix for calorimetric dosimetry.

Volume	**Power**	**Amplitude (%)**						
(mL)	**(W)**	20	40	50	60	70	80	90
500	100						×	
	150						×	
	200					×	×	
	225					×		
	250		×	×	×	×	×	×
	275					×		
1000	100						×	
	150						×	
	200						×	
	225						×	
	250		×		×	×	×	×
	275	×					×	
2000	100						×	
	150						×	
	200				×		×	
	225				×			
	250		×	×	×	×	×	×
	275				×			

(2) Ultrasonic Sludge Disintegration

The ultrasonic disintegration of sludge was studied at different sample volumes of 500 mL, 1000 mL and 2000 mL by using the optimum power output and the amplitude values, determined during the calibration of the ultrasonicator. The purpose of the ultrasonic sludge disintegration experiments was to test the performance of ultrasonic homogenizer for the subject sludge samples having constant TS concentration (specific field conditions) at the different operational conditions like sonication duration, power density etc. to determine the optimum disintegration conditions.

For ultrasonic disintegration experiments, sludge samples were collected from the aeration tank of Paşaköy Advanced Biological Wastewater Treatment Plant. Sludge samples was stored at 4 ^{o}C and brought to room temperature before using for analysis. Untreated sludge were analyzed for characterization by measuring TS, TVS, MLSS, MLVSS, SVI, TCOD, SCOD, viscosity, CST, pH, settling velocity, particle size and supernatant turbidity of sludge.

Batch experiments without temperature regulation were carried out in glass beakers for ultrasonication experiments. The sludge volume of 500 mL, 1000 mL and 2000 mL at the ambient temperature was placed in a 600 mL low form griffin beaker, 1000 mL and 2000 mL of glass beaker, respectively. The ultrasonic probe was immersed into the sludge. The immersion depth was selected as 20 mm in order to avoid sucking and/or mixing air into the sludge samples.

In the ultrasonic sludge disintegration experiments, 500 mL, 1000 mL and 2000 mL of sludge volume (having the power density of 0.059 W/mL, 0.114 W/mL and 0.228 W/mL, respectively) was subjected to different sonication duration of 5 min, 10 min, 20 min, 30 min, 60 min and 90 min.

The experiment conditions matrix for ultrasonic sludge disintegration experiments is shown in Table 3.5.

Table 3.5. The experiment conditions matrix for ultrasonic sludge disintegration.

Time (min)	**Volume (mL)**		
	500	1000	2000
5	×	×	×
10	×	×	×
20	×	×	×
30	×	×	×
60	×	×	×
90	×	×	×

After sludge samples were disintegrated by ultrasonication, TS, TVS, MLSS, MLVSS, SV, SCOD, supernatant turbidity, viscosity, CST, pH and particle size of samples was measured. Settling velocity (SV) tests were performed in glass cylinders (by using 100 mL of sample). After 6 h of settling, supernatant turbidity was analyzed by collecting of necessary amount of sample from the supernatant. Furthermore, 50 mL of sonicated samples were centrifuged at 5000 rpm for 15 min to get the supernatant. Soluble chemical oxygen demand (SCOD) was measured in the supernatant of centrifuged samples.

Disintegration degree (DD) and specific energy (SE) are two important parameters to quantify the sludge disintegration. They are often used in the studies to describe the decomposition of the sludge by ultrasound irradiation. Therefore, DD and SE are calculated in this study as following:

Disintegration Degree. The degree of disintegration (DD_{COD}) was defined by Müller and Pelletier as the comparison between SCOD after ultrasonic pretreatment and the maximum SCOD obtained by alkaline hydrolysis ($SCOD_{NaOH}$), as presented in Equation 3.4 (Yan et al., 2010, El-Hadj et al., 2007). For alkaline hydrolysis, sludge was mixed with NaOH (in the ratio of 1 mol/L) for 24 h at room temperature as described by Bougrier et al. (2005).

$$DD_{COD} = \left[\frac{SCOD_{Ultrasonic} - SCOD_0}{SCOD_{NaOH} - SCOD_0} \right] \times 100\% \qquad (3.4)$$

where,

$SCOD_{Ultrasonic}$: the COD in the supernatant of the sonicated sample (mg/L)

$SCOD_0$: the COD in the supernatant of the original (untreated) sample (mg/L)

$SCOD_{NaOH}$: the COD in the supernatant of the alkaline hydrolyzed sample (mg/L)

Specific Energy. The specific energy input is defined as the energy input per unit of sludge (as TS) to achieve a certain degree of disintegration (Khanal et al., 2007). Specific Energy (SE) is a function of ultrasonic power, ultrasonic duration, and volume of sonicated sludge and TS concentration, and can be calculated using the following Equation (Bougrier et al., 2005):

$$SE = \frac{P \times t}{V \times TS_0} \tag{3.5}$$

where,

SE : the specific energy input in kWs/kg TS (kJ/kg TS)

P : the ultrasonic power in kW

t : the ultrasonic duration in seconds

V : the volume of sonicated sludge in liters

TS_0 : the total solids concentration in kg/L

3.2.1.2. Membrane Bioreactors (MBRs). In this laboratory-scale study, a flat sheet type PVDF (polyvinylidene difluoride) microfiltration (MF) membrane (Shanghai Megavision Membrane Engineering and Technology Co. Ltd., China.) was used. The pore size of the membrane was 0.3 μm and the effective surface area of membrane panel was 0.064 m^2. Characteristics of the membrane panel are given in Table 3.6.

Table 3.6. Characteristics of the membrane panel used in the study.

Membrane Panel	
Shape	Flat sheet
Membrane material	PVDF
Membrane frame material	PVC
Panel effective membrane area	0.064 m^2
Pore size	0.3 μm
Dimensions (width×length×thickness)	170×300×16 mm

The MBR system was constructed to treat municipal wastewater. In order to minimize membrane fouling, an aeration system was located just below the membrane panel. Due to the vigorous aeration for fouling control, the dissolved oxygen (DO) in the reactor was maintained at relatively high level (4-5.5 mg/L). The air was fed into the reactor with KnF Neuberger compressor. The feed water was also aerated before giving to the reactor. The effluent (permeate) was drawn from the membrane by a peristaltic pump (Heidolph PD 5001 Peristaltic Pump). The experimental set-up is shown in Figure 3.3.

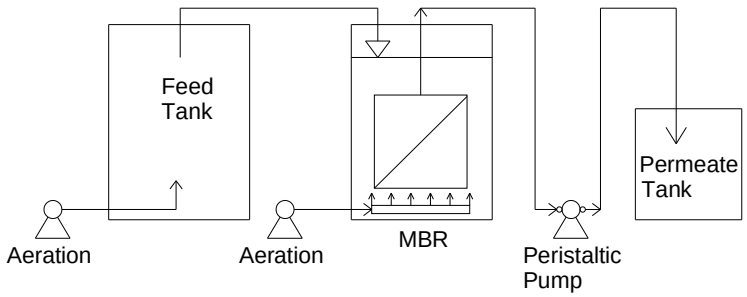


Figure 3.3. Schematic representation of experimental set-up for MBR.

The volume of aerobic reactor was 18.5 L with the working volume of 15 L. The MBR system was operated with flow rate of 3 L/day. The hydraulic retention time (HRT) of MBR was 5 d. There was no excess sludge wastage from the system during the experimental period. The MBR system was operated semi-continuously.

In this study, the membrane bioreactor-ultrasound (MBR-US) combined systems were operated in order to investigate the effect of ultrasonic sludge disintegration on the excess sludge minimization and on the MBR performance. The process configuration of MBR is shown in Figure 3.4.



Figure 3.4. The process configuration of MBR.

In the study, the membrane bioreactor-ultrasound (MBR-US) combined systems were operated in two cases.

For the first case, the MBR-US system was operated at different sonication conditions such as power density, sludge sonication ratio, sludge sonication time and sonication frequency. In the first condition (MBR-US1), MBR sludge was ultrasonically disintegrated for 15 minutes at the ratio of 1:15 (v:v) and power density of 0.114 W/mL, i.e. 1000 mL of MBR sludge was sonicated and returned directly into the MBR-US1 system. This batch sonication process was performed daily. The reactor was operated 10 days. In the second condition (MBR-US2), 1:30 (v:v) of MBR sludge was sonicated daily for 5 minutes at the power density of 0.228 W/mL, i.e. 500 mL of MBR sludge was sonicated and then fed into the MBR-US2 system. The operation time for this run was also 10 days. In this case, the MBR system was operated as a control reactor, CMBR, at first and then as MBR-US1 and MBR-US2.

For the second case, a 15-L sequential batch reactor, a sequential batch reactor-ultrasound (SBR-US) combined system and a membrane bioreactor-ultrasound (MBR-US3) combined system were operated in parallel. The sequential batch reactor was the control reactor, CR, and fed only with wastewater. The SBR-US and MBR-US3 combined systems were operated in the same manner: these systems fed with wastewater and ultrasonically disintegrated excess sludge. In this case, to compare the efficiency of MBR with SBR in terms of sludge minimization and effluent quality, the SBR-US combined system was additionally operated. CR and SBR-US did not include membrane panel. The sonication conditions of sludges fed into SBR-US and MBR-US3 was same. For these reactors, instead of daily sonication, the sludge was sonicated as needed basis. 1:30 (v:v) reactor sludge was sonicated for 5 min at power density of 0.228 W/mL. The operation time was 21 days. The schematic representation of second case is depicted in Figure 3.5.

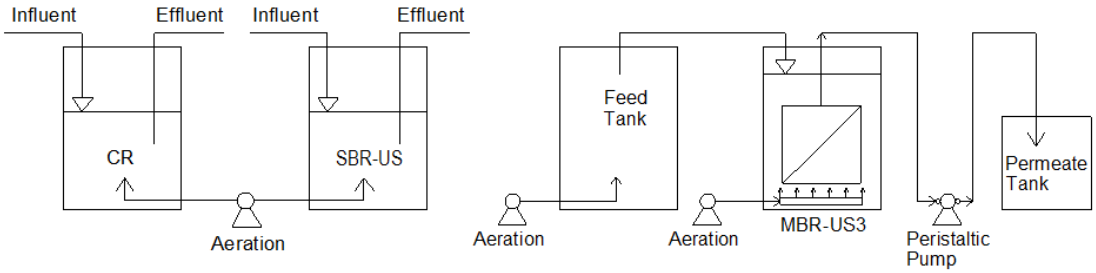


Figure 3.5. Schematic representation of reactors in the second case.

There was no excess sludge wastage from the all reactors during the experimental period, therefore the solids retention time (SRT) of reactors were infinite (∞). For both cases, sonicaiton and operational conditions of reactors are summarized in the Table 3.7.

Table 3.7. Sonicaiton and operational conditions of reactors.

Sonication & operational conditions	**First Case**			**Second Case**		
	CMBR	**MBR-US1**	**MBR-US2**	**CR**	**SBR-US**	**MBR-US3**
Working volume (L)	15	15	15	15	15	15
Flow rate (L/d)	3	3	3	3	3	3
HRT (d)	5	5	5	5	5	5
SRT (d)	∞	∞	∞	∞	∞	∞
Power density (W/mL)	0	0.114	0.228	0	0.228	0.228
Sludge sonication ratio (v:v)	0	1:15	1:30	0	1:30	1:30
Sonicated sludge volume (mL)	0	1000	500	0	500	500
Sludge sonication time (min)	0	15	5	0	5	5
Sonication time interval	0	Daily	Daily	0	As needed	As needed
Operation time (d)	10	10	10	21	21	21

3.2.1.3. Determination of Sonicated Sludge Ratio. Three laboratory-scale sequential batch reactors (SBRs) were operated in parallel at different ratios of sonicated sludge. One of these three reactors was used as control reactor (CR) and two of them were pretreatment reactors (R1 and R2) in which a part of the sludges in the reactors was sonicated and returned back in them for the purpose of sludge minimization. The reactors contained an aeration device to maintain the dissolved oxygen (DO) in the reactors at the level of 4-5 mg/L. The air flow inside the reactor was controlled by air pumps. The SBRs were operated at a cycle of 24 hours. Each cycle consisted of four phases: inflow phase (filling), aeration (phase), settling phase (sedimentation) and outflow phase. During each cycle, 0.6 L wastewater was exchanged in all reactors to keep the process going. The experimental set-up for SBRs is shown in Figure 3.6.

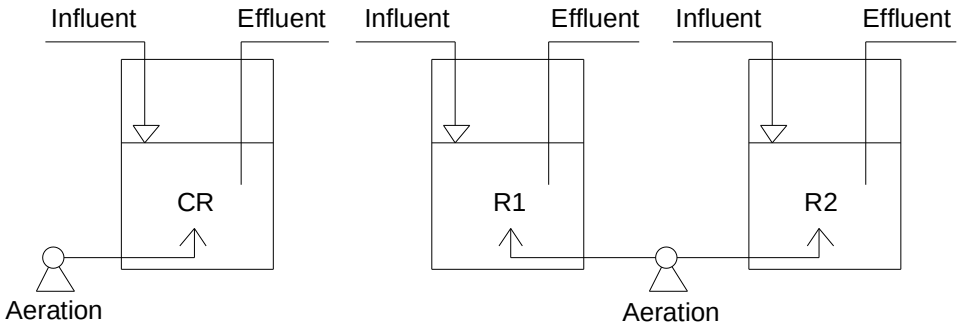


Figure 3.6. Schematic representation of experimental set-up for SBRs.

The volume of each SBRs was 5 L with 3-L working volume. The SBR systems were operated with flow rate of 0.6 L/d. The HRT of SBRs was 5 d. There was no excess sludge wastage from the systems during the experimental period. After 3 days of operation, sonication was applied to sludges fed into R1 and R2 at a sonication frequency of 1 batch/3 days. Sonication was applied between the phases of inflow and aeration. All analyses were done before sonication application.

For R1, reactor sludge was ultrasonically disintegrated for 5 min at the ratio of 1:60 (v:v) and power density of 0.92 W/mL, i.e. 50 mL of reactor sludge was sonicated and returned directly into the R1. This batch process was performed once three days. The operation time was 13 days.

For R2, 1:30 (v:v) of reactor sludge was sonicated once third day for 5 min at the power density of 0.84 W/mL, i.e. 100 mL of reactor sludge was sonicated and then fed into the R2 system. The operation time for R2 was 13 days. The operational conditions for each SBRs are listed in Table 3.8.

Table 3.8. Operational conditions for SBRs systems.

Operational Condition	**CR**	**R1**	**R2**
Working volume (L)	3	3	3
Flow rate (L/d)	0.6	0.6	0.6
HRT (d)	5	5	5
SRT (d)	∞ (No wasting)	∞ (No wasting)	∞ (No wasting)
Power density (W/mL)	0	0.92	0.84
Sludge sonication ratio (v:v)	0	1:60	1:30
Sonicated sludge volume (mL)	0	50	100
Sludge sonication time (min)	0	5	5
Sonication frequency	0	Once 3 days	Once 3 days
Operation time (day)	13	13	13

3.2.2. Analytical Methods

3.2.2.1. Chemical Analyses. Chemical analyses used in the study are described as follows:

Chemical Oxygen Demand (COD) and Soluble Chemical Oxygen Demand (SCOD). These were performed using the Method 5220D (closed reflux, colorimetric method) in Standard Methods (APHA, 1998). It was determined via ignition to 150 ^{o}C followed by monitoring of the absorbance at 600 nm. For SCOD analysis, supernatant of samples were used which are obtained by centrifugation samples at 5000 rpm for 15 min. All analyses were run in two parallel samples.

Total Kjeldahl Nitrogen (TKN). Sample digestion (pretreatment) was performed using the General Digesdahl Digestion procedure via ignition 25 mL sample to 440 ^{o}C followed by dilution it to 100 mL as defined in Hach DR/2010 Procedures Manual (Hach, 1997). Sample pretreatment were carried out at Digesdahl Digestion Apparatus which is shown in Figure 3.7 (Hach, 1999). Digested sample was analyzed using the Nessler Method (Method 8075) for TKN measurement according to Hach DR/2010 Procedures Manual (Hach, 1997). TKN concentrations were calculated using the following Equation (Hach, 1997):

$$TKN\ (mg/L) = \frac{A \times 75}{B \times C} \tag{3.6}$$

where,

A : mg/L reading from instrument

B : mL sample amount (sample volume used for digestion)

C : mL analysis volume

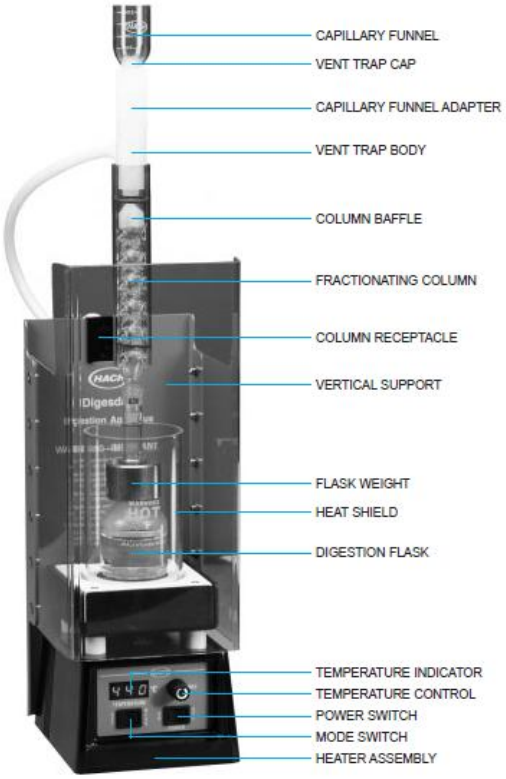


Figure 3.7. Digesdahl Digestion Apparatus used in the study (Hach, 1999).

Total Phosphorus (TP). Same digested sample prepared as mentioned above (in TKN measurement) was used for TP experiment. Digested (pretreated) sample was analyzed for TP measurement using Ascorbic Acid Method (Method 8048) in accordance with Hach DR/2010 Procedures Manual (Hach, 1997). PhosVer 3 Phosphate Powder Pillow kits were used for this analysis. TP concentrations were calculated using the following Equation (Hach, 1997):

$$Total\ P\ (mg/L) = \frac{A \times 2500}{B \times C} \tag{3.7}$$

where,

A : mg/L reading from instrument

B : mL sample amount (sample volume used for digestion)

C : mL analysis volume

Ammonia Nitrogen (NH_3-N). The NH_3-N concentrations of samples were analyzed using the Nessler Method (Method 8038) with Hach DR/2010 spectrophotometer. The analyses were carried out using Mineral Stabilizer, Polyvinyl Alcohol Dispersing Agent and Nessler Reagent following the procedures outlined in Hach DR/2010 Procedures Manual (Hach, 1997).

Nitrite Nitrogen (NO_2^--N). These were performed with standard NitriVer 3 Nitrite Reagent Powder Pillow kits provided by Hach and according to the Diazotization Method (Method 8507) as described in Hach DR/2010 Procedures Manual (Hach, 1997).

Nitrate Nitrogen (NO_3^--N). These analyses were performed with standard NitraVer 5 Nitrate Reagent Powder Pillow kits provided by Hach and according to the Cadmium Reduction Method (for high range and medium range, Method 8039 and Method 8171, respectively) as described in Hach DR/2010 Procedures Manual (Hach, 1997). For low range analyses (Method 8192), NitraVer 6 Nitrate Reagent Powder Pillow and NitriVer 3 Nitrite Reagent Powder Pillow kits were used.

Mixed Liquor Suspended Solids (MLSS) and Mixed Liquor Volatile Suspended Solids (MLVSS). MLSS and MLVSS were determined in accordance with the Standard Methods (APHA, 1998) using the Method 2540D and 2540E, respectively.

Total Solids (TS) and Total Volatile Solids (TVS). TS and TVS were measured according to the Standard Methods (APHA, 1998) using the Method 2540B and 2540E, respectively.

3.2.2.2. Physical Analyses. Physical analyses used in the study are described as following:

Capillary Suction Time (CST). The rate of dewaterability was determined by measuring the capillary suction time (CST), using the capillary suction apparatus is shown in the Figure 3.8. The capillary suction apparatus consisted of a sludge column contained in the sample cylinder which is centered in the middle in the two of concentric electrodes at diameter D1 and D2 resting on a Whatman-17 filter paper. A timing device is started when the waterfront reaches the inner electrode (1) at D1 and is stopped when the water reaches the outer electrode (2) at D2. The time elapsed is the CST (Yin et al., 2004; Navaneethan, 2007; Moonkhum, 2007). The CST apparatus used in this study was manufactured by Venture Innovations Inc. (LA, USA). CST filtration test papers were obtained from Triton Electronics Ltd.

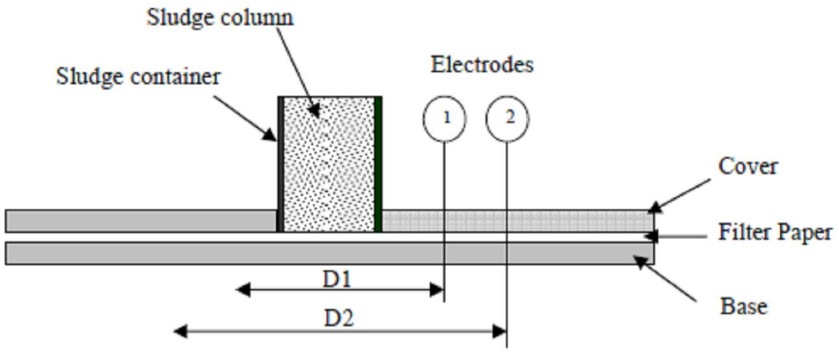


Figure 3.8. Schematic representation of CST apparatus (Moonkhum, 2007).

Viscosity. Viscosimetry gives information on the liquor behaviour when it is flowing, i.e. on the shear stress (Pa)–shear rate (s^{-1}) relation $\tau_{xy} = \tau_{xy}(\gamma)$ (Van Kaam et al., 2008). In this study, viscosity measurements were carried out using a Brookfield RVDV-I PRIME digital viscometer.

For making measurements, viscometer was turned on, leveled and autozeroed. The level was adjusted using the three feet on the bottom of the base and confirmed using the bubble on the top of the head. The level was set prior to autozeroing and was checked prior to each measurement. 500 mL of sludge samples were filled in 600 mL Low Form Griffin Beaker. Measurements were performed using RV Guardleg and #2 spindle (S02) of the RV spindle set. In the viscosity experiments, the operating speed was 100 rpm. All samples were measured in triplicate.

Particle size distribution. Particle size analyses were carried out using Malvern Mastersizer 2000 (with the wet dispersion unit of Hydro2000MU). Water was used as dispersant liquid and its refractive index was 1.33. Sludge samples had refractive index of 1.5. The stirrer and pump speed were kept at 600 rpm, which is the minimum pump/stirrer speed available, to minimize the damage of sludge particles. Sampling was made using a pasteur pipette with as wide an end-opening as possible (Gonze et al., 2003). 1000-mL low form beaker which had 800 mL deionized water in it was used as dispersion tank. For analysis, each sample was diluted in this dispersion tank and introduced into the measuring cell. Each sample was analyzed in triplicate.

The principle of operation is as following: The dispersion unit uses a 1000-mL “low form” beaker that holds the sample/dispersant liquid. A stirrer, controlled from the keypad, agitates the sample and stops it from settling or separating out. The pump, controlled from the keypad, forces the sample from the “to cell port” of the dispersion unit to the flow cell located in the optical bench (Mastersizer 2000), via the sample tubing. The sample is pumped through the flow cell and then returns to the beaker via the sample tubing and the “from cell port” (Malvern, 2007).

Turbidity. The supernatant turbidity of sludge samples (after 6 h of gravity sedimentation and collection of sample from the supernatant) and the turbidity of reactors' permeate was determined by measuring their light transmission with a Hach 2100P Turbidimeter. Experiments were carried out using 20 mL-cells having working volume of 16 mL.

Settling Velocity (SV). For ultrasonic sludge disintegration experiments, sonicated sludge samples (100 mL) were transferred to a graduated cylinder with a diameter of 27 mm and working height of 170 mm. SV was then determined from the height of the sludge that had settled after a certain fixed interval of time. Although a strong wall effect exists in settling tests conducted in tubes, this effect is immaterial however for relative comparison of different SV values obtained under identical conditions (Feng et al., 2009).

Sludge Volume Index (SVI). For reactors, 30-min settled sludge volume was performed according to the Standard Methods (APHA, 1998) using the Method 2710C. Sludge was settled for 30 min in a 1-L graduated cylinder and determined settled sludge volume was used in the calculation of sludge volume index (SVI). SVI was determined in accordance with the Standard Methods (APHA, 1998) using the Method 2710D.

4. RESULTS AND DISCUSSION

4.1. The Calibration of Ultrasonicator

To determine the optimum power output and amplitude values for the volumes of 500 mL, 1000 mL and 2000 mL, ultrasonic system was calibrated by calorimetry.

4.1.1. Determination of Optimum Power Output and Amplitude

In the calorimetric dosimetry experiments, different power output values and amplitude values were studied for each volume of 500 mL, 1000 mL and 2000 mL. Batch experiments were conducted in beakers as described in Section 3.2.1.1.

The results of calorimetric analyses showed that the optimum power output for all volumes was the same and it was 250 W. However, optimum amplitude values were determined as 60%, 80% and 70% for the volumes of 2000 mL, 1000 mL and 500 mL, respectively. The results suggested that there was no correlation between the sonicated volume and the amplitude value. Therefore, in order to avoid unnecessary loss of energy, calibration experiments should be conducted for each volume to be studied before all ultrasonically aided experiments. The optimum results for this study are listed in Table 4.1.

Table 4.1. Optimum power output and amplitude values for each selected volume.

Volume (mL)	Power Output (W)	Amplitude (%)
2000	250	60
1000	250	80
500	250	70

All experimental results related to the calibration of ultrasonicator are given in Appendix A.

4.1.2. Determination of Ultrasonic Power, Power Density and Power Intensity

Ultrasonic power, power density and power intensity was calculated according to Equation 3.1, 3.2 and 3.3, respectively. The results of calorimetric analysis showed that the power density in the glass beaker was 0.059 W/mL, 0.114 W/mL and 0.228 W/mL for the solution volume of 2000 mL, 1000 mL and 500 mL, respectively. All determined optimum conditions are summarized in Table 4.2. It was observed that as sample volume increased, the power density decreased. This is due to the fact that the energy is more easily delivered into the solution when the sample volume is less.

Table 4.2. Ultrasonic power, power density and power intensity for each selected volume.

Volume (mL)	**Ultrasonic Power (W)**	**Power Density (W/mL)**	**Power Intensity (W/cm^2)**
2000	118.0	0.059	24.04
1000	113.8	0.114	23.18
500	114.0	0.228	23.23

Most of the energy delivered into the solution (deionized water) by sonication is dissipated as heat. The temperature rise with time in glass beaker (filled with deionized water) for each selected power density is shown in Figure 4.1.

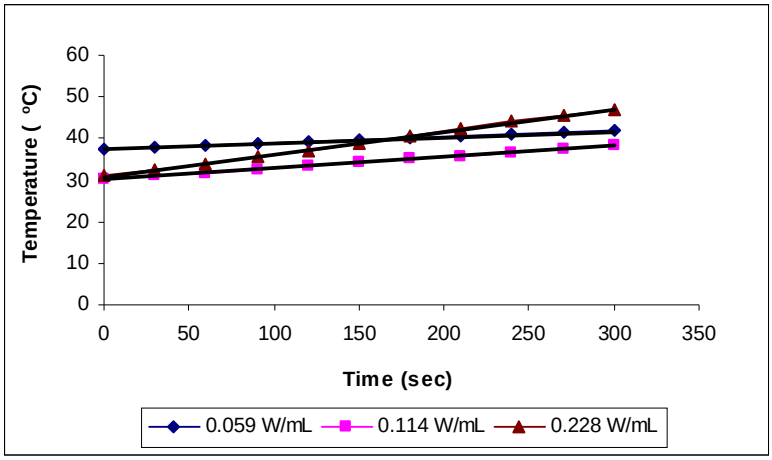


Figure 4.1. The effect of time on heat release for each selected power density.

4.2. Ultrasonic Sludge Disintegration

On this step of the study, batch tests were carried out to investigate the influence of sonication on sludge properties.

4.2.1. Untreated Sludge Characteristics

Sludge samples taken for ultrasonic disintegration experiments were collected from the aeration tank of Paşaköy Advanced Biological Wastewater Treatment Plant. Before ultrasonic sludge disintegration experiments, untreated sludge were analyzed for characterization. The characterization of untreated sludge is given in Table 4.3. After sludge samples were disintegrated by ultrasonication, it was waited until its temperature reached to room temperature. Then sludge samples were analyzed for TS, TVS, MLSS, MLVSS, settling velocity (SV), SCOD, supernatant turbidity, viscosity, CST and particle size. The results were given in the following sections.

Table 4.3. The characterization of untreated sludge for ultrasonic sludge disintegration experiments.

Parameter	Value	Unit
TS	8130	mg/L
TVS	4914	mg/L
MLSS	7320	mg/L
MLVSS	4510	mg/L
MLVSS/MLSS	0.62	-
SVI	130	mL/g
TCOD	6841.5	mg/L
SCOD	51.8	mg/L
Supernatant Turbidity	12.5	NTU
Viscosity	22.8	mPa s
CST	20.5	s
pH	7.15	-

4.2.2. Determination of Specific Energy

In the ultrasonic sludge disintegration experiments, 2000 mL, 1000 mL and 500 mL of sludge volumes (having the power density of 0.059 W/mL, 0.114 W/mL and 0.228 W/mL, respectively) were subjected to different sonication duration of 5 min, 10 min, 20 min, 30 min, 60 min and 90 min. The specific energy input is defined as the energy input per unit of sludge (as TS) to achieve a certain degree of disintegration (Khanal et al., 2007). Specific Energy (SE) is a function of ultrasonic power, ultrasonic duration, and volume of sonicated sludge and TS concentration. The SE values were calculated using the Equation 3.5. SE values for each sample are given in Table 4.4.

Table 4.4. SE values for each sample volume and sonication duration.

Sample Volume (mL)	**Power Density (W/mL)**	**Sonication Duration (min)**	**SE (kJ/kg TS)**
2000	0.059	5	2177
		10	4354
		20	8708
		30	13063
		60	26125
		90	39188
1000	0.114	5	4199
		10	8399
		20	16797
		30	25196
		60	50391
		90	75587
500	0.228	5	8413
		10	16827
		20	33653
		30	50480
		60	100959
		90	151439

SE increased with the increasing of power density and sonication duration as can be seen from Table 4.4. This is due to the fact that SE is directly proportionate to ultrasonic power and ultrasonic duration. Furthermore, SE decreased with the increasing of sludge volume because SE input is inversely proportional to the volume of sonicated sludge.

4.2.3. Effect of Ultrasonic Disintegration on Sludge Temperature

In the ultrasonic experiments, almost all the mechanical energy produces heat and most of the energy delivered into the solution by sonication is dissipated as heat (Mason and Peters, 2002). Therefore, during the course of sonication, the temperature of the system rose gradually. Effect of sonication duration on sludge temperature is given in Figure 4.2. It was observed that the temperature of sludge samples increased with power density and time. For instance, at the ultrasonicaiton time of 30 min, temperature rise was 24.2, 42.5 and 53.4 for the power density of 0.059 W/mL, 0.114 W/mL, 0.228 W/mL, respectively. The rise of temperature helps to ultrasonic disintegration (Huan et al., 2009). Therefore, disintegration experiments in this study were carried out without cooling.

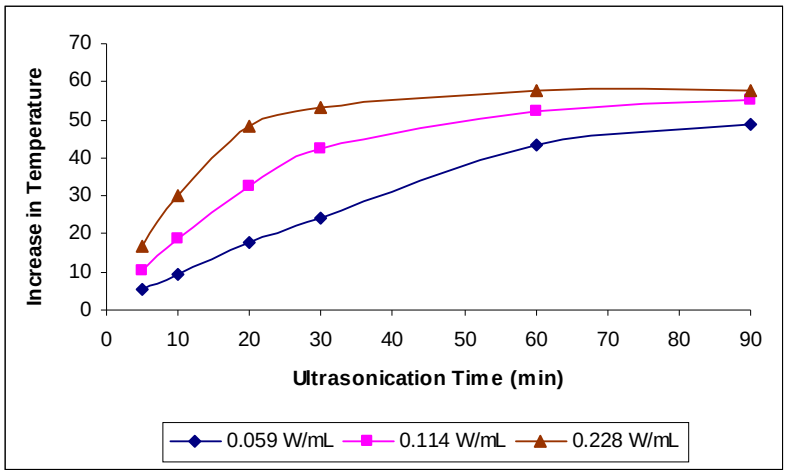


Figure 4.2. Temperature variations with ultrasonication time at various power densities.

Chu et al. (2001) noted that two effects occur simultaneously in ultrasonic treatment: vigorous agitation caused by tiny bubble formation and explosion, and the increase in the bulk temperature. The temperature effect on sludge can clearly be seen from Figure 4.2.

4.2.4. Effect of Ultrasonic Disintegration on SCOD

For each experiment, while sonicaiton time and power density increased, total COD was maintained constant. Similar results were obtained by some authors (Bougrier et al., 2005, El-Hadj et al., 2007). While sonication does not change TCOD (total COD), it improves the soluble organic concentration expressed as SCOD. And it is also known that the organic material solubilized by the ultrasound may be intracellular material (cytoplasm), but also extracellular organic compounds contained in the bacterial flocs (Gonze et al., 2003). Figure 4.3 shows the change of SCOD with sonication time. By applying sonication, cells underwent lysis and organic compounds were released into the liquid phase (Bougrier et al., 2005) and this caused an increase in SCOD. Figure 4.3 indicates SCOD increased gradually and the rate of increase of SCOD became slower with increasing sonication time and ultrasonic density. Even for the lowest power density of 0.059 W/mL and sonication time of 5 min, SCOD increased too much: from 51.8 mg/L (which is the SCOD of untreated sludge) to 467.5 mg/L.

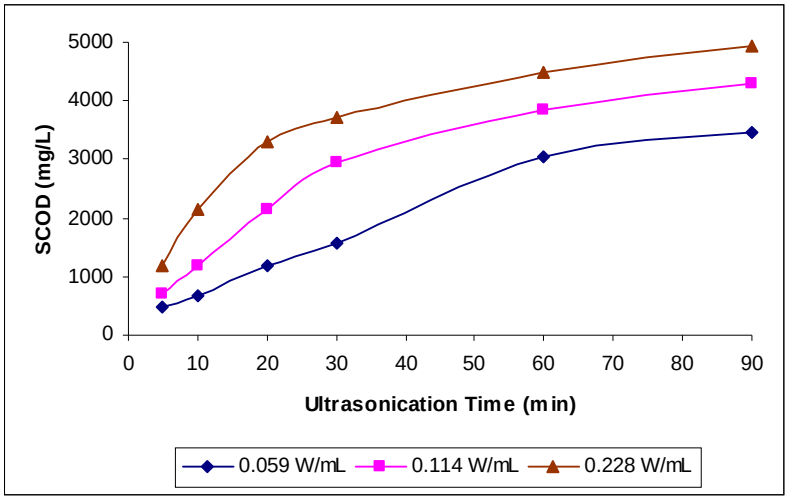


Figure 4.3. Variation of SCOD with ultrasonication time at various power densities.

From the obtained SCOD values disintegration degree (DD_{COD}) was calculated according to Equation 3.4. DD_{COD} is directly proportional to SCOD, i.e. SCOD increases with the increasing value of DD_{COD}. The values of calculated DD_{COD} are given in Table 4.5.

Table 4.5. The values of disintegration degree (DD_{COD} %).

Power Density (W/mL)	**Sonication Duration (min)**	**SE (kJ/kg TS)**	**DD_{COD} (%)**
0.059	5	2177	14.0
	10	4354	20.9
	20	8708	38.1
	30	13063	51.6
	60	26125	100.4
	90	39188	115.2
0.114	5	4199	22.1
	10	8399	38.5
	20	16797	70.7
	30	25196	97.3
	60	50391	127.7
	90	75587	143.3
0.228	5	8413	37.7
	10	16827	71.0
	20	33653	109.4
	30	50480	123.6
	60	100959	149.9
	90	151439	164.0

In some cases, DD_{COD} and specific energy (SE) are compared in order to evaluate the solubilization of sludge. The variations of SCOD and DD_{COD} with SE are presented in Figure 4.4. The transport of COD from the particulate fraction to the soluble fraction was clearly enhanced by increasing the SE (El-Hadj et al. 2007). SCOD increased strongly for specific supplied energy between 0 and 25000 kJ/kg TS: it increased from 51.8 mg/L to 2940 mg/L. For higher SE values, the rate of increase of SCOD decreased. For SE values higher than 26000 kJ/kg TS, the SCOD of ultrasonically disintegrated sludge samples was higher than that of alkaline disintegrated sludge sample ($SCOD_{NaOH}$=3020.7 mg/L), leading to disintegration degrees higher than 100 %.

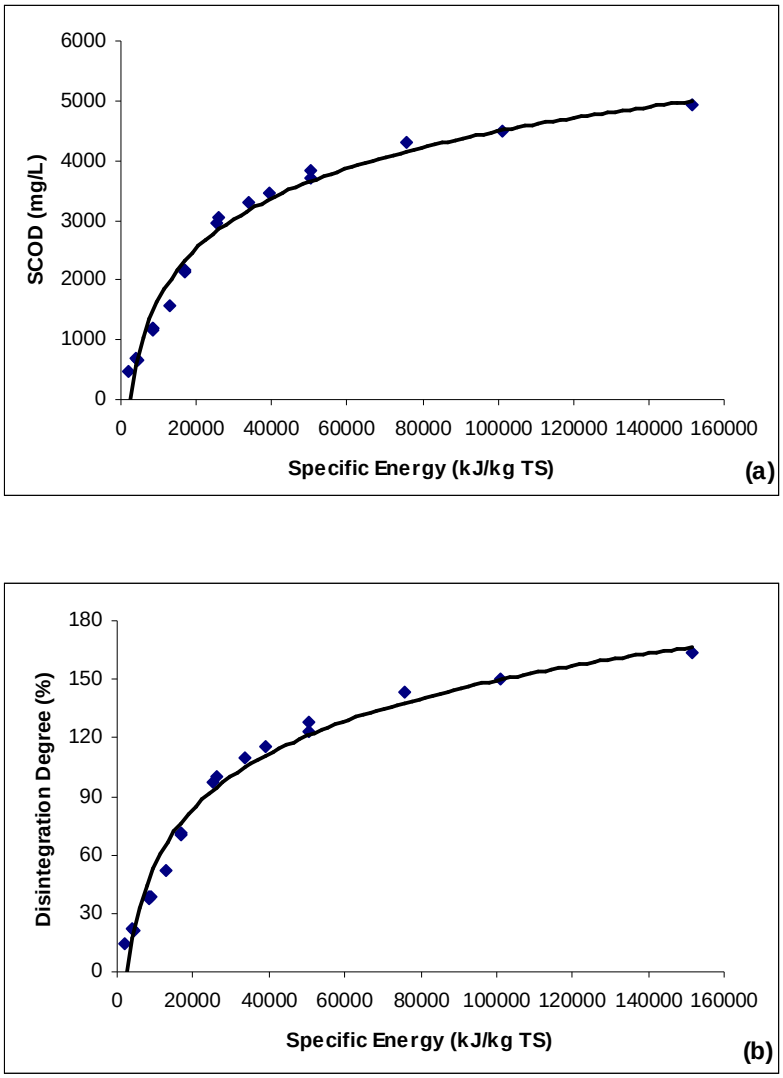


Figure 4.4. Variation of (a) SCOD and (b) disintegration degree with specific energy.

For SE of about 2000 kJ/kg TS, the disintegration degree (DD_{COD}) was 14%. This value is in the range obtained by Bougrier et al. (2005) for SE=1000 kJ/kg TS and Lehne et al. (2001) for SE=3000 kJ/kg TS. The SE of about 2000 kJ/kg TS was not used to lyse cells; it was used just to break flocs (Bougrier et al., 2005). For SE higher than 2000 kJ/kg TS, DD_{COD} rose strongly. Furthermore, the DD_{COD} of ultrasonically disintegrated sludges calculated according to Müller formula exceeded 100% for SE values higher than 26000 kJ/kg TS. It seems that for SE values higher than 26000 kJ/kg TS, ultrasonication is a more effective disintegration method than NaOH alkaline treatment.

4.2.5. Effect of Ultrasonic Disintegration on Sludge Settling Properties

For settling velocity (SV) tests, 100 mL-graduated cylinders with a diameter of 27 mm and working height of 170 mm were used. In order to decide the sedimentation time, the settling velocity of untreated sludge was recorded for 24 h at first. SV was determined from the height of the sludge that had settled after a certain fixed interval of time. The SV of untreated sludge after 24 h is shown in Figure 4.5.

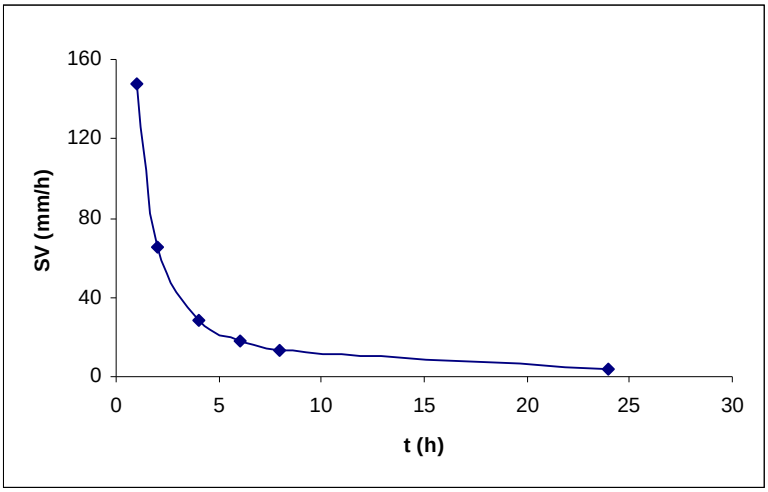


Figure 4.5. The SV of untreated sludge after 24 h.

As can be seen from Figure 4.5, there was no extreme change after 6 h. Therefore the analyses of SV were conducted for 6 h. The wall effect exists for SV tests due to narrow settling tube diameter. However, in the relative comparison of the SV values, the wall effect can be ignored since all the settling tests were done under identical conditions (Chu et al., 2001; Feng et al., 2009).

After the untreated sludge disintegrated ultrasonically, the sludge samples were analyzed for settling velocity (SV). The effect of ultrasonic disintegration on the sludge settling velocity is shown in Figure 4.6. The results were given in terms of specific energy (SE). The figure includes summary of the SV tests results. All of the SV test results are given in Appendix C.

Ultrasonically disintegrated sludge with an SE of about 2000 kJ/kg TS had a SV value of 139 mm/h after 1 h, which was faster than 121 mm/h for the untreated sludge. The SV of the ultrasonically disintegrated sludge with SE higher than 2000 kJ/kg TS was slower than that of the untreated sludge. Increasing the SE decreased the SV. Also, SV was almost undetectable for the SE higher than 25000 kJ/kg TS. It was clear that ultrasonic disintegration for SE higher than 2000 kJ/kg TS deteriorated the sludge settling properties. In this study, in order to enhance the sludge settling properties, the optimum SE of disintegration process was found to be 2000 kJ/kg TS. Feng et al. (2009) suggested that a SE of 1000 kJ/kg TS was optimum energy level for improving the settleability of sludge by applying ultrasonic disintegration. However, in their study the SE between 1000 kJ/kg TS and 5000 kJ/kg TS were not tested.

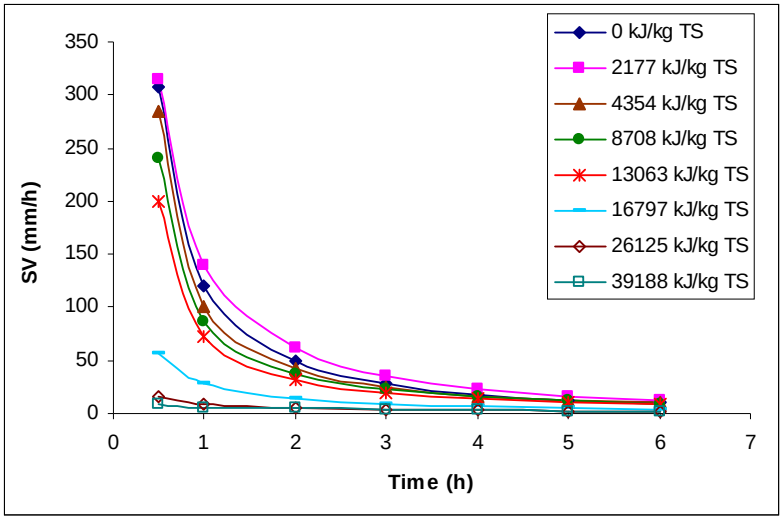


Figure 4.6. Variation of sludge settling velocity with specific energy.

Huan et al. (2009) stated that high-energy ultrasonic pretreatment can disrupt flocs and increase the fine particles. These micro particles, which have a density almost equal to that of water, settle very slowly (Feng et al., 2009). Therefore, only low-energy ultrasonic pretreatment is generally used to improve sludge settleability. It seems that ultrasonic sludge disintegration for SE of about 2000 kJ/kg TS improves sludge settleability.

4.2.6. Effect of Ultrasonic Disintegration on Supernatant Turbidity

The supernatant turbidity is thought as indirect indicator of sludge disintegration. Hence, the supernatant turbidity was analyzed in order to see the effect of sonication on sludge solubilization. The samples were collected from the supernatant of sludge after 6 h of gravity sedimentation (after SV measurement). The effect of sonication on supernatant turbidity is given in Figure 4.7 and Figure 4.8.

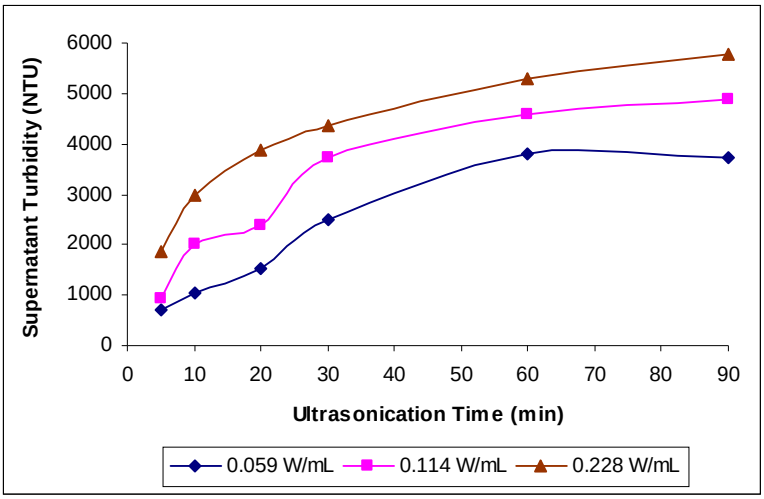


Figure 4.7. Variation of turbidity with ultrasonication time at various power densities.

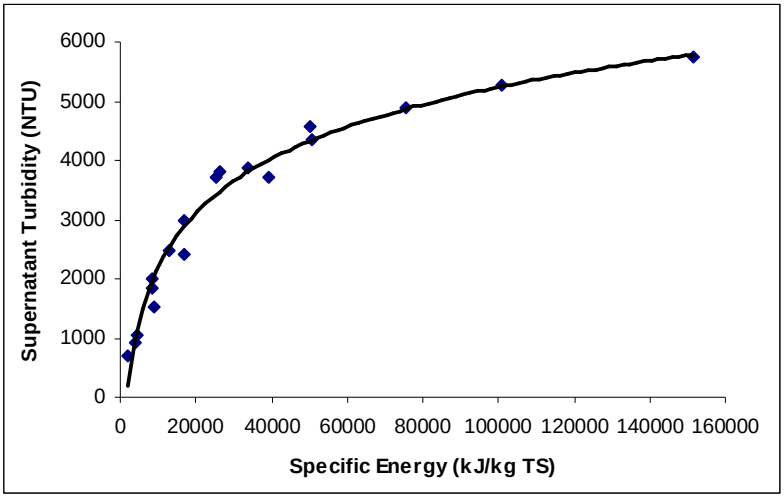


Figure 4.8. Variation of supernatant turbidity with specific energy.

Form the Figure 4.7 and Figure 4.8 (when compared to Figure 4.3 and 4.4.a) it can be seen that SCOD and supernatant turbidity of ultrasonically disintegrated sludge had same trend. It was observed that supernatant turbidity was increased with the increasing SE. Supernatant turbidity increased strongly from 0 NTU to 3727 NTU for SE values between 0 and 25000 kJ/kg TS. For higher SE values, the rate of increase of supernatant turbidity decreased. The turbidity of supernatant increased from 3727 NTU to 5760 NTU for SE values between 25000 kJ/kg TS and 150000 kJ/kg TS. The increasing supernatant turbidity can be explained with the increasing micro particles, released from sludge flocs through disintegration process in the supernatant. It is known that as SE increases, much more small particles are produced. These micro particles, which have a density almost equal to that of water, settle very slowly (Feng et al., 2009).

4.2.7. Effect of Ultrasonic Disintegration on Solids Concentration

4.2.7.1. Effect of Ultrasonic Disintegration on Total Solids. During ultrasonic treatment of sludge, both TS and VS were maintained almost constant averaging 7800 mg/L and 4600 mg/L (Figure 4.9). Hence, the evaporation and mineralization phenomenon was not induced by the ultrasonic disintegration. This result was in agreement with the results of the studies performed by Bougrier et al. (2005), El-Hadj et al. (2007), Feng et al. (2009). The results also showed that TS and VS were not related with SE.

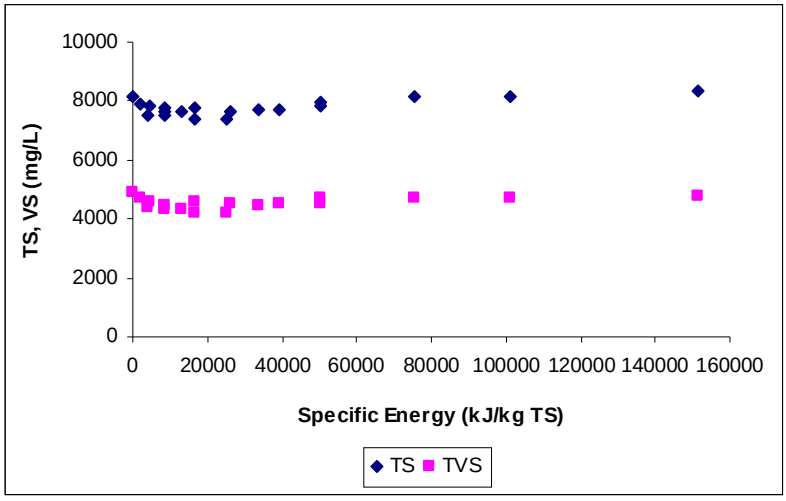


Figure 4.9. Variation of TS and VS with specific energy.

4.2.7.2. Effect of Ultrasonic Disintegration on MLSS. Although total solid concentration did not change with the application of ultrasonic pretreatment, the disintegration of sludge was reflected in the decreasing MLSS. The variations of suspended solid concentrations are displayed in Figure 4.10. MLSS concentrations of the ultrasonically disintegrated sludge decreased until the SE of about 50000 kJ/kg TS. For the SE of about 50000 kJ/kg TS, 18% of the MLSS and 20% of the MLVSS concentration was oxidized and solubilized. The increase in MLSS concentration for SE values between 50000 kJ/kg TS and 100000 kJ/kg TS could be explained with the reflocculation phenomenon. Bougrier et al. (2005) and Gonze et al. (2003) observed the reflocculation effect in their particle size experiments. This reflocculation may occur after ultrasonic disintegration, because of the release of intracellular or extracellular material (Bougrier et al., 2005). The released organic materials and polymers by ultrasonic pretreatment may act as flocculent during ultrasonication.

While MLSS and MLVSS concentrations varied during the ultrasonic disintegration of sludge, the ratio of MLVSS/MLSS did not undergo a significant change. The value of MLVSS/MLSS was almost the same around 0.60 and 0.64.

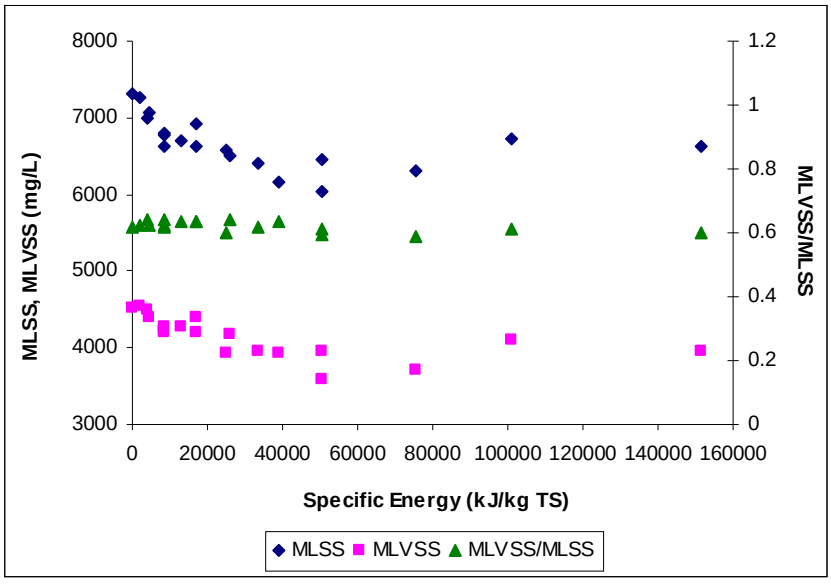


Figure 4.10. Variation of MLSS, MLVSS and MLVSS/MLSS with specific energy.

4.2.8. Effect of Ultrasonic Disintegration on Dewaterability

In order to determine the effect of ultrasonic pretreatment on sludge dewaterability, CST measurements were conducted. Variation of CST is given in Figure 4.11. The CST values increased with the increasing of SE applied to sludge. Therefore, the dewaterability of sludge has been seriously deteriorated after ultrasonic pretreatment. This more difficult dewaterability is the result of considerably increased micro particles (reduced floc size), offering an extended surface area (Dewil et al., 2006b). With the large surface area, more surface water is bound. Even at the low level of SE value (about 2000 kJ/kg TS), CST increased sharply from 20.5 s (the CST value of untreated sludge) to 282.4 s. It continued to increase strongly until the SE of about 50000 kJ/kg TS. For higher SE values, the rate of increase of CST decreased.

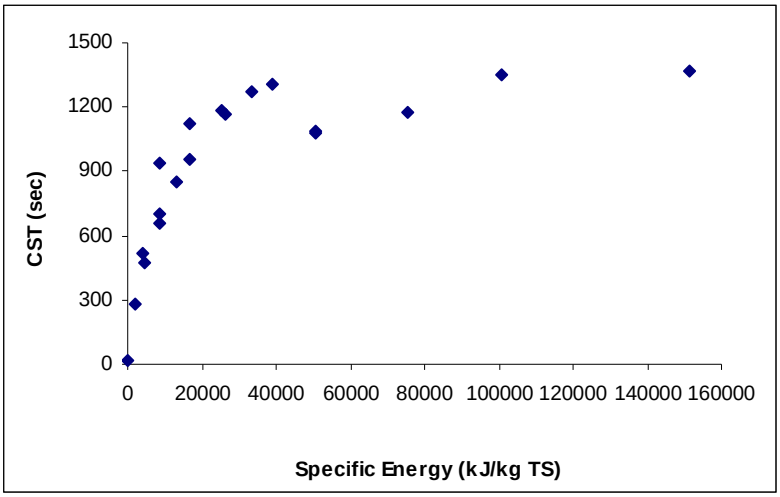


Figure 4.11. Variation of CST with specific energy.

4.2.9. Effect of Ultrasonic Disintegration on the Rheological Properties of Sludge

In order to determine the effect of ultrasonic disintegration on the rheological properties of sludge, viscosity measurements were conducted. The results of viscosity analyses are given in Figure 4.12 and Figure 4.13.

Results showed that viscosity decreased with increasing sonication time, power density and SE. The value of viscosity decreased strongly from 22.8 mPa s (which is the untreated sludge viscosity) to 20.8 mPa s for the SE between 0 and about 2000 kJ/kg TS. For SE values higher than 8500 kJ/kg TS, the rate of decrease of viscosity was lower. The solid concentration of sludge is the most important parameter that influences the viscosity (Hasar et al., 2004; Wu et al., 2007). The viscosity decreased with decreasing MLSS concentrations.

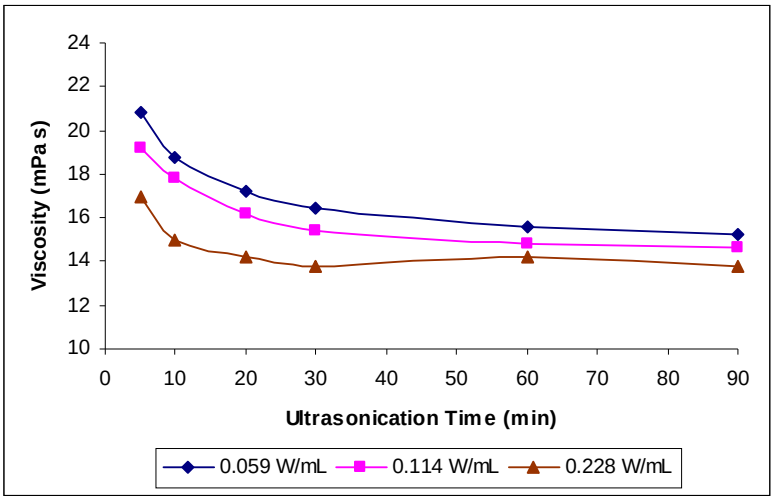
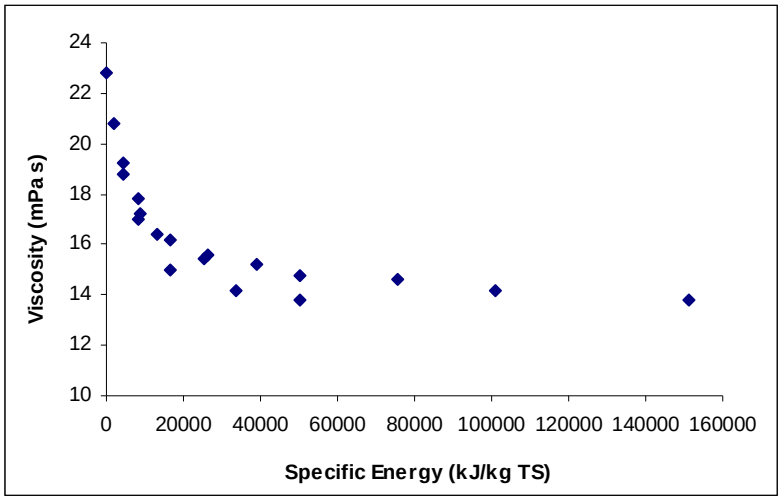


Figure 4.12. Variation of viscosity with ultrasonication time at various power densities.



(b)

Figure 4.13. Variation of viscosity with specific energy.

4.2.10. Effect of Ultrasonic Disintegration on Particle Size Distribution

The changes in the particles size distribution of untreated and ultrasonically disintegrated sludge samples are shown in Figure 4.14, Figure 4.15 and Figure 4.16. The results are summarized in Table 4.6. In this table, d_{10}, d_{50}, and d_{90} demonstrate 10%, 50%, and 90% of particles (in volume) having the diameter lower or equal to d_{10}, d_{50} and d_{90}, respectively.

Table 4.6. Particle size distribution of untreated and ultrasonically disintegrated sludges.

Power Density (W/mL)	**Sonication Duration (min)**	**SE (kJ/kg TS)**	**d_{10} (µm)**	**d_{50} (µm)**	**d_{90} (µm)**
0	0	0	20.015	58.665	148.786
0.059	5	2177	8.029	26.247	58.860
	10	4354	6.145	21.061	55.205
	20	8708	6.676	21.874	46.910
	30	13063	5.409	21.523	65.066
	60	26125	2.828	21.818	86.766
	90	39188	2.253	18.085	131.301
0.114	5	4199	8.811	24.878	52.642
	10	8399	7.135	23.768	54.228
	20	16797	3.325	23.145	80.912
	30	25196	3.146	23.219	156.051
	60	50391	2.405	20.572	123.152
	90	75587	3.585	30.682	135.454
0.228	5	8413	9.387	24.122	52.345
	10	16827	6.015	23.251	60.615
	20	33653	3.798	24.722	126.834
	30	50480	4.439	28.462	128.099
	60	100959	4.518	30.552	138.991
	90	151439	4.158	28.388	151.358

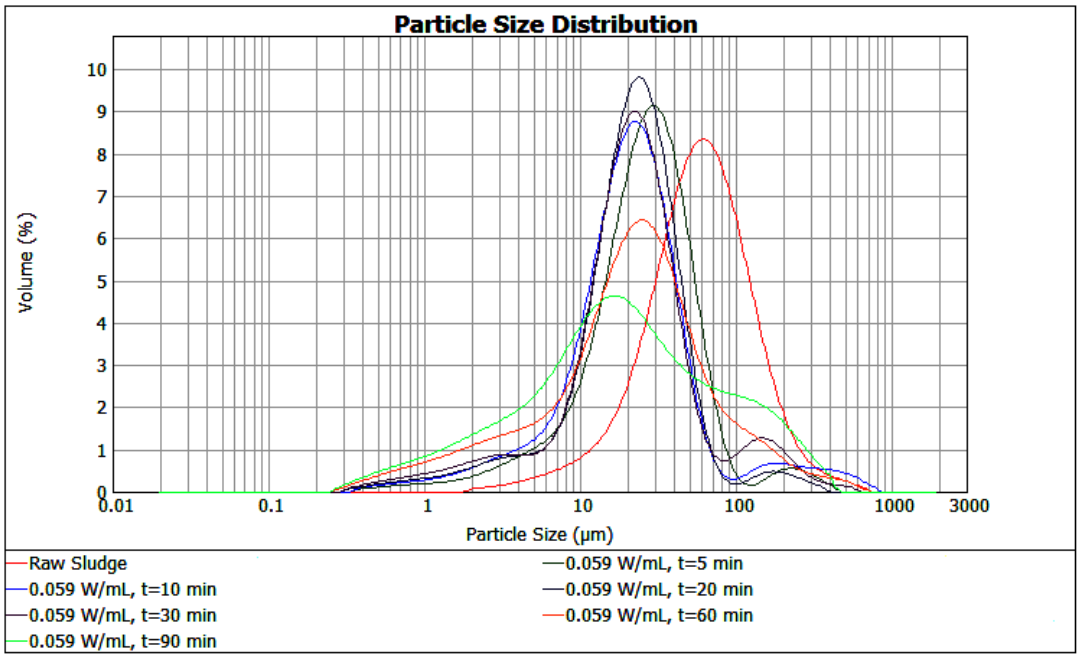


Figure 4.14. Variation of particle size distribution with sonication time at power density of 0.059 W/mL.

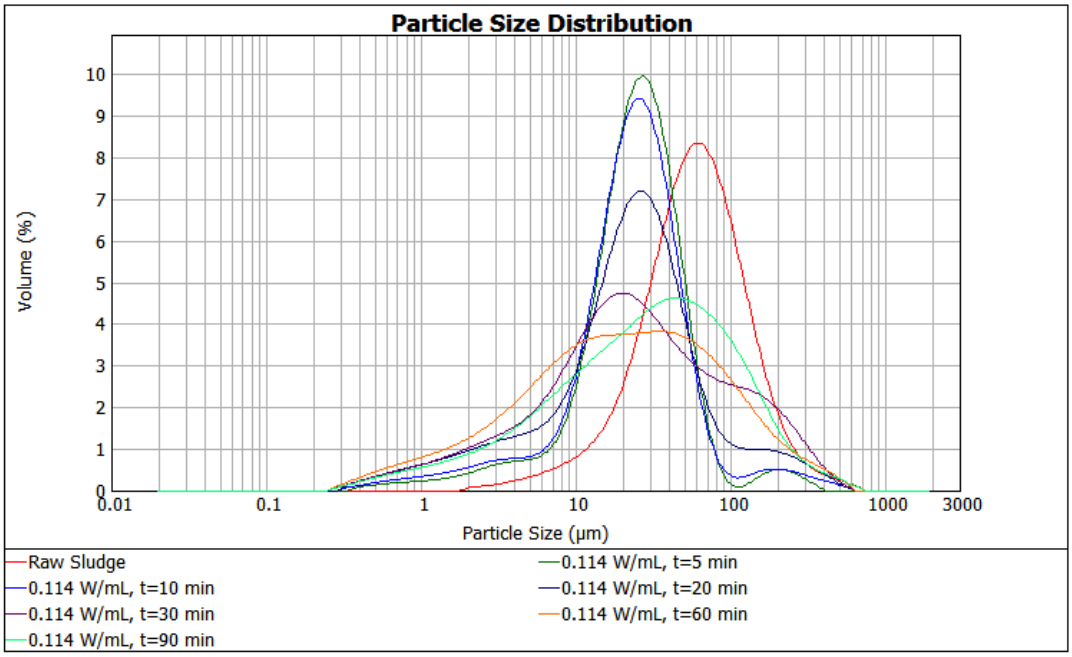


Figure 4.15. Variation of particle size distribution with sonication time at power density of 0.114 W/mL.

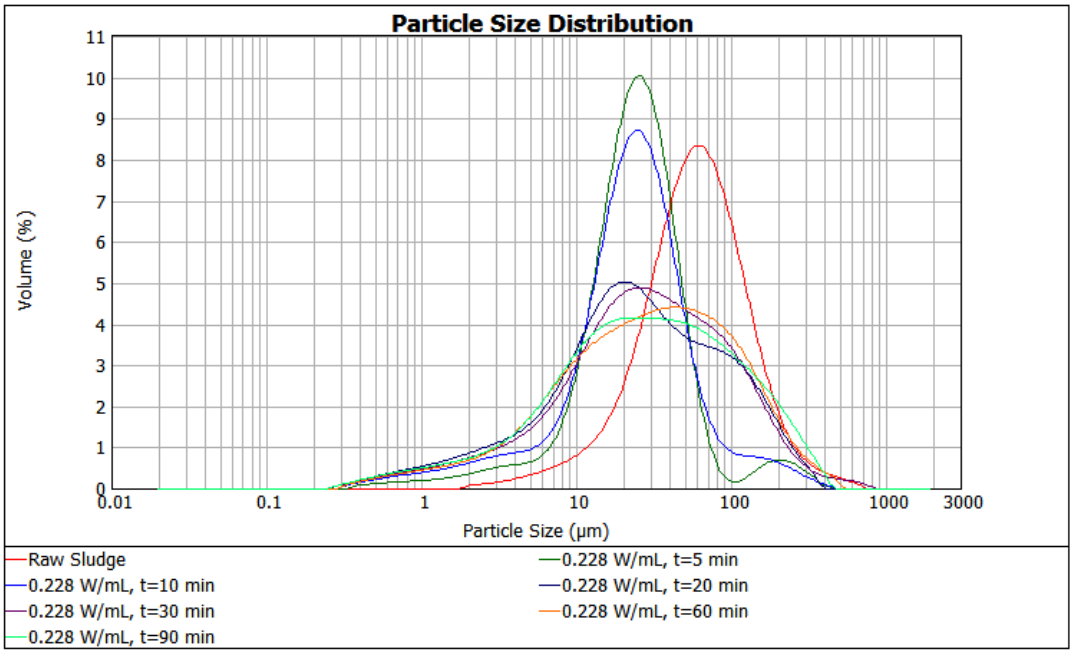


Figure 4.16 Variation of particle size distribution with sonication time at power density of 0.228 W/mL.

As can be seen form the figures, for untreated sludge, particle size distribution had a peak on the value of about 60 µm and the curve of particle size distribution was narrow. For ultrasonically disintegrated sludge samples, the peaks of particles size distribution became wider and shifted to the left indicating smaller particle sizes, and particle size distribution became more heterogeneous.

During the disintegration of the sludge samples at all power densities of 0.059 W/mL, 0.114 W/mL and 0.228 W/mL, the median diameter, d_{50}, decreased for the first 5 min period (corresponding to SE values of about 2000 kJ/kg TS, 4000 kJ/kg TS and 8400 kJ/kg TS, respectively). During this period there was around 55%-59% decrease in the median diameter, d_{50}. For the SE value higher than 50000 kJ/kg TS, the median diameters increased, indicating the reflocculation of the sludge particles which confirmed by the MLSS experiments also. This reflocculation may occur after ultrasonic pretreatment, because of the release and then regrouping of intracellular and/or extracellular materials (Bougrier et al., 2005; El-Hadj et al., 2007). Similar results were observed by Gonze et al. (2003), Bougrier et al. (2005) and El-Hadj et al. (2007).

Although it has not been really proved yet, many authors agreed that the applied energy is used to reduce flocs size at first, and then energy is used to break the flocs and release the extracellular material or to break the cells and release the intracellular materials out (Bougrier et al., 2005; Zhang et al., 2007).

For this study, the minimum SE of disintegration process required to break cells was found to be about 2000 kJ/kg TS. This result is in agreement with Lehne et al. (2001) found the same SE value of 2000 kJ/kg TS, and with Bougrier et al. (2005) determined the minimum SE of about 1000 kJ/kg TS.

4.3. Membrane Bioreactor-Ultrasonic Sludge Disintegration (MBR-US) Combined Systems for Sludge Minimization

4.3.1. Performance of Membrane

The MBR process provides a selective removal of products and solid-liquid separation because membranes are an absolute barrier. Therefore, higher and more consistent effluent quality can be obtained as a result of membrane filtration. In order to determine the effect of the filtration itself (membrane performance), reactor was operated only with wastewater (without activated sludge) taken from the effluent of aerated grit chamber of Paşaköy Advanced Biological Wastewater Treatment Plant. The permeate was sucked from the reactor by a peristaltic pump. Then, the wastewater (influent) and permeate (effluent) were analyzed for the measurements of COD, TS, VS, SS, VSS, TP, TKN, NH_3-N, NO_2^--N, NO_3^--N, turbidity and pH. The results are listed in Table 4.7.

By applying the filtration itself, 100% solids removal was obtained. This means the microfiltration system was very effective on organic removal performing. The filtration was also very effective on COD removal and reached to 90% total COD removal efficiency. The filtration removes the particulate COD, a large fraction of the total COD in municipal wastewaters (Ferraris et al., 2009).

With the application of the filtration itself, only about 10% of NH_3-N could be removed. For the membrane performance experiments, the removal efficiency of TP and TKN was 72% and 37%, respectively. Filtration achieved the turbidity removal efficiency of 100%.

Table 4.7. The results of membrane performance experiments.

Item	Influent	Effluent
COD (mg/L)	604.3	58.5
TS (mg/L)	1465	ND
VS (mg/L)	562	ND
SS (mg/L)	835	ND
VSS (mg/L)	372	ND
TP (mg/L)	9.6	2.7
TKN (mg/L)	75	47
NH_3-N (mg/L)	52.5	47.5
NO_2^--N (mg/L)	0.35	1.2
NO_3^--N (mg/L)	0.35	1.9
Turbidity (NTU)	616	0.63
pH	8.57	8.50

ND: non-detectable

4.3.2. Untreated Sludge and Feedwater Characteristics

Sludge samples and feedwater were collected from Paşaköy Advanced Biological Wastewater Treatment Plant and they were handled as explained in the Section 3.1.1 and 3.1.2, respectively. Both the untreated sludge and feedwater samples were analyzed for characterization whenever new samples were taken from plant. The characterization of the untreated sludge and municipal wastewater are summarized in Table 4.8 and Table 4.9, respectively.

Table 4.8. Characterization of the untreated sludge for combined systems experiments.

Parameter	**Range**	**Unit**
TS	8040-9680	mg/L
TVS	4850-5806	mg/L
MLSS	7410-9230	mg/L
MLVSS	4390-5800	mg/L
MLVSS/MLSS	0.59-0.65	-
SVI	104-130	mL/g
TCOD	6840-8900	mg/L
SCOD	32-52.8	mg/L
Viscosity	24.4-27.2	mPa s
CST	15.1-19.3	s
pH	6.98-7.38	-

Table 4.9. Characterization of the municipal wastewater used as feedwater.

Item	**Range**	**Unit**
COD	320-830	mg/L
SCOD	85-300	mg/L
TS	709-1465	mg/L
VS	214-562	mg/L
TP	2.7-12.8	mg/L
TKN	27-89	mg/L
NH_3-N	52.5-74	mg/L
NO_2^--N	0.35-5	mg/L
NO_3^--N	0.35-4.1	mg/L
pH	7.28-8.57	-

4.3.3. The First Case

In the first case, The MBR system was operated as CMBR (only wastewater feeding, no disintegrated sludge feeding) at first and then ultrasonic sludge disintegration was incorporated to MBR system in order to prevent the excess sludge production and the system was operated as MBR-US1 and MBR-US2 (wastewater and disintegrated sludge feeding). The MBR-US systems were operated at different sonication conditions such as power density, sludge sonication ratio, sludge sonication time and sonication frequency. Operational and sonication conditions are summarized in Table 3.7. During the operation of 10 days, all reactors were operated without sludge withdrawal except for the cases of sampling for analysis. The sampling volume was relatively small (10-15 mL) and ignored.

4.3.3.1. The Variations of MLSS Concentrations in Reactors. The effect of sludge disintegration on MLSS of all reactor systems is displayed in Figure 4.17. While the MLSS of CMBR increased from 7680 mg/L to 8090 mg/L, the MLSS of the combined systems (MBR-US1 and MBR-US2) decreased. 29% and 11% of sludge reduction was observed for the systems of MBR-US1 and MBR-US2, respectively. Reactor sludge decreased from 7410 mg/L to 5260 mg/L and from 7600 mg/L to 6750 mg/L for the MBR-US1 and MBR-US2, respectively.

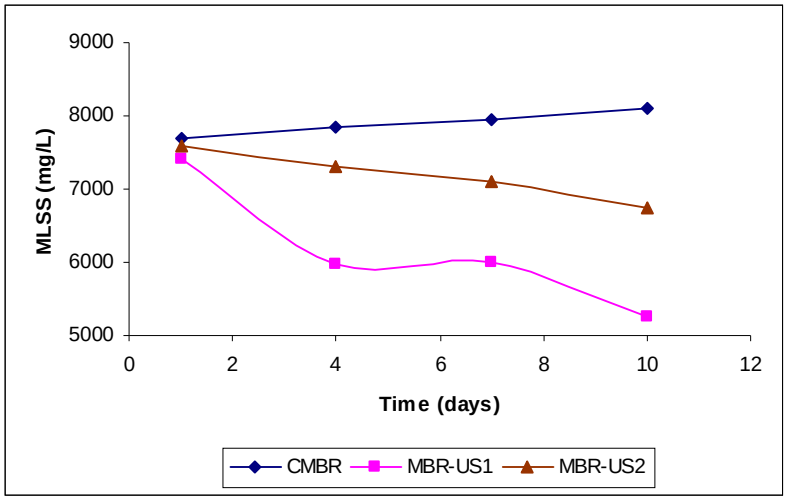


Figure 4.17. Variations of MLSS concentration in the first case.

The results given in Figure 4.17 showed that sonication time and sonicated sludge volume is more effective than power density in reactor sludge reduction. The MLSS reduction in the MBR-US1 (having disintegrated sludge volume of 1000 mL and the power density of 0.114 W/mL) was higher than that in the MBR-US2 (having disintegrated sludge volume of 500 mL and the power density of 0.228 W/mL). While sonication of smaller volumes of sludges led to higher DD and SE values, the sonicaiton of larger volumes caused to higher MLSS reduction in reactors. This effect can be explained with the disintegration of higher amounts of sludge that will lead to lower amount of excess sludge production.

In the MBR-US systems, none of insoluble inorganic particles can pass the physical barrier of the membrane. Therefore, accumulation of insoluble inorganic materials might be a major obstacle for zero sludge production (Yoon et al., 2004). MLVSS/MLSS were measured to investigate this effect (Figure 4.18). Inorganic accumulation was observed neither in CMBR system nor in MBR-US1 and MBR-US2 systems although they were fed with real municipal wastewater (not synthetic wastewater). However, in long term studies, some inorganic accumulation is inevitable for an MBR-US system treating real wastewater containing insoluble inorganic particles (Yoon et al., 2004). Hence, further studies with the MBR-US system should be studied in long term to investigate the effects of influent inorganics on inorganic accumulation.

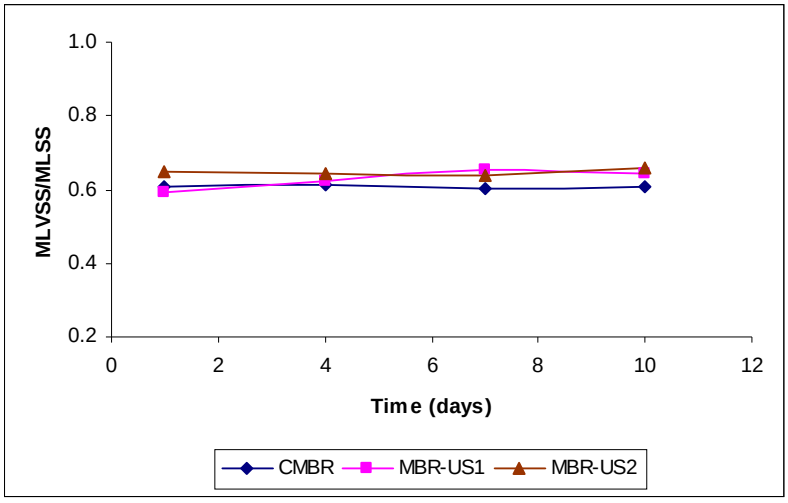


Figure 4.18. Variations of MLVSS/MLSS in the first case.

4.3.3.2. The Variations of SVI in Reactors. The effect of sludge disintegration on SVI is shown in Figure 4.19. A value of 100 mL/g is considered a good settling sludge and so SVI values below 100 mL/g are desired (Metcalf & Eddy, 2004). The SVI value of the reactor sludges was in the range between 120-130 mL/g considered as a sludge with poor settling characteristics when the systems starting up. Although SVI were constant at the value 120 mL/g in CMBR, it increased in the MBR-US1 and MBR-US2. Although it gradually increased in the MBR-US2, it sharply increased from 130 mL/g to 157 mL/g and then slightly decreased to 144 mL/g in the MBR-US1. The rate of increase of SVI was faster in the MBR-US1 than in the MBR-US2 because sonicated sludge volume and sonication time were higher in the MBR-US1 (1000 mL and 15 min., respectively) than in the MBR-US2 (500 mL and 5 min., respectively). In the ultrasonic sludge disintegration experiments, it was determined that settling deteriorated as sonication time increased (Section 4.2.5). That is the reason of increase in SVI.

It is known that ultrasound pretreatment produces a lot of separate cells and short pieces of filaments (Dewil et al., 2006a) and SVI values above 150 mL/g are typically associated with filamentous growth (Metcalf & Eddy, 2004). In the MBR-US1 system, SVI increased up to 157 mL/g. Therefore, it can be deduced that there was a filamentous growth in the MBR-US1 system.

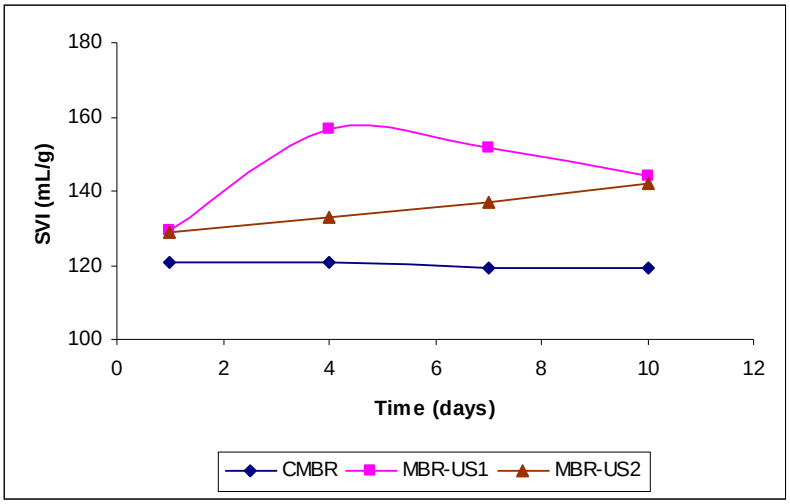


Figure 4.19. Variations of SVI in the first case.

After an increase, a decrease was observed in the SVI of MBR-US1 system. The rate of decrease in settled sludge volume was higher than that of the rate of decrease in MLSS concentration. This caused a drop in SVI value.

During the course of the MBR-US1 experiments, some amount of foam was observed on the surface of reactor (Figure 4.20). It is considered that the foaming problem was caused by shock organic loading and protoplasmic polymers from disintegrated cells (Yoon et al., 2004).



Figure 4.20. Foaming problem in MBR-US1.

4.3.3.3. The Variations of Sludges Rheology in Reactors. In order to determine the effect of ultrasonically disintegrated sludge on sludge rheology in the reactors, viscosity of reactor sludges was measured. The results are given in Figure 4.21. Results showed that the viscosity of MBR-US2 system strongly decreased while it was slightly increased in the CMBR. The solid concentration of sludge is the most important parameters that influence the viscosity (Hasar et al., 2004; Wu et al., 2007). The viscosity of CMBR system increased from 19.8 mPa s to 20.6 mPa s, the reason of this increase is the increasing of MLSS concentration in the system (Section 4.3.3.1). The viscosity of MBR-US2 system decreased from 24.4 mPa s to 16.8 mPa s, because the MLSS concentration of system decreased as a result of sludge disintegration (Section 4.3.3.1). As mentioned in Section 3.2.1.2, a part of the reactor sludge (500 mL) was disintegrated and returned into the MBR-US2 system as a feed solution once a day in a batch manner.

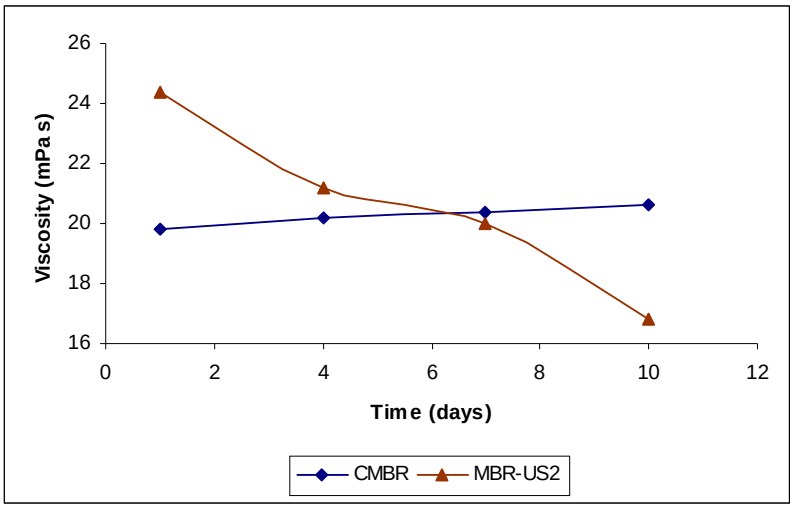


Figure 4.21. Variations of viscosity in the first case.

4.3.3.4. The Variations of SCOD Concentrations of Sludges in Reactors. In the Section 4.2.4, it was determined that sonication enhanced the soluble organic concentration, which is indicated as SCOD. Therefore, in order to observe the changes in soluble organic concentrations of reactors, SCOD of sludges was monitored. As can be seen from the Figure 4.22, the SCOD of MBR-US1 and MBR-US2 system increased gradually while the SCOD concentration of CMBR was almost constant at the range of 50-60 mg/L. The rate of increase of SCOD in the MBR-US1 system was slightly higher than in the MBR-US2 system because the sonicated sludge volume was higher in the MBR-US1 system (1000 mL) than in the MBR-US2 system (500 mL). The SCOD concentration of MBR-US1 increased from 52.8 mg/L to 122.4 mg/L while SCOD increased from 50.4 mg/L to 93.6 mg/L in the MBR-US2 system. By applying sonication, cells underwent lysis and organic compounds were released into the liquid phase (Bougrier et al., 2005) and this caused an increase in SCOD concentrations of MBR-US1 and MBR-US2 systems. In the CMBR, the SCOD concentration was stable, because there was not an extra organic loading. In the MBR-US1 and MBR-US2 systems there was an extra organic loading coming from disintegrated sludge feeding.

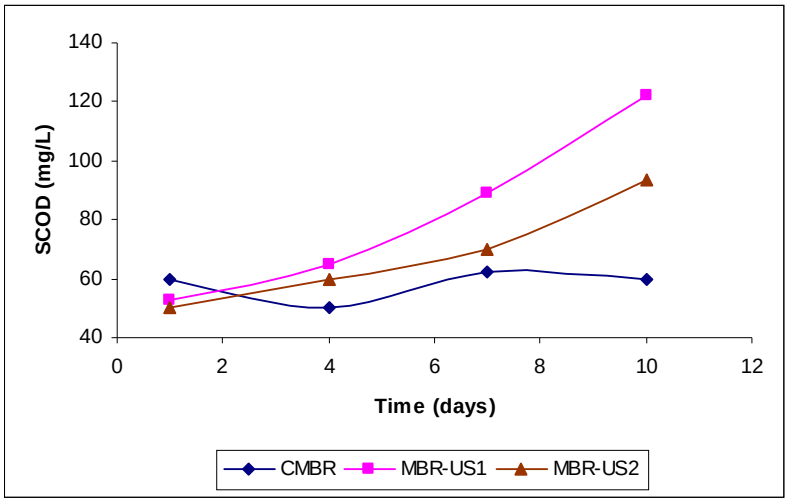


Figure 4.22. Variations of SCOD concentration of sludges in the first case.

4.3.3.5. The Variations of Dewaterability of Sludges in Reactors. CST has been widely accepted and used for the evaluation of dewaterability of activated sludge (Wu et al., 2007). Therefore, in order to determine the effect of ultrasonically disintegrated sludge on the dewaterability of sludges in reactors, CST measurements were conducted. Variations of CST are given in Figure 4.23. In CMBR, the increase of CST was very low (from 21.3 s to 22.4 s) caused by MLSS increasing. The CST of MBR-US1 increased from 18.9 s to 50.9 s while CST increased from 19.3 s to 30.2 s in the MBR-US2 system. The rate of increase of CST in the MBR-US1 system was higher than in the MBR-US2 system because the sonicated sludge volume was higher in the MBR-US1 system (1000 mL) than that in the MBR-US2 system (500 mL). Disintegrated sludge was fed into the MBR-US1 and MBR-US2 systems and this caused an increase of small particles, which is offering an extended surface area, in these systems. With the large surface area, more surface water was bound and so CST values of MBR-US1 and MBR-US2 systems increased. The rate of increase of CST values of MBR-US1 and MBR-US2 systems was faster than that of CMBR. The dewaterability of MBR-US1 and MBR-US2 systems has been seriously deteriorated after feeding ultrasonically disintegrated sludges into these systems. And this high value of CST usually implies a poor filterability and dewaterability (Wu et al., 2007).

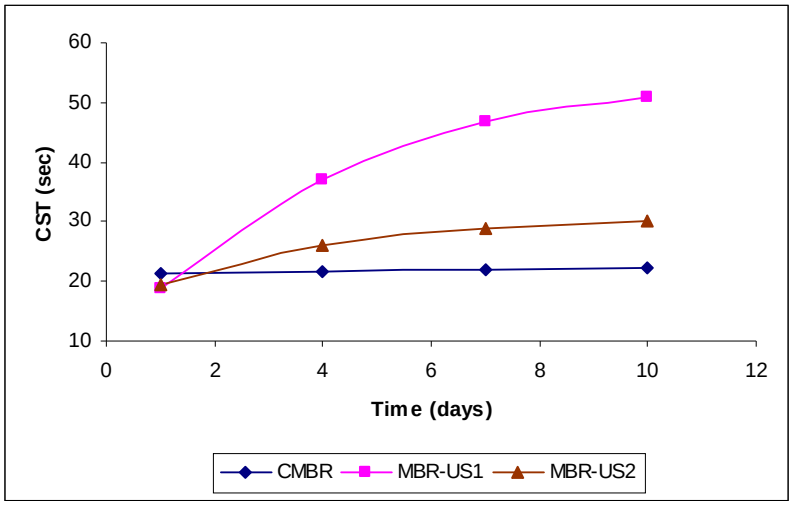


Figure 4.23. Variations of CST in the first case.

4.3.3.6. The Variations of Particle Size Distribution of Sludges in Reactors. The results of particle size distribution are given in Figure 4.24 and Table 4.10. In the ultrasonic sludge disintegration experiments, it was determined that sonication caused the reduction of floc size (Section 4.2.10). Figure 4.24 and Table 4.10 are also in agreement with the previous results. The particle size of MBR-US1 and MBR-US2 systems reduced after 10-day operating time while the particle size of CMBR was maintained almost constant. Ultrasonically disintegrated sludge was fed into the MBR-US1 and MBR-US2 systems and this caused the reduction in the median diameter, d_{50}, and therefore small particles increased. Small particles were not favorable for the filtration and settling of sludges (Chu et al., 2001). The deterioration of sludge dewaterability and, increasing of SCOD and SVI values of MBR-US1 and MBR-US2 systems correlates well with the decrease in sludge floc size.

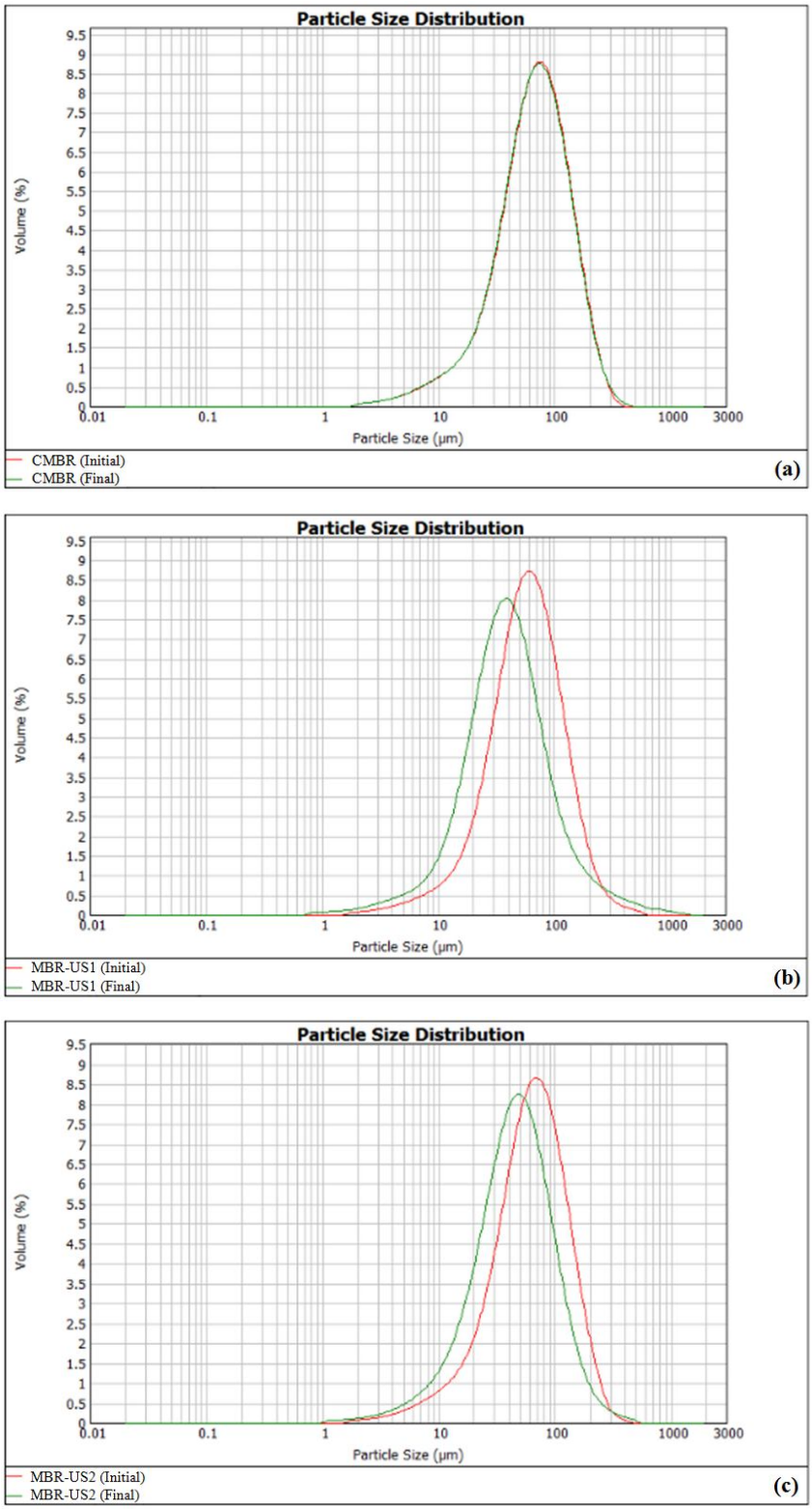


Figure 4.24. Variations of particle size distribution in (a) CMBR (b) MBR-US1 and (c) MBR-US2.

Table 4.10. Particle size of sludge samples of CMBR, MBR-US1 and MBR-US2 at the beginning and at the end of operation.

Reactor	**Experiment Time**	**d_{10} (μm)**	**d_{50} (μm)**	**d_{90} (μm)**
CMBR	1st day	22.659	68.157	154.499
	10th day	22.537	67.622	154.416
MBR-US1	1st day (before US application)	20.848	58.178	138.692
	10th day (after US application)	13.391	38.911	116.346
MBR-US2	1st day (before US application)	20.938	63.194	146.252
	10th day (after US application)	14.761	45.362	113.415

The mean diameter, d_{50}, of MBR-US1 sludges decreased from 58 μm to 39 μm and the mean diameter of MBR-US2 sludges decreased from 63 μm to 45 μm. The particles size of CMBR sludges was stable at the value of 68 μm.

4.3.3.7. The Effect of Ultrasonic Sludge Disintegration on the Effluent Quality. In order to determine the effect of ultrasonic pretreatment application on the effluent quality of the reactors, COD of feedwater (influent) and permeate (effluent) were analyzed. The variations of COD removal efficiency are shown in Figure 4.25. During the operation time, the COD removal efficiency of CMBR was stable at 95%. However, the COD removal efficiency of the MBR-US1 and MBR-US2 systems decreased from 94% to 86% and from 95% to 90%, respectively. Although the permeate COD of the CMBR did not exceed 29 mg/L during the experiment, it increased from 38 mg/L to 62 mg/L in the MBR-US1 system. The permeate COD of MBR-US2 system increased from 26 mg/L to 36 mg/L. Therefore, the effluent quality of the MBR-US systems was deteriorated. The additional organic loading caused by the recycle of ultrasonically disintegrated sludge was considered to be a reason for this. The MBR-US2 system was less deteriorated than MBR-US1 system because ultrasonically disintegrated sludge volume of MBR-US2 system (500 mL) was less than that of MBR-US1 system (1000 mL).

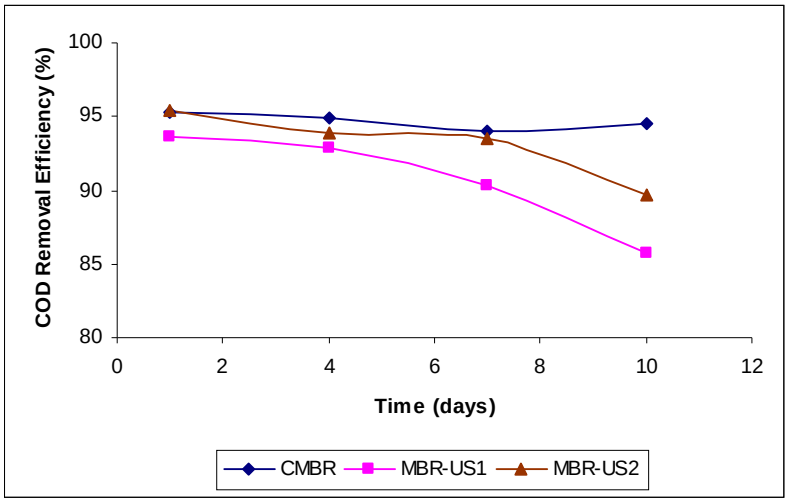


Figure 4.25. Variations of COD removal efficiency in the first case.

In order to determine the effect of recycled disintegrated sludge on the effluent quality of the reactors, the permeate turbidity was also analyzed (Figure 4.26). Results showed that the permeate turbidity of CMBR was at the level of 0.25 NTU and it did not change during the operation. The turbidity of permeate increased gradually both in the MBR-US1 system and in the MBR-US2 system. The results of turbidity experiments are in agreement with effluent COD experiments.

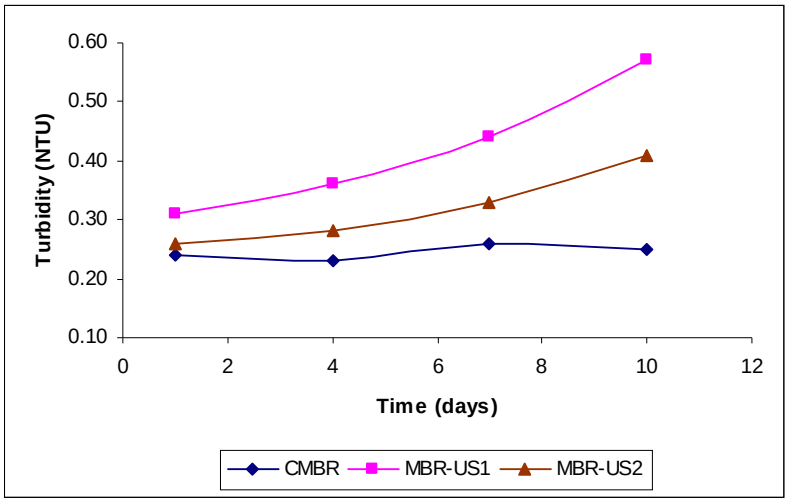


Figure 4.26. Variations of permeate turbidity in the first case.

For CMBR, TKN removal efficiency was 87%. At the beginning of the operation, TP and TKN removal efficiency of MBR-US2 system was 70% and 94%, respectively. After 10 days of operation, TKN removal efficiency was decreased to 91% while TP could not be removed from the system. The influent TP was 12.8 mg/L and effluent TP was 28 mg/L. After ultrasonically disintegration of sludge, nitrogen and phosphorus release to supernatant and the concentration of nitrogen and phosphorus increased significantly in the supernatant (Gündüz, 2009). The additional nitrogen and phosphorus loading caused by the recycle of disintegrated sludge was considered to be a reason of the reduction in TP and TKN removal efficiency.

4.3.4. The Second Case

In the second case, a 15-L sequential batch reactor, a sequential batch reactor-ultrasound (SBR-US) combined system and a membrane bioreactor-ultrasound (MBR-US3) combined system were operated in parallel. The sequential batch reactor was the control reactor, CR, and fed only with wastewater. The SBR-US and MBR-US3 combined systems were operated in the same manner: these systems fed with wastewater and ultrasonically disintegrated excess sludge. For these reactors, instead of daily sonication, the sludge was sonicated as needed basis. In this case, to compare the efficiency of MBR with SBR in terms of sludge minimization and effluent quality, the SBR-US combined system was additionally operated. CR and SBR-US did not include membrane panel. The sonication conditions of sludges fed into SBR-US and MBR-US3 was same. Operational and sonication conditions were summarized in Table 3.7. During the operation of 21 days, all reactors were operated without sludge withdrawal except for the cases of sampling for analysis. The sampling volume was relatively small (10-15 mL) and ignored. The sonication conditions of sludges fed into SBR-US and MBR-US3 was same. For these reactors, instead of daily sonication, the sludge was sonicated as needed basis (1st, 4th, 15th day).

4.3.4.1. The Variations of MLSS Concentrations in Reactors. The results of MLSS experiments during 21 days of operation are given in Figure 4.27. The MLSS increased from 8440 mg/L to 10680 mg/L in the CR. In the first day of operation after all analyses

were carried out, the parts of sludge (500 mL) from SBR-US and MBR-US3 were sonicated and returned into the systems (SBR-US and MBR-US3) as a feed. This process was repeated in the 4th day of operation in order to keep MLSS concentrations in the SBR-US and MBR-US3 systems around 9200 mg/L. In the 8th day of operation, it was observed that the MLSS concentration of SBR-US and MBR-US3 was lower than 9200 mg/L, therefore sonication was not applied to the systems until it reach the value of 9200 mg/L. In the 15th day of operation, MLSS increased to the initial value in the SBR-US and MBR-US3, hence sonication applied to keep MLSS concentration constant in these systems. In the last day of operation, the MLSS was maintained at 9430 mg/L and 9260 mg/L in the SBR-US and MBR-US3, respectively. Zero excess sludge production was achieved with SBR-US and MBR-US3 systems with the strategy of as needed basis excess sludge disintegration.

Some authors (Yoon et al., 2004; Ming-he and Chao-hai, 2010), using synthetic wastewater as a feed, were achieved zero excess sludge production with daily sludge disintegration. However, in this study, as a result of using real municipal wastewater, sludge yield was not constant and therefore excess sludge production varied during operation time.

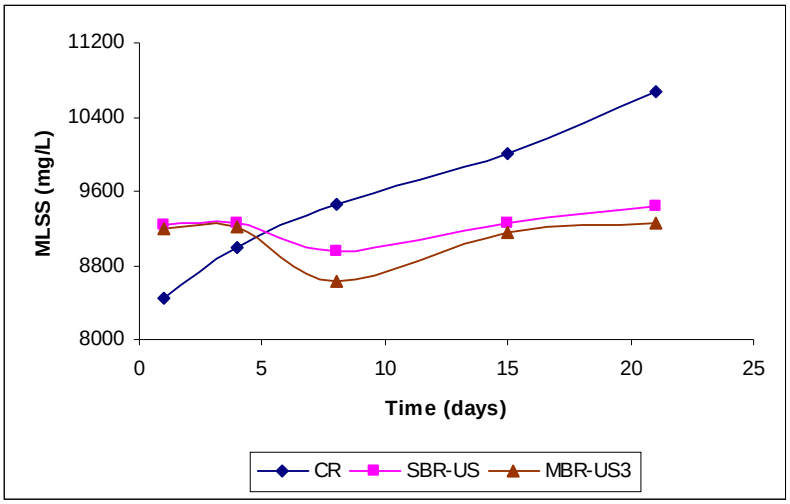


Figure 4.27. Variations of MLSS concentration in the second case.

MLVSS/MLSS were measured in order to investigate the accumulation of insoluble inorganic materials (Figure 4.28). The MLVSS/MLSS varied between 0.60-0.64 and was almost constant in all systems. Inorganic accumulation was observed neither in CR system nor in SBR-US and MBR-US3 systems although they were fed with real municipal wastewater (not synthetic wastewater). However, in long term studies, some inorganic accumulation is inevitable for an MBR-US system treating real wastewater containing insoluble inorganic particles such as silts (Yoon et al., 2004). Hence, further studies with the MBR-US system should be studied in long term to investigate the effects of influent inorganics on inorganic accumulation.

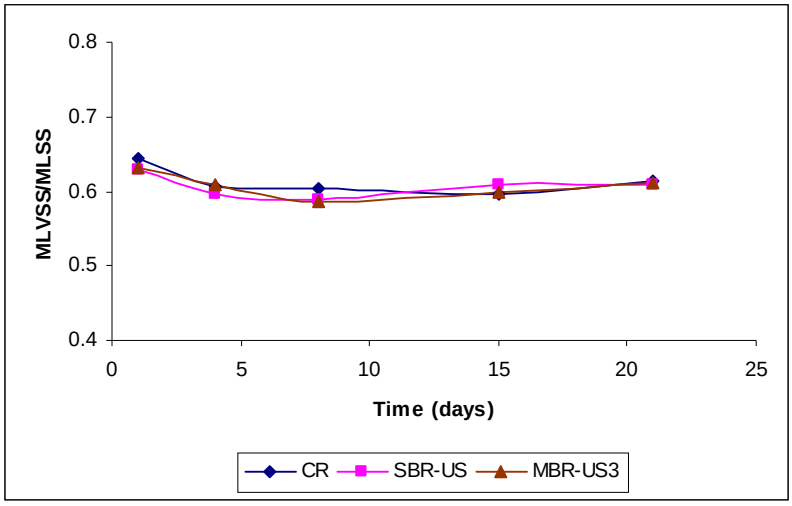


Figure 4.28. Variations of MLVSS/MLSS in the second case.

4.3.4.2. The Variations of SVI in Reactors. The variations of SVI in the second case are given in Figure 4.29. In the CR, the SVI improved; it decreased from 113 mL/g to 92 mL/g which is considered as a good settling sludge (Metcalf & Eddy, 2004). Although some parts of disintegrated sludge returned into the SBR-US and MBR-US3 systems, SVI deterioration was not observed in these systems. Lower excess sludge sonication frequency was considered to be a reason for this. It seems that the MBR-US systems can tolerate the effects of returned disintegrated excess sludge when the sonication time interval increases. The SVI of the SBR-US and MBR-US3 systems was in the ranges between 100-105 mL/g.

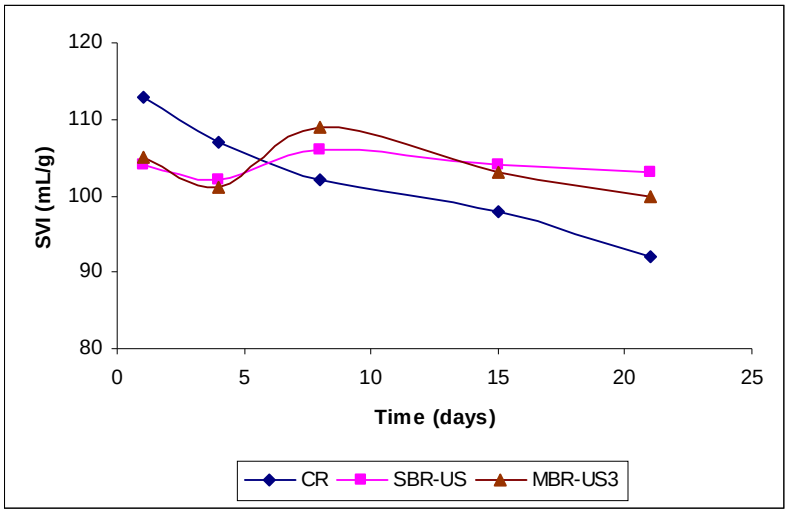


Figure 4.29. Variations of SVI in the second case.

4.3.4.3. The Variations of Sludges Rheology in Reactors. In order to determine the effect of ultrasonically disintegration sludge on the sludge rheology in reactors, viscosity of reactor sludges was measured. The results are given in Figure 4.30. Due to an increase in MLSS concentration of CR, the viscosity of system increased from 24.8 mPa s to 31.6 mPa s. The viscosity of SBR-US and MBR-US3 systems was slightly decreased and then it was maintained at 20-22 mPa s in these systems.

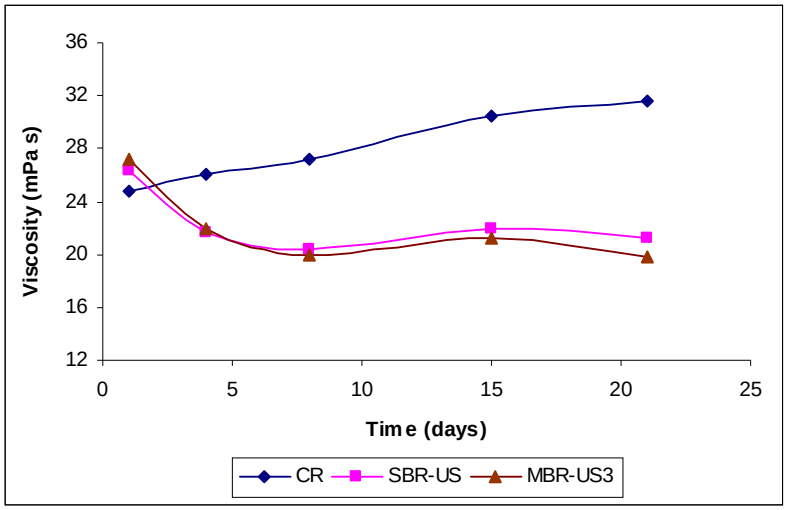


Figure 4.30. Variations of viscosity in the second case.

4.3.4.4. The Variations of SCOD Concentrations of Sludges in Reactors. Sonication improves the soluble organic concentration indicated as SCOD. Therefore, in order to observe the changes in soluble organic concentrations of reactors, the SCOD values of sludges in the reactors were monitored (Figure 4.31). The SCOD concentration of CR was almost constant at the range between 35-45 mg/L. In the CR, the SCOD concentration was stable, because there was not an extra organic loading. In the SBR-US and MBR-US3 systems there was an additional organic loading caused by disintegrated sludge feeding. Therefore, SCOD concentration in the SBR-US and MBR-US3 systems increased during the experimental period of 21 days. However, the rate of increase of SCOD in the MBR-US3 system (and also in the SBR-US) was lower than in the MBR-US1 and MBR-US2 systems. Excess sludge sonication frequency was lower in the MBR-US3. An increased sonication time interval was considered to be a reason for this.

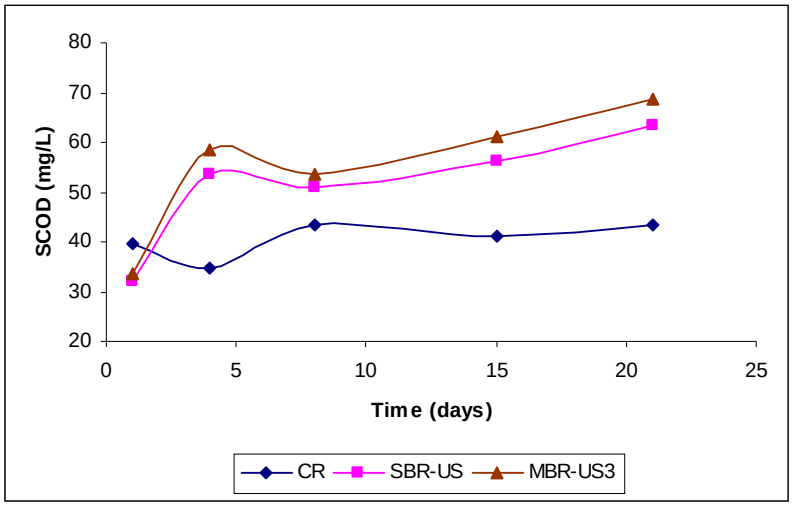


Figure 4.31. Variations of SCOD concentration of sludges in the second case.

4.3.4.5. The Variations of Dewaterability of Sludges in Reactors. In order to determine the effect of ultrasonically disintegrated sludge on sludge dewaterability in the reactors, CST measurements were conducted. The variations of CST are given in Figure 4.32. The CST of CR increased from 18.9 s to 27.7 s caused by increase of MLSS. The CST of SBR-US increased from 15.1 s to 33.2 s and the CST increased from 17.3 s to 34.9 s in the MBR-US3 system. Disintegrated sludge was fed into the SBR-US and MBR-US3 systems and this caused an increase of small particles, which is offering an extended surface area,

in these systems. With the large surface area, more surface water was bound and so CST values of the SBR-US and MBR-US3 systems increased. The dewaterability of the SBR-US and MBR-US3 systems has been deteriorated after feeding ultrasonically disintegrated sludges into these systems. And this high value of CST usually implies a poor filterability and dewaterability (Wu et al., 2007).

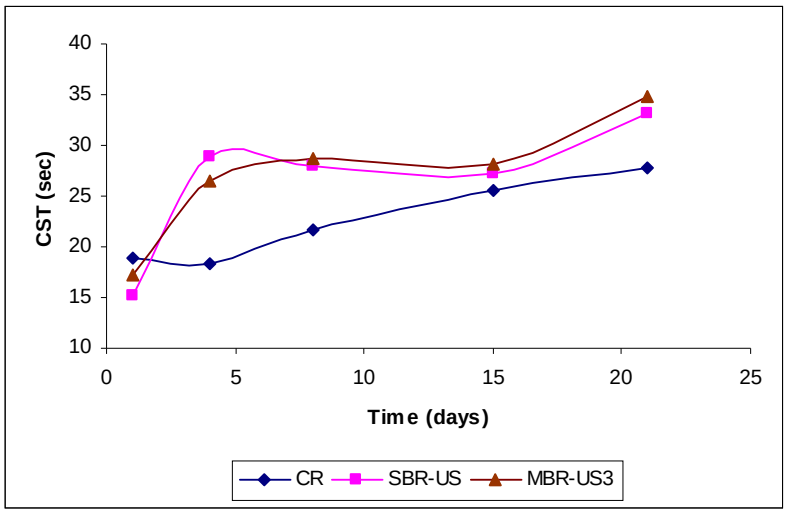


Figure 4.32. Variations of CST in the second case.

4.3.4.6. The Variations of Particle Size Distribution of Sludges in Reactors. The results of particle size distribution are given in Figure 4.33 and Table 4.11. Sonication causes the reduction of floc size (Section 4.2.10). Therefore, the particle size of SBR-US and MBR-US3 systems reduced after 21-day operation time while the particle size of CR was maintained almost constant. Ultrasonically disintegrated sludge was fed into the SBR-US and MBR-US3 systems and this caused the reduction in the median diameter, d_{50}, and therefore small particles increased. Small particles were not favorable for the filtration and settling (Chu et al., 2001). The deterioration of sludge dewaterability and increasing of SCOD values of SBR-US and MBR-US3 systems correlates well with the decrease in sludge floc size.

The mean diameter, d_{50}, of SBR-US sludges decreased from 68 μm to 58 μm and the mean diameter of MBR-US3 sludges decreased from 68 μm to 59 μm. The particles size of CR sludges was stable around 66-64 μm.

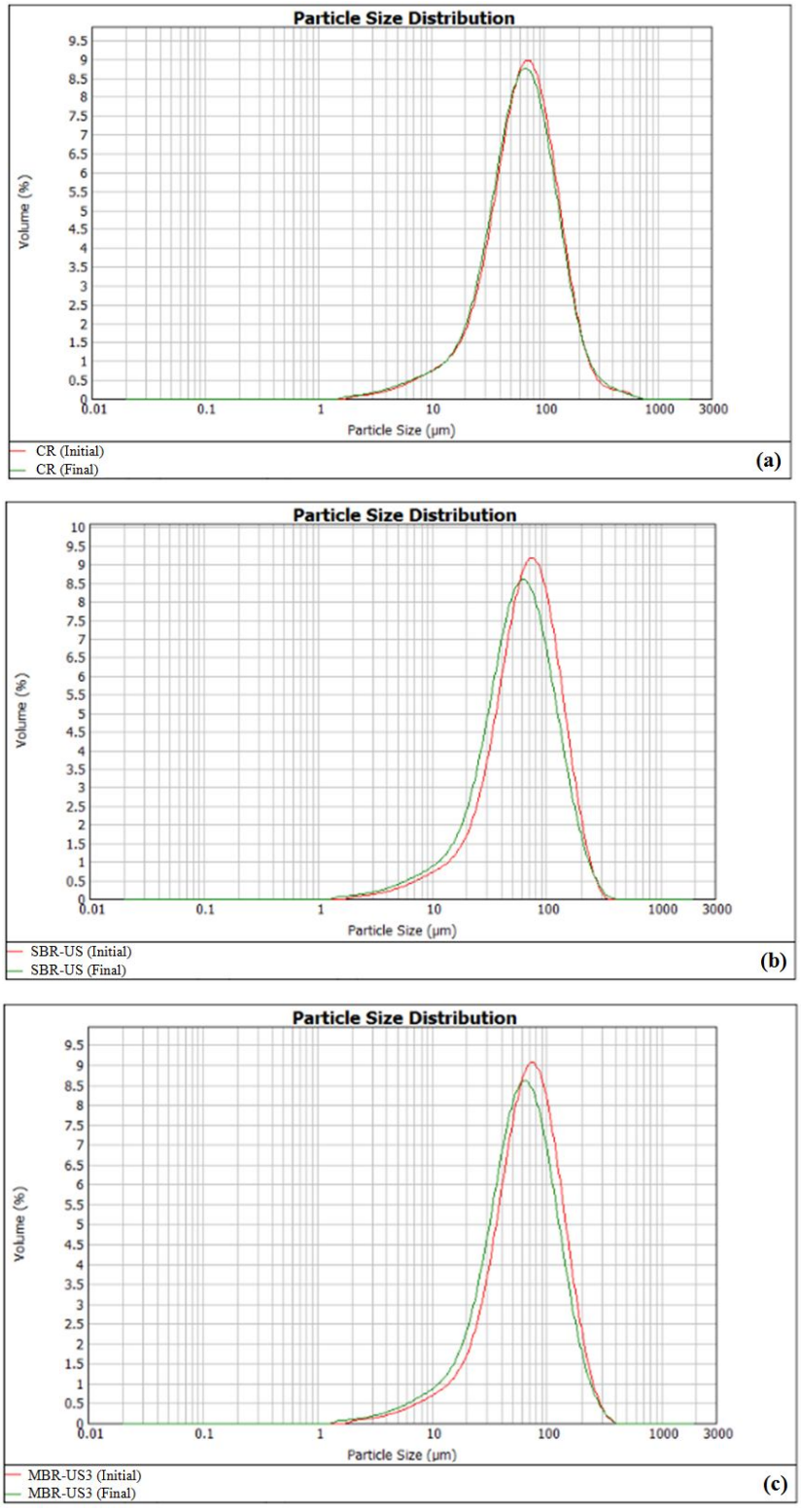


Figure 4.33. Variations of particle size distribution in (a) CR (b) SBR-US and (c) MBR-US3.

Table 4.11. Particle size of sludge samples of CR, SBR-US and MBR-US3 at the beginning and at the end of operation.

Reactor	**Experiment Time**	**d_{10} (µm)**	**d_{50} (µm)**	**d_{90} (µm)**
CR	1st day	22.773	66.029	151.022
	21st day	21.889	63.828	150.246
SBR-US	1st day (before US application)	23.435	67.759	147.558
	21st day (after US application)	19.152	58.108	136.893
MBR-US3	1st day (before US application)	23.596	68.283	151.860
	21st day (after US application)	19.442	59.444	139.792

4.3.4.7. The Effect of Ultrasonic Sludge Disintegration on the Effluent Quality. In order to determine the effect of ultrasonic pretreatment application on the effluent quality of the reactors, the influent COD and the effluent COD were analyzed. The variations of effluent COD and COD removal efficiency are shown in Figure 4.34. The COD removal efficiency of CR and MBR-US3 was at the level of 95-96% while it was 94-95% in the SBR-US. Although additional organic loading caused by the return of ultrasonically disintegrated sludge into the SBR-US and MBR-US3 systems, the effluent quality of these systems was not deteriorated. The effluent quality of the MBR-US1 and MBR-US2 systems was deteriorated due to additional organic loading caused by disintegrated sludge feeding. The sonication time intervals was considered to be a reason for this (the sonication frequency of the MBR-US1 and MBR-US2 systems was daily while it was as needed basis in the MBR-US3 system).

During the operation time, the effluent COD of the CR and MBR-US3 systems did not exceed 33 mg/L while it increased up to 39 mg/L in the SBR-US. It seems that the MBR systems are more efficient in tolerating the effects of returned disintegrated excess sludge than the SBR systems.

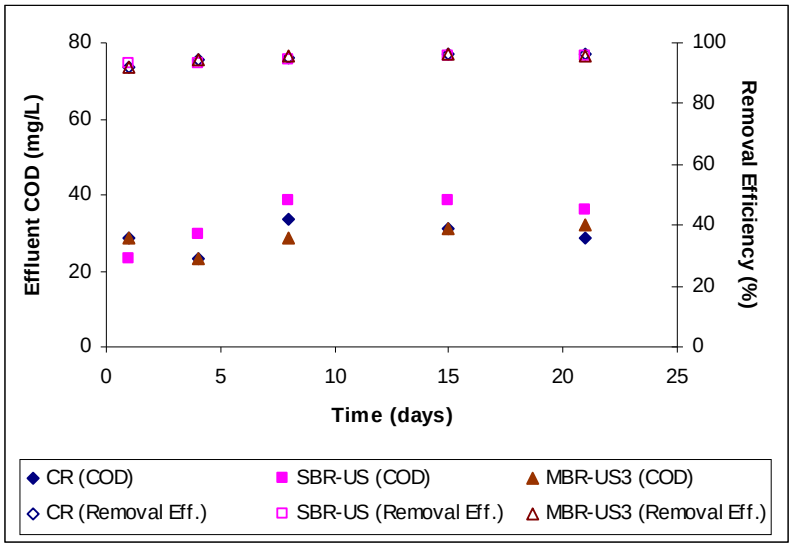


Figure 4.34. Variations of effluent COD and COD removal efficiency in the second case.

In order to determine the effect of recycled disintegrated sludge on the effluent quality of the reactors, the effluent turbidity was also analyzed (Figure 4.35). The results showed that the effluent turbidity of CR was at the level of 1.35 NTU and it did not change during the operation. The effluent turbidity of MBR-US3 system also did not change and it was at the level of 0.35 NTU. Although MBR-US3 system was fed with an additional organic loading coming from returned disintegrated sludge, its effluent turbidity was better than CR. The effluent turbidity of SBR-US sharply increased from 1.08 NTU to 2.74 NTU with the additional loading caused by disintegrated sludge. With the decreasing of sonication frequency (after the 4th day, sonication did not apply until 15th day) it was gradually decreased to 1.96 NTU. The effluent turbidity of MBR-US3 was lower than that of SBR-US during the operation and also it was almost constant in the MBR-US3. This proved that the MBR systems are more efficient in tolerating the effects of returned disintegrated excess sludge than the SBR systems. The results of turbidity experiments are in agreement with effluent COD experiments.

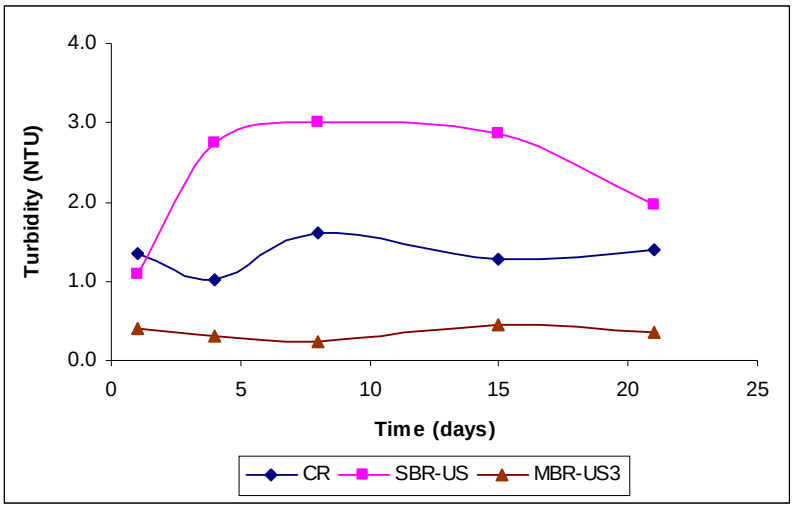


Figure 4.35. Variations of effluent turbidity in the second case.

In order to investigate the influence of sonicated sludge on the nutrient removal efficiency of systems, TP and TKN measurements were carried out. The results of TKN measurements are shown in Figure 4.36. The average TKN removal efficiency for CR, SBR-US and MBR-US3 was determined as 91%, 93% and 93%, respectively. The TKN removal efficiency in combined systems (SBR-US and MBR-US3) was higher. Such differences in the TKN removal might be due to the addition of soluble organic matter (sonicated sludge) into SBR-US and MBR-US3 as a carbon source for the TKN removal. Similar results were obtained by some other authors (Song et al., 2003; Ming-he and Chao-hai, 2010).

The results of TP measurements are shown in Figure 4.37. At the beginning of the operation, the TP removal efficiency of CR, SBR-US and MBR-US3 was 62%, 60% and 41%, respectively. At the end of operation (21st day), the TP removal efficiency for CR, SBR-US and MBR-US3 was determined as 60%, 53% and 78%, respectively. The increase of TP removal efficiency in the MBR-US3 might be due to the addition of soluble organic matter (sonicated sludge) into the system as a carbon source for the TP removal. Similar results were obtained by Song et al. (2003).

The results of TKN and TP measurements showed that the MBR-US3 was more efficient in nutrient removal than CR.

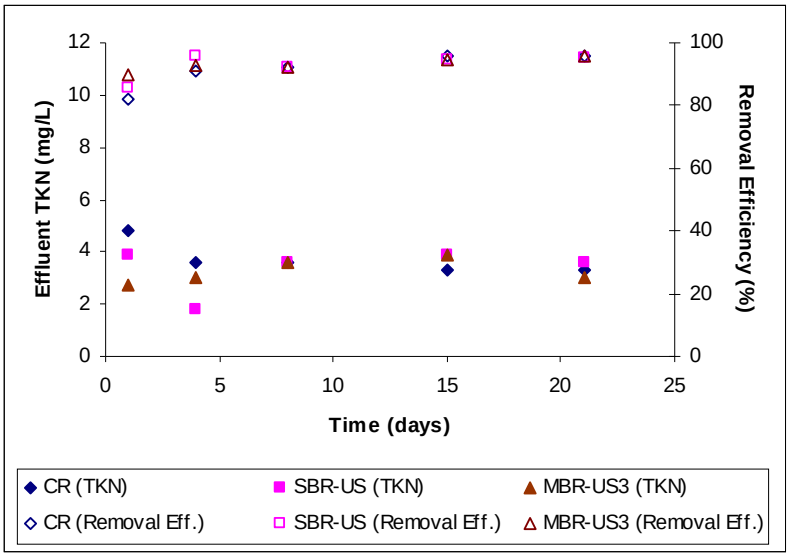


Figure 4.36. Variations of effluent TKN and TKN removal efficiency in the second case.

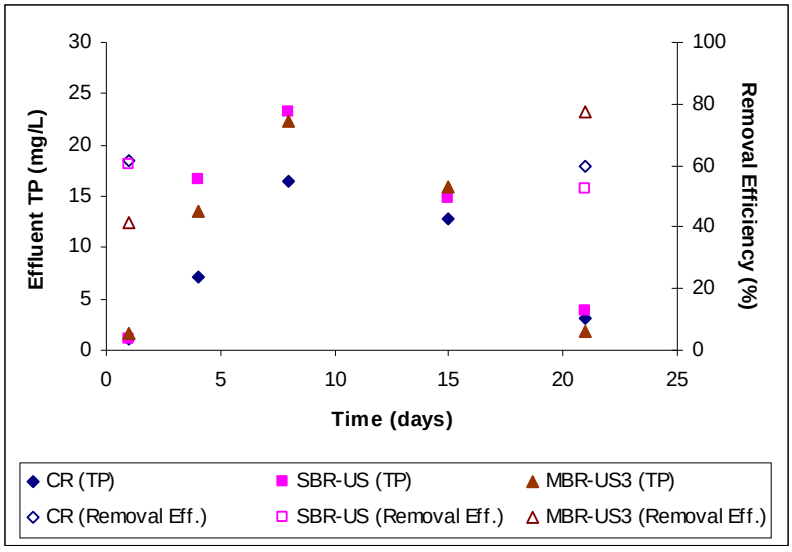


Figure 4.37. Variations of effluent TP and TP removal efficiency in the second case.

In the second case, the effluent quality of all reactors was better than that in the first case. Although the addition of disintegrated sludge caused to an increase in the organic loading in the SBR-US and MBR-US3, the effluent quality of these systems was not deteriorated. In these two systems, high effluent quality could be achieved by increasing the sonication time intervals.

4.4. Sequential Batch Reactor-Ultrasonic Sludge Disintegration (SBR-SD) Combined Systems for Sludge Minimization

Three laboratory-scale sequential batch reactors (SBRs) were operated in parallel at different ratios of sonicated sludge in order to determine an optimum sonicated sludge volume ratio. One of these three reactors was used as control reactor (CR) and two of them were pretreatment reactors (R1 and R2) in which a part of the sludges in the reactors was sonicated and returned into these sytems for the purpose of sludge minimization. In the first three days of operation, sonication application was not applied to R1 and R2 in order to observe the sludge production. Then, in the 4th day sonication was applied the R1 and R2 systems as described in Section 3.2.1.3, and this batch process was repeated once three days. The operational conditions for SBRs are summarized in Table 3.8.

4.4.1. Untreated Sludge and Feedwater Characteristics

Sludge samples and feedwater were collected from Paşaköy Advanced Biological Wastewater Treatment Plant. The characterization of the untreated sludge samples is listed in Table 4.12. The characterization of feedwater is given in Section 4.3.2 (Table 4.9).

Table 4.12. Characterization of the untreated sludge for SBR-US systems experiments.

Parameter	Range	Unit
TS	7080-7245	mg/L
TVS	4044-4145	mg/L
MLSS	6360-6450	mg/L
MLVSS	3790-3950	mg/L
MLVSS/MLSS	0.60-0.61	-
SVI	126-138	mL/g
TCOD	5200-5500	mg/L
SCOD	26.4-33.6	mg/L
Viscosity	19.0-19.2	mPa s
CST	10.5-10.9	s
pH	7.10-7.30	-

4.4.2. The Variations of MLSS Concentrations in Reactors

The results of MLSS experiments during 13-day of operation time are given in Figure 4.38. In the first three days of operation, sonication application was not applied to pretreatment reactors (R1 and R2) therefore, in all reactors an increase was observed in the MLSS concentration. In the 4th day of operation, to keep the MLSS concentration in the pretreatment reactors around 6400 mg/L, sonication applied to the R1 and R2 at the sonication ratio of 1:60 (v:v) and 1:30 (v:v), respectively. After 10 days of operation, although the MLSS concentration of CR continued to increase, it was maintained at 6560 mg/L and 6490 mg/L at the pretreatment reactors of R1 and R2.

After 10 days of operation, the MLSS concentration increased from 6540 mg/L to 7060 mg/L, it increased from 6500 mg/L to 6560 mg/L in the R1 and from 6480 mg/L to 6490 mg/L in the R2. Therefore, it was determined that the sludge sonication ratio of 1:30 (v:v) was the optimum value in order to achieve the zero excess sludge production.

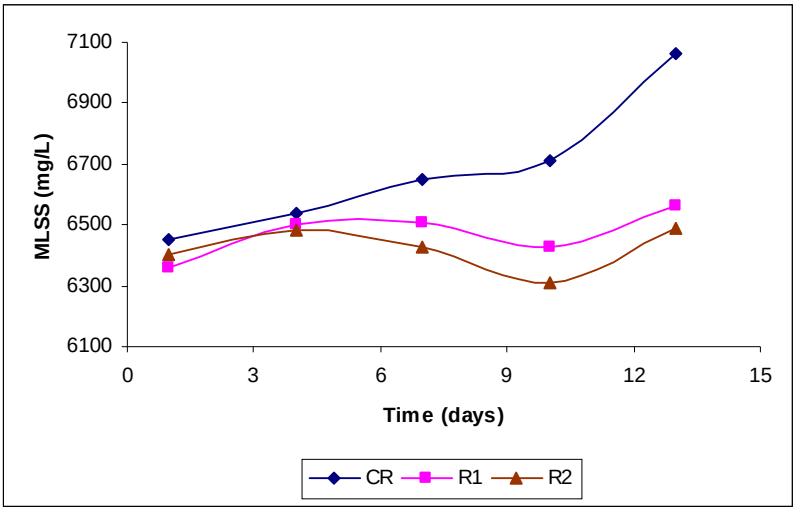


Figure 4.38. Variations of MLSS concentration in the SBRs.

4.4.3. The Variations of SVI in Reactors

The variations of SVI are shown in Figure 4.39. In the all reactors, the SVI improved; it decreased from 131 mL/g to 121 mL/g in the CR, from 136 mL/g to 127 mL/g in the R1 and, from 139 mL/g to 132 mL/g in the R2. Although some parts of disintegrated sludge returned into the R1 and R2, SVI deterioration was not observed in these systems.

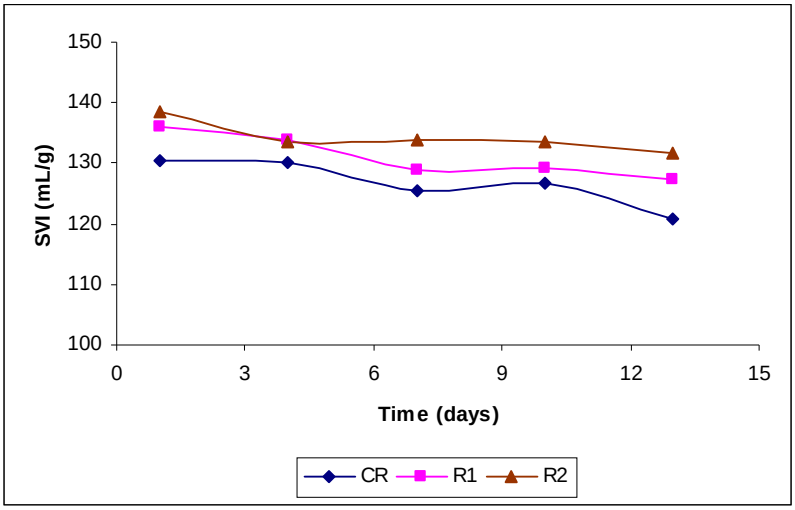


Figure 4.39. Variations of SVI in the SBRs.

4.4.4. The Variations of Sludges Rheology in Reactors

In order to determine the effect of ultrasonically disintegration sludge on the reactors sludge rheology, viscosity of reactor sludges was measured, the results are given in Figure 4.40. Due to an increase in MLSS concentration of CR, the viscosity of system increased from 19.2 mPa s to 21.0 mPa s. After the 4th day of operation, the viscosity of R1 and R2 started to decreased due to the returned disintegrated sludge. The viscosity decreased from 19.2 mPa s to 16.8 mPa s in the R1 and from 19.2 mPa s to 16.2 mPa s in the R2.

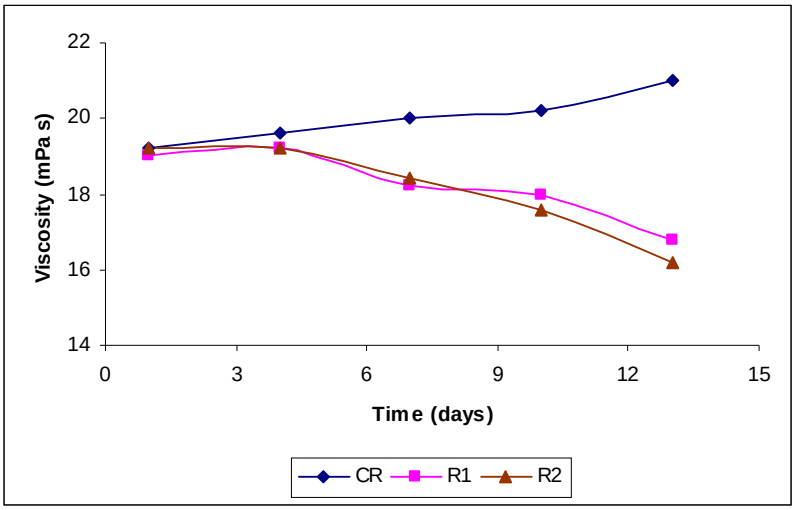


Figure 4.40. Variations of viscosity in the SBRs.

4.4.5. The Variations of SCOD Concentrations of Sludges in Reactors

In order to observe the change in soluble organic concentrations of reactors, SCOD of sludges was monitored (Figure 4.41). In the CR, the SCOD concentration was almost stable at the range between 30-35 mg/L, because there was not an extra organic loading into the system. After the 4th day of operation, there was an additional organic loading caused by disintegrated sludge feeding in the R1 and R2. Therefore, SCOD concentration in the R1 and R2 systems increased after the day of 4th.

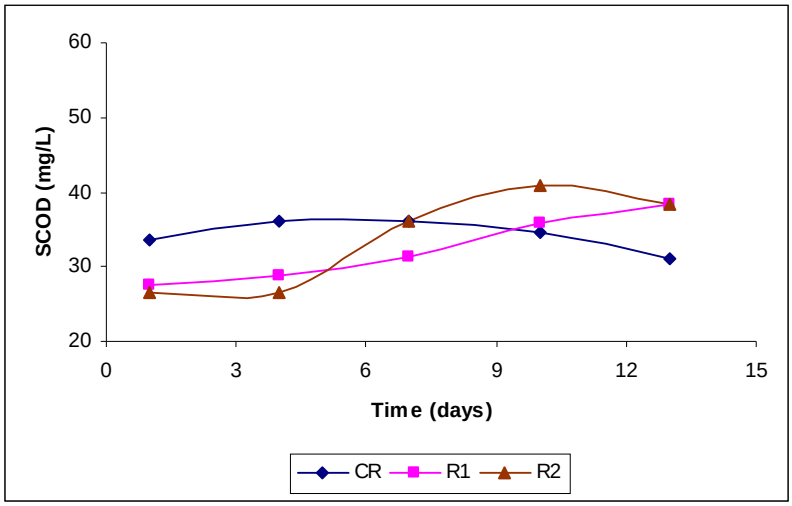


Figure 4.41. Variations of SCOD concentration of sludges in the SBRs.

4.4.6. The Variations of Dewaterability of Sludges in Reactors

In order to determine the effect of ultrasonically disintegrated sludge returned into reactors on the dewaterability of sludges in the reactors, CST measurements were conducted. The variations of CST are given in Figure 4.42. The CST of CR increased from 10.5 s to 12.7 s caused by increase of MLSS. The dewaterability of the R1 and R2 systems has been deteriorated after feeding ultrasonically disintegrated sludges into these systems.

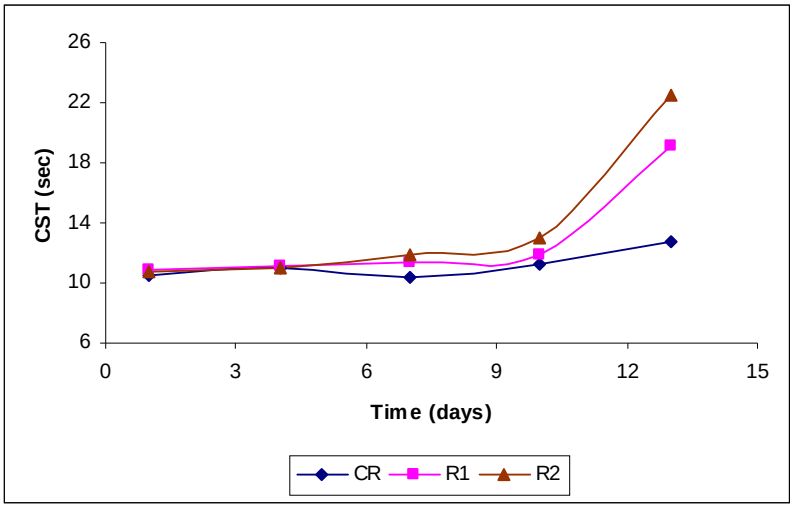


Figure 4.42. Variations of CST in the SBRs.

4.4.7. The Variations of Particle Size Distribution of Sludges in Reactors

The results of particle size distribution are given in Figure 4.43 and Table 4.13. The particle size of R1 and R2 reduced after 13-day operation time while the particle size of CR was maintained almost constant. Ultrasonically disintegrated sludge was fed into the R1 and R2 systems and this caused the reduction in the median diameter, d_{50}, and therefore small particles increased. The mean diameter of R1 sludges decreased from 91 µm to 84 µm and the mean diameter of R2 sludges decreased from 103 µm to 84 µm. The particles size of CR sludges was stable around 90 µm. The decrease in the mean diameter of R1 sludges was less than that of R2 sludges because ultrasonically disintegrated sludge volume of R1 system (50 mL) was less than that of R2 system (100 mL).

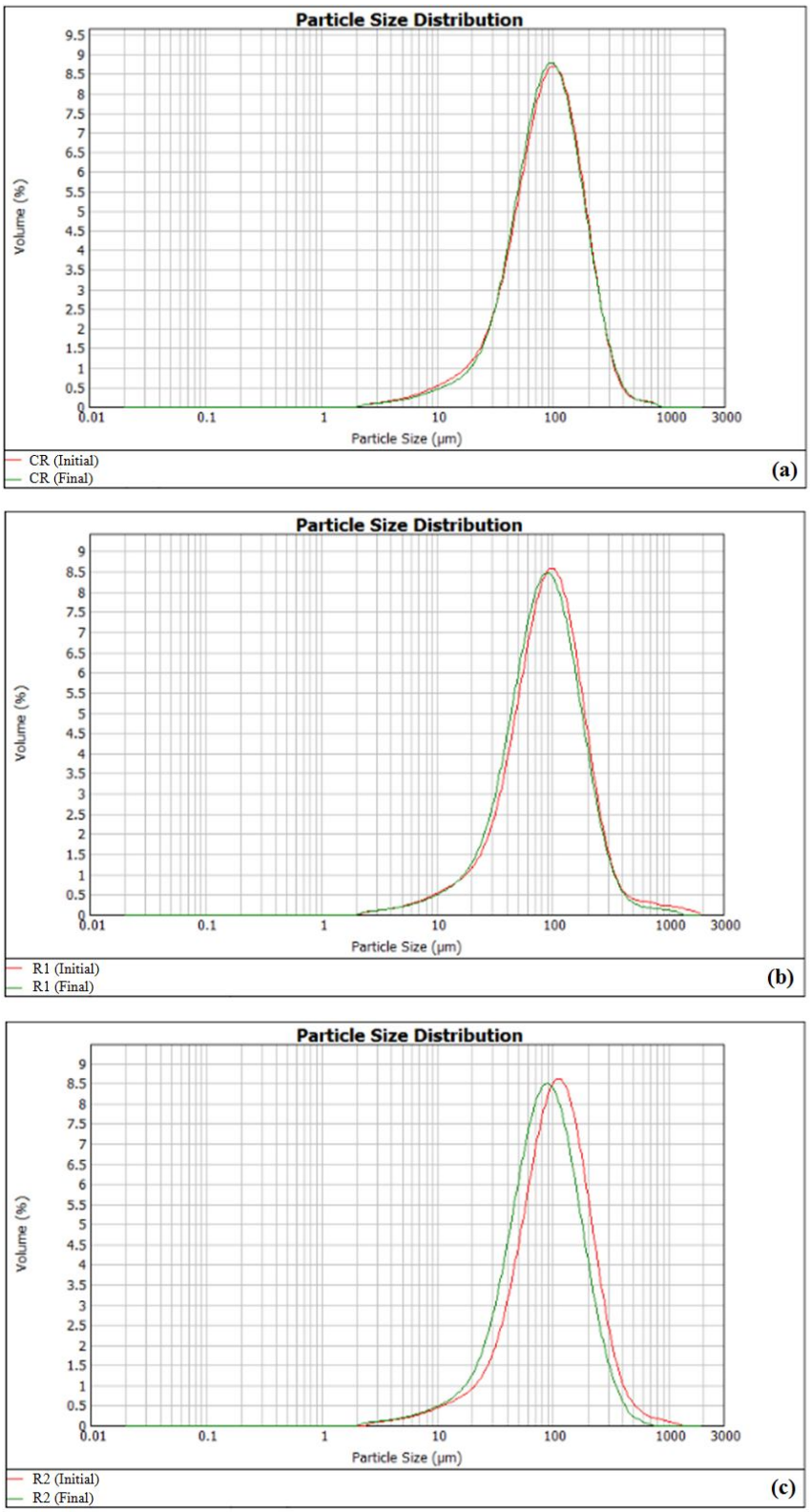


Figure 4.43. Variations of particle size distribution in (a) CR (b) R1 and (c) R2.

Table 4.13. Particle size of sludge samples of SBR systems at the beginning and at the end of operation.

Reactor	Experiment Time	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
CR	1st day	29.641	90.069	206.045
	13th day	31.769	89.459	206.563
R1	1st day (before US application)	29.975	91.021	219.260
	13th day (after US application)	28.867	84.480	205.025
R2	1st day (before US application)	34.750	103.247	244.853
	13th day (after US application)	29.148	83.929	201.653

4.4.8. The Effect of Ultrasonic Sludge Disintegration on the Effluent Quality

In order to determine the effect of ultrasonic pretreatment application on the effluent quality of the reactors, the influent COD and the effluent COD were analyzed. The variations of effluent COD and COD removal efficiency are shown in Figure 4.44.

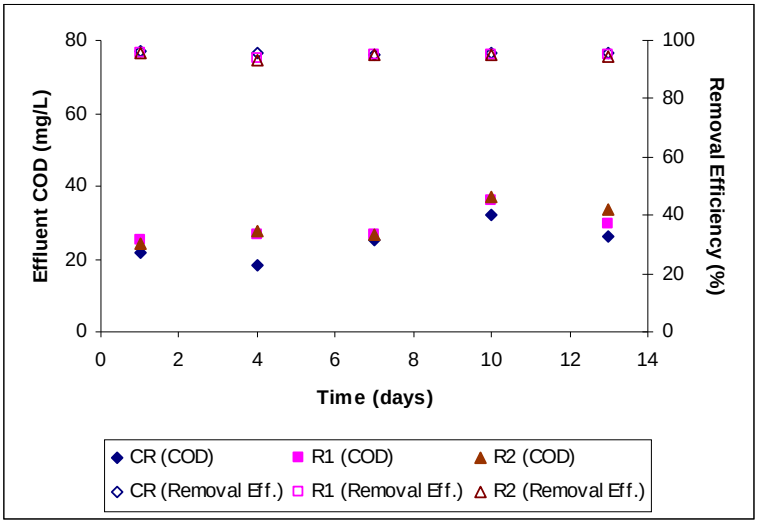


Figure 4.44. Variations of effluent COD and COD removal efficiency in SBRs.

The average COD removal efficiency was 96%, 95% and 95% for the CR, R1 and R2, respectively. Although additional organic loading caused by the recycle of ultrasonically disintegrated sludge supplied to the R1 and R2, the effluent quality of these systems was not deteriorated. The sonication time intervals were considered to be a reason for this situation: instead of daily sonication, the part of excess sludge of R1 and R2 systems was disintegrated and returned back into the systems once three days.

In order to determine the effect of recycled disintegrated sludge on the effluent quality of the reactors, the effluent turbidity was also analyzed (Figure 4.45). The results showed that the effluent turbidity of CR was at the level of 1.30 NTU and it did not change during the operation. During the first three days of operation, no change was observed in the effluent turbidity of R1 and R2. After the 4th day of operation, there was an additional organic loading caused by disintegrated sludge feeding in the R1 and R2. Therefore, effluent turbidity in the R1 and R2 systems increased gradually after the day of 4th.

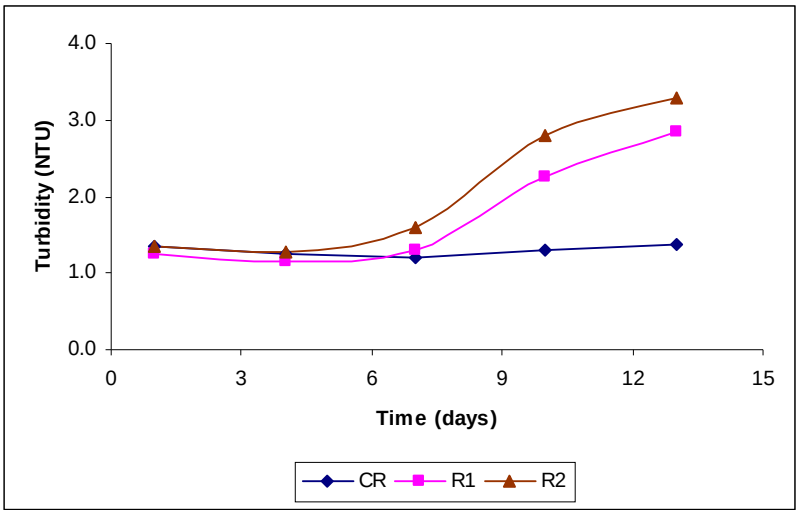


Figure 4.45. Variations of effluent turbidity in SBRs.

5. CONCLUSIONS

This study investigated the effect of ultrasonic sludge disintegration on the minimization of excess sludge production in the treatment of municipal wastewater by using membrane bioreactors (MBRs). This study derived several conclusions that can be summarized under two topics:

1) The main results of ultrasonic sludge disintegration batch experiments

At the first stage of the study, the series of batch studies were carried out to investigate the effect of ultrasonic disintegration on sludge properties. This study demonstrated that ultrasonicaiton is an effective technique for disintegrating sludge and changing its characteristics. The following findings emphasize the main results of this stage of the study.

- Ultrasonic disintegration pretreatment increases the solubility of the organic matter in sludge depending on the applied SE and leads to an improvement in the biodegradability of the sludge.

  The SCOD of ultrasonically disintegrated sludge increased strongly for SE between 0 and 25000 kJ/kg TS. For higher SE values, the rate of increase of SCOD was lower. For this study, the minimum SE of disintegration process required to break cells was found to be about 2000 kJ/kg TS.

- The disintegration degree (DD_{COD}) of ultrasonically disintegrated sludges calculated according to Müller formula exceeded 100% for SE values higher than 26000 kJ/kg TS. It seems that for SE values higher than 26000 kJ/kg TS, ultrasonication is a more effective disintegration method than NaOH alkaline treatment.

- The supernatant turbidities of ultrasonically disintegrated sludge samples increased with the increasing SE applied to the sludges due to the increase of micro particles,

released from sludge flocs during the ultrasonic disintegration process. The supernatant turbidity of ultrasonically disintegrated sludges had same trend with their SCOD as an indirect indicator of disintegration.

- Ultrasonic disintegration improved the sludge settling properties when only the low value of SE is applied to sludges. An increase in SE caused to a decrease in the sludge settling velocity.

The optimum SE of ultrasonic disintegration was found to be 2000 kJ/kg TS in order to enhance the sludge settling velocity. Ultrasonic disintegration for SE higher than 2000 kJ/kg TS deteriorated the sludge settling properties.

- Ultrasonic disintegration deteriorated the filterability of sludge samples by decreasing the size of sludge particles. CST of the sludge samples increased even for the application of low values of SE.

The CST of ultrasonically disintegrated sludge increased strongly until the SE of about 50000 kJ/kg TS. For higher SE values, the rate of increase of CST was lower.

- Ultrasonic disintegration deteriorated the rheological properties of the sludge samples with the application of increasing SE.

The value of viscosity decreased strongly until the SE of about 8500 kJ/kg TS. For SE values higher than 8500 kJ/kg TS, the rate of decrease of viscosity was lower.

- Ultrasonic disintegration caused to a decrease in the MLSS concentration and particles size distribution until a certain value of SE. With the increasing SE applications, sludge particle size and MLSS values increased again.

The MLSS concentration and the median diameters, d_{50}, of ultrasonically disintegrated sludges decreased for SE values lower than 50000 kJ/kg TS. For higher values of SE the median diameters increased, indicating the reflocculation of the sludge particles which confirmed by the MLSS experiments also.

2) The main results of MBR-US combined systems experiments

At the second stage of the study, MBR systems with different amounts of ultrasonically disintegrated excess sludge were operated in two cases to evaluate the effect of sludge sonication conditions on the sludge minimization and effluent quality. In the first case, the effect of pretreatment through ultrasonic sludge disintegration on the MBR performance was investigated (by comparing CMBR, MBR-US1 and MBR-US2). In the second case, it was focused on both filter performance and sludge minimization (by comparing CR, SBR-US and MBR-US3). The following findings emphasize the main results of the second stage of the study.

- The results of the second stage of the study showed that the MBR-US combined systems can be successfully operated for wastewater treatment in order to minimize excess sludge production without any deterioration in the effluent quality and the membrane performances.

- The application of ultrasonic sludge disintegration to a part of the excess sludge, returned back into the reactor, increased the solubility and so the biodegradability of sludge and minimized the sludge production in the MBR.

- In the MBR-US systems, ultrasonic disintegration applied as needed basis instead of fixed periodical time intervals is the most appropriate method for reaching to zero excess sludge production and high effluent quality.

- When reactors are fed with real wastewater instead of synthetic wastewater, the sonication frequency of as needed basis should be preferred to daily sonication.

- The MBR-US systems can tolerate the negative effects of returned disintegrated excess sludge on the effluent quality when the sonication time interval increases.

- The MBR systems are more efficient in tolerating the effects of returned disintegrated excess sludge than the SBR systems.

- The enhancement of nutrient removal is possible in MBR-US combined systems.

- While sonication of smaller volumes of sludges led to higher DD and SE values, the sonicaiton of larger volumes caused to higher MLSS reduction. This effect can be explained with the disintegration of higher amounts of sludge that will lead to lower amount of excess sludge production.

REFERENCES

APHA, AWWA, WEF, 1998. Standard methods for the examination of water and wastewater. 20th edition, American Public Health Association/American Water Works Association/Water Environment Federation, Washington, D.C.

Bandelin, 2009. Sonopuls Ultrasonic Homogenizers Operating Instructions. Bandelin Electronic GmbH & Co. KG.

Boucaud, A., Felix, N., Pizarro, L., Patat, F., 1999. High power low frequency ultrasonic transducer: vibration amplitude measurements by an optical interferometric method. IEEE Ultrasonics Symposium, 1095-1098.

Bougrier, C., Carrère, H., Delgenès, J. P., 2005. Solubilisation of waste-activated sludge by ultrasonic treatment. Chemical Engineering Journal, 106, 163–169.

Bougrier, C., Delgenès, J.P., Carrère, H., 2007. Impacts of thermal pre-treatments on the semi-continuous anaerobic digestion of waste activated sludge. Biochemical Engineering Journal, 34, 20-27.

Bougrier, C., Delgenès, J.P., Carrère, H., 2008. Effects of thermal treatments on five different waste activated sludge samples solubilization, physical properties and anaerobic digestion. Chemical Engineering Journal, 139, 236-244.

Cao, X. Q., Liu, L., Wang, H. C., Gan, Y. P., Zhou, J., Bai, Y., 2006. Enhanced sludge decomposition by ultrasound. 607-614.

Carrère, H., Dumas, C., Battimelli, A., Batstone, D.J., Delgenès, J.P., Steyer, J.P., Ferrer, I., 2010. Pretreatment methods to improve sludge anaerobic degradability: A review. Journal of Hazardous Materials, 183, 1-15.

Chu, C. P., Chang, B-V., Liao, G. S., Jean, D. S., Lee, D. J., 2001. Observations on changes in ultrasonically treated waste-activated sludge. Water Research, 35 (4), 1038-1046.

Chu, L., Yan, S., Xing, X. H., Sun, X., Jurcik, B., 2009. Progress and perspectives of sludge ozonation as a powerful pretreatment method for minimization of excess sludge production. Water Research, 43, 1811-1822.

Cicek, N., 2003. A review of membrane bioreactors and their potential application in the treatment of agricultural wastewater. Canadian Biosystems Engineering, 45, 6.37-6.49.

Clark, P. B., Nujjoo, I., 1998. Ultrasonic sludge pretreatment for enhanced sludge digestion. Innovation 2000 Conference, Cambridge, UK, 7-10 July 1998.

Clouzot, L., Roche, N., Marrot, B., 2011. Effect of membrane bioreactor configurations on sludge structure and microbial activity. Bioresource Technology, 102, 975-981.

Dewil, R., Baeyens, J., Neyens, E., 2005. Fenton peroxidation improves the drying performance of waste activated sludge. Journal of Hazardous Materials, B117, 161-170.

Dewil, R., Baeyens, J., Goutvrind, R., 2006a. The use of ultrasonics in the treatment of waste activated sludge. Chinese J. Chemical Engineering, 14(1), 105–113.

Dewil, R., Baeyens, J., Goutvrind, R., 2006b. Ultrasonic treatment of waste activated sludge. Environmental Progress, 25(2), 121–128.

Doğan, I., Sanin, F. D., 2009. Alkaline solubilization and microwave irradiation as a combined sludge disintegration and minimization method. Water Research, 43, 2139-2148.

Drews, A., 2010. Membrane fouling in membrane bioreactors–Characterization, contradictions, cause and cures. Journal of Membrane Science, 363, 1-28.

El-Hadj, T. B., Dosta, J., Márquez-Serrano, R., Mata- Álvarez, J., 2007. Effect of ultrasound pretreatment in mesophilic and thermophilic anaerobic digestion with emphasis on naphthalene and pyrene removal. Water Research, 41, 87-94.

Elliott, A., Mahmood, T., 2007. Pretreatment technologies for advancing anaerobic digestion of pulp and paper biotreatment residues. Water Research, 41, 4273-4286.

Feng, X., Lei, H., Deng, J., Yu, Q., Li, H., 2009. Physical and chemical characteristics of waste activated sludge treated ultrasonically. Chemical Engineering and Processing, 48, 187-194.

Ferraris, M., Innella, C., Spagni, A., 2009. Start-up of a pilot-scale membrane bioreactor to treat municipal wastewater. Desalination, 237, 190-200.

Filibeli, A., Kaynak, G. E., 2006. Arıtma çamuru miktarının azaltılması ve özelliklerinin iyileştirilmesi amacıyla yapılan ön işlemler. İTÜ Dergisi, Su Kirlenmesi Kontrolü, 16 (1-3), 3-12.

Gonze, E., Pillot, S., Valette, E., Gonthier, Y., Bernis, A., 2003. Ultrasonic treatment of an aerobic activated sludge in a batch reactor. Chemical Engineering and Processing, 42, 965-975.

Gündüz, Ç., 2009. Ultrasonic disintegration of sewage sludge. M.S. Thesis. Dokuz Eylül University.

Guo, W. S., Vigneswaran, S., Ngo, H. H., Xing W., 2008. Comparison of membrane bioreactor systems in wastewater treatment. Desalination, 231, 61-70.

Guo, W.S., Vigneswaran, S., Ngo, H.H., Kandasamy, J., Yoon, S., 2008. The role of a membrane performance enhancer in a membrane bioreactor: a comparison with other submerged membrane hybrid systems. Desalination, 231, 305-313.

Hach, 1997. DR/2010 Spectrophotometer Procedures Manual. Hach Company, USA.

Hach, 1999. Digesdahl Digestion Apparatus Instrument Manual. Hach Company, USA.

Hasar, H., Kinaci, C., 2004. Comparison of a sMBR and a CASP System for Wastewater Reclamation and Re-use. ISSN 0015-1882/04, Filtration+Separation, 35-39.

Hasar, H., Kinaci, C., Ünlü, A., Toğrul, H., Ipek, U., 2004. Rheological properties of activated sludge in a sMBR. Biochemical Engineering Journal, 20, 1-6.

He, J., Wan, T., Zhang, G., Yang, J., 2011. Ultrasonic reduction of excess sludge from activated sludge system: Energy efficiency improvement via operation optimization. Ultrasonics Sonochemistry, 18, 99-103.

He, S., Xue, G., Wang, B., 2006. Activated sludge ozonation to reduce sludge production in membrane bioreactor (MBR). Journal of Hazardous Materials, B135, 406-411.

Howell, J. A., Arnot, T. C., Liu, W., 2003. Membrane bioreactors for treating waste streams. New York Academy of Sciences, 984, 411-419.

Huan, L., Yiying, J., Mahar, R. B., Zhiyu, W., Yongfeng, N., 2009. Effects of ultrasonic disintegration on sludge microbial activity and dewaterability. Journal of Hazardous Materials, 161, 1421-1426.

Huysmans, A., Weemaes, M., Fonseca, P. A., Verstraete, W., 2001. Ozonation of activated sludge in the recycle stream. Journal of Chemical Technology and Biotechnology, 76, 321-324.

International Water Association, the IWA WaterWiki Articles.
http://www.iwawaterwiki.org/xwiki/bin/view/Articles/SequencingBatchReactor_R
(accessed January 2011)

International Water Association, the IWA WaterWiki Articles.
http://www.iwawaterwiki.org/xwiki/bin/view/Articles/SludgeProduction.
(accessed January 2011)

International Water Association, the IWA WaterWiki Articles. http://www.iwawaterwiki.org/xwiki/bin/view/Articles/REDUCTIONOFSLUDGEPRODUCTIONINWASTEWATERTREATMENTPLANTS. (accessed January 2011)

Judd, S., 2006. The MBR Book: Principles and Applications of Membrane Bioreactors in Water and Wastewater Treatment, First ed., Elsevier Ltd., Oxford.

Judd, S., 2008. The status of membrane bioreactor technology (Review). TRENDS in Biotechnology, 26 (2), 109-116.

Kawasaki, K., Matsuda, A., Tanabe, S., Katagiri, N., Iritani, E., 2007. Effect of suction pressure and aeration on the hollow fibre microfiltration of excess activated sludge. Trans IChemE, Part B, Process Safety and Environmental Protection, 85(B2): 176-180.

Khanal, S. K., Grewell, D., Sung, S., Leeuwen, J. H. V., 2007. Ultrasound Applications in Wastewater Sludge Pretreatment: A Review. Critical Reviews in Environmental Science and Technology, 37, 277-313.

Kim, D-H, Jeong, E., Oh, S-E, Shin, H-S, 2010. Combined (alkaline+ultrasonic) pretreatment effect on sewage sludge disintegration. Water Research, 44, 3093-3100.

Koda, S., Kimura, T., Kondo, T., Mitome, H., 2003. A standard method to calibrate sonochemical efficiency of an individual reaction system. Ultrasonics Sonochemistry, 10, 149-156.

Le-Clech, P., Fane, A., Leslie, G., 2005. MBR focus: The operators' perspective. Industry Focus, Filtration+Separation, 20-23.

Le-Clech, P., Chen, V., Fane, T. A. G., 2006. Fouling in membrane bioreactors used in wastewater treatment. Journal of Membrane Science, 284, 17–53.

Le-Clech, P., 2010. Membrane bioreactors and their uses in wastewater treatments. Appl Microbiol Biotechnol, 88, 1253-1260.

Lee, J.W., Cha, H.-Y., Park, K.Y., Song, K.-G., Ahn, K.-H., 2005. Operational strategies for an activated sludge process in conjunction with ozone oxidation for zero excess sludge production during winter season. Water Research, 39, 1199-1204.

Lehne, G., Müller, A., Schwedes, J., 2001. Mechanical disintegration of sewage sludge. Water Science and Technology, 43 (1), 19-26.

Li, H., Jin, Y., Mahar, R., Wang, Z., Nie, Y., 2008. Effects and model of alkaline waste activated sludge treatment. Bioresource Technology, 99, 5140-5144.

Liu, Y., Tay, J-H., 2001. Strategy for minimization of excess sludge production from the activated sludge process. Biotechnology Advances, 19, 97-107.

Liu, Y., 2003. Chemically reduced excess sludge production in the activated sludge process. Chemosphere, 50, 1-7.

Mahmood, T., Elliott, A., 2006. A review of secondary sludge reduction technologies for the pulp and paper industry. Water Research, 40, 2093-2112.

Malvern, 2007. Hydro 2000MU User Manual. Malvern Instruments Ltd.

Mason, T., Peters, D., 2002. Practical Sonochemistry: Uses and Applications of Ultrasound, Second Ed., Horwood Publishing Limited International Publishers, England.

Melin, T., Jefferson, B., Bixio, D., Thoeye, C., De Wilde, W., De Koning, J., Van Der Graaf, J., Wintgens, T., 2006. Membrane bioreactor technology for wastewater treatment and reuse. Desalination, 187, 271-282.

Metcalf & Eddy, 2004. Wastewater Engineering: Treatment and Reuse, Fourth Ed., Revised by Tchobanoglous, G., Burton, F. L., Stensel, H. D., McGraw- Hill Companies Inc., New York, USA.

Ming-he, H., Chao-hai, W., 2010. Performance of membrane bioreactor (MBR) system with sludge Fenton oxidation process for minimization of excess sludge production. Journal of Hazardous Materials, 176, 597-601.

Moonkhum, M., 2007. Aerobic digestion of waste activated sludge with ultrasonic pretreatment, M.S. Thesis, Asian Institute of Technology.

Moussavi, G., Asilian, H., Jamal, A., 2008. Effect of Ozonation on Reduction of Volume and Mass of Waste Activated Sludge. Journal of Applied Sciences Research, 4 (2), 122-127.

Müller, J., 2000. Disintegration as a key-step in sewage sludge treatment. Water Science and Technology, 41 (8), 123-130.

Müller, J.A., Winter, A., Strünkmann, G., 2004. Investigation and assessment of sludge pre-treatment processes. Water Science and Technology, 49 (10), 97-104.

Navaneethan, N., 2007. Anaerobic digestion of waste activated sludge with ultrasonic pretreatment, M.S. Thesis, Asian Institute of Technology.

Oh, Y-K, Lee, K-R, Ko, K-B, Yeom, I-T, 2007. Effects of chemical sludge disintegration on the performances of wastewater treatment by membrane bioreactor. Water Research, 41, 2665-2671.

Pérez-Elvira, S. I., Diez, P. N., Fdz-Polanco, F., 2006. Sludge minimization technologies. Reviews in Environmental Science and Bio/Technology, 5, 375-398.

Pham, T. T. H., Brar, S. K., Tyagi, R. D., Surampalli, R. Y., 2009. Ultrasonication of wastewater sludge–Consequences on biodegradability and flowability. Journal of Hazardous Materials, 163, 891-898.

Pilli, S., Bhunia, P., Yan, S., LeBlanc, R.J., Tyagi, R.D., Surampalli, R.Y., 2011. Ultrasonic pretreatment of sludge: A review. Ultrasonics Sonochemistry, 18, 1-18.

Prasad, K., Pinjari, D.V., Pandit, A.B., Mhaske, S.T., 2010. Synthesis of titanium dioxide by ultrasound assisted sol–gel technique: Effect of amplitude (power density) variation. Ultrasonics Sonochemistry, 17, 697-703.

Ramakrishna, D. M., Viraraghavan, T., 2005. Strategies for Sludge Minimization in Activated Sludge Process: A Review. Fresenius Environmental Bulletin, 14 (1), 2-12.

Roxburgh, R., Sieger, R., Johnson, B., Rabinowitz, B., Goodwin, S., Crawford, G., Daigger, G., 2006. Sludge Minimization Technologies-Doing more to get Less. Proceedings of the Water Environment Federation, WEFTEC, 20, 506-525.

Sakai, Y., Fukase, T., Yasui, H., Shibata, M., 1997. An activated sludge process without excess sludge production. Water Science and Technology, 36 (11), 163-170.

Salsabil, M. R., Prorot, A., Casellas, M., Dagot, C., 2009. Pre-treatment of activated sludge: Effect of sonication on aerobic and anaerobic digestibility. Chemical Engineering Journal, 148, 327-335.

Shim, J. K., Yoo, I-K., Lee, Y. M., 2002. Design and operation considerations for wastewater treatment using a flat submerged membrane bioreactor. Process Biochemistry, 38, 279-285.

Sivakumar, M., Pandit, A. B., 2001. Ultrasound enhanced degradation of Rhodamine B: optimization with power density. Ultrasonics Sonochemistry, 8, 233-240.

Song, K-G, Choung, Y-K, Ahn, K-H, Cho, J, Yun, H., 2003. Performance of membrane bioreactor system with sludge ozonation process for minimization of excess sludge production. Desalination, 157, 353-359.

Teo, K. C., Xu, Y., Yang, C., 2001. Sonochemical degradation for toxic halogenated organic compounds. Ultrasonics Sonochemistry, 8, 241-246.

Tian, J-Y., Liang, H., Nan, J., Yang, Y-L., You, S-J., Li, G-B., 2009. Submerged membrane bioreactor (sMBR) for the treatment of contaminated raw water. Chemical Engineering Journal, 148, 296–305.

Tiehm, A., Nickel, K., Zellhorn, M., Neis, U., 2001. Ultrasonic waste activated sludge disintegration for improving anaerobic stabilization. Water Research, 35 (8), 2003-2009.

Tokumura, M., Sekine, M., Yoshinari, M., Znad, H. T., Kawase, Y., 2007. Photo-Fenton process for excess sludge disintegration. Process Biochemistry, 42, 627-633.

Tokumura, M., Katoh, H., Katoh, T., Znad, H.T., Kawase, Y., 2009. Solubilization of excess sludge in activated sludge process using the solar photo-Fenton reaction. Journal of Hazardous Materials, 162, 1390-1396.

Trussell, R. S., Adham, S., Trussell, R. R., 2005. Process Limits of Municipal Wastewater Treatment with the Submerged Membrane Bioreactor. Journal of Environmental Engineering, 131 (3), 410-416.

Turovskiy, I. S., Mathai, P. K., 2006. Wastewater Sludge Processing. John Wiley & Sons Inc., Hoboken, New Jersey

Ueda, T., Hata, K., Kikuoka, Y., Seino, O., 1997. Effects of aeration on suction pressure in a submerged membrane bioreactor. Water Research, 31, 489-494.

Uggetti, E., Ferrer, I., Llorens, E., Garcia, J., 2010. Sludge treatment wetlands: A review on the state of the art. Bioresource Technology, 101, 2905-2912.

Van Kaam, R., Anne-Archard, D., Alliet Gaubert, M., Albasi, C., 2008. Rheological characterization of mixed liquor in a submerged membrane bioreactor: Interest for process management. Journal of Membrane Science, 317, 26-33.

Wang, F., Wang, Y., Ji, M., 2005. Mechanisms and kinetics models for ultrasonic waste activated sludge disintegration. Journal of Hazardous Materials, B123, 145-150.

Wang, Z., Wang, L., Wang, B. Z., Jiang, Y. F., Liu, S., 2008. Bench-scale study on zero excess activated sludge production process coupled with ozonation unit in membrane bioreactor. Journal of Environmental Science and Health, 43, 1325-1332.

Wei, Y., Houten, R. T. V., Borger, A. R., Eikelboom, D. H., Fan, Y., 2003. Minimization of excess sludge production for biological wastewater treatment. Water Research, 37, 4453-4467.

Winter, A., 2002. Minimization of costs by using disintegration at a full-scale anaerobic digestion plant. Water Science and Technology, 46 (4-5), 405-412.

Wojciechowska, E., 2005. Application of microwaves for sewage sludge conditioning. Water Research, 39, 4749-4754.

Wu, Z., Wang, Z., Zhou, Z., Yu, G., Gu, G., 2007. Sludge rheological and physiological characteristics in a pilot-scale submerged membrane bioreactor. Desalination, 212, 152-164.

Yan, Y., Feng, L., Zhang, C., Zhu, H., Zhou, Q., 2010. Effect of ultrasonic specific energy on waste activated sludge solubilization and enzyme activity. African Journal of Biotechnology, 9 (12), 1776-1782.

Yang, W., Cicek, N., 2008. Treatment of swine wastewater by submerged membrane bioreactors with consideration of estrogenic activity removal. Desalination, 231, 200-208.

Yasui, H., Nakamura, K., Sakuma, S., Iwasaki, M., Sakai, Y., 1996. A full-scale operation of a novel activated sludge process without excess sludge production. Water Science and Technology, 34 (3-4), 395-404.

Yin, X., Han, P., Lu, X., Wang, Y., 2004. A review on the dewaterability of bio-sludge and ultrasound pretreatment. Ultrasonics Sonochemistry, 11, 337-348.

Yin, X., Lu, X., Han, P., Wang, Y., 2006. Ultrasonic treatment on activated sewage sludge from petro-plant for reduction. Ultrasonics, 44, e397-e399.

Yoon, S-H, 2003. Important operational parameters of membrane bioreactor-sludge disintegration (MBR-SD) system for zero excess sludge production. Water Research, 37, 1921-1931.

Yoon, S-H., Kim, H-S., Lee, S., 2004. Incorporation of ultrasonic cell disintegration into a membrane bioreactor for zero sludge production. Process Biochemistry, 39, 1923-1929.

Yu, Q., Lei, H., Li, Z., Li, H., Chen, K., Zhang, X., Liang, R., 2010. Physical and chemical properties of waste-activated sludge after microwave treatment. Water Research, 44, 2841-2849.

Zhang, G., Zhang, P., Yang, J., Chen, Y., 2007. Ultrasonic reduction of excess sludge from the activated sludge system. Journal of Hazardous Materials, 145, 515-519.

Zhang, G., He, J., Zhang, P., Zhang, J., 2009. Ultrasonic reduction of excess sludge from the activated sludge system II: Urban sewage treatment. Journal of Hazardous Materials, 164, 1105-1109.

Zhang, G., Yang, J., Liu, H., Zhang, J., 2009. Sludge ozonation: Disintegration, supernatant changes and mechanisms. Bioresource Technology, 100, 1505-1509.

Zhang, P., Zhang, G., Wang, W., 2007. Ultrasonic treatment of biological sludge: Floc disintegration, cell lysis and inactivation. Bioresource Technology, 98, 207-210.

APPENDIX A

The Calibration of Ultrasonicator

Table A.1. Results of ultrasonicator calibration for 2000-mL solution volume.

2000-mL Solution Volume				
Power Output and Amplitude		**Ultrasonic Power (Watt)**	**Power Density (Watt/mL)**	**Power Intensity (W/cm^2)**
80 % Amplitude	**100 W**	44.4	0.022	9.04
	150 W	72.8	0.036	14.83
	200 W	91.2	0.046	18.58
	250 W	110.5	0.055	22.50
250 W	**90 % Amplitude**	102.1	0.051	20.80
	80 % Amplitude	110.5	0.055	22.50
	70 % Amplitude	113.0	0.056	23.01
	60 % Amplitude	118.0	0.059	24.04
	50 % Amplitude	103.8	0.052	21.14
	40 % Amplitude	107.9	0.054	21.99
60 % Amplitude	**200 W**	96.2	0.048	19.60
	225 W	109.6	0.055	22.33
	250 W	118.0	0.059	24.04
	275 W	107.1	0.054	21.82

Table A.2. Results of ultrasonicator calibration for 1000-mL solution volume.

1000-mL Solution Volume				
Power Output and Amplitude		**Ultrasonic Power (Watt)**	**Power Density (Watt/mL)**	**Power Intensity (W/cm^2)**
80 % Amplitude	**100 W**	47.3	0.047	9.63
	150 W	76.1	0.076	15.51
	200 W	99.2	0.099	20.20
	250 W	113.8	0.114	23.18
250 W	**90 % Amplitude**	112.5	0.113	22.93
	80 % Amplitude	113.8	0.114	23.18
	70 % Amplitude	90.8	0.091	18.50
	60 % Amplitude	79.1	0.079	16.11
	40 % Amplitude	59.0	0.059	12.02
	20 % Amplitude	36.8	0.037	7.50
80 % Amplitude	**200 W**	99.2	0.099	20.20
	225 W	113.8	0.114	23.18
	250 W	113.8	0.114	23.18
	275 W	120.1	0.120	24.46

Table A.3. Results of ultrasonicator calibration for 500-mL solution volume.

500-mL Solution Volume				
Power Output and Amplitude		**Ultrasonic Power (Watt)**	**Power Density (Watt/mL)**	**Power Intensity (W/cm^2)**
80 % Amplitude	**100 W**	49.6	0.099	10.10
	150 W	81.2	0.162	16.54
	200 W	103.6	0.207	21.10
	250 W	110.7	0.221	22.55
250 W	**90 % Amplitude**	111.3	0.223	22.67
	80 % Amplitude	110.7	0.221	22.55
	70 % Amplitude	114.0	0.228	23.23
	60 % Amplitude	112.5	0.225	22.93
	50 % Amplitude	109.4	0.219	22.29
	40 % Amplitude	112.1	0.224	22.84
70 % Amplitude	**200 W**	101.7	0.203	20.71
	225 W	111.3	0.223	22.67
	250 W	114.0	0.228	23.23
	275 W	111.3	0.223	22.67

APPENDIX B

The Calibration Curves for COD Measurements

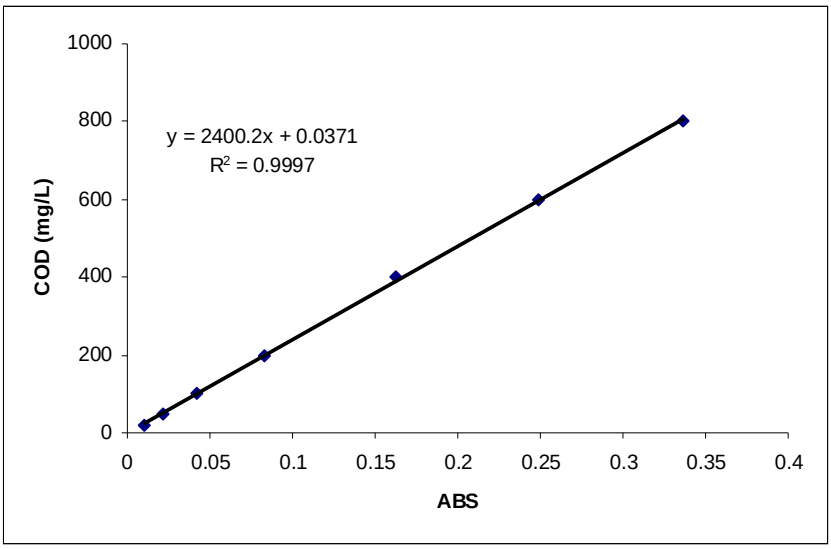


Figure B.1. Calibration curve 1 for COD.

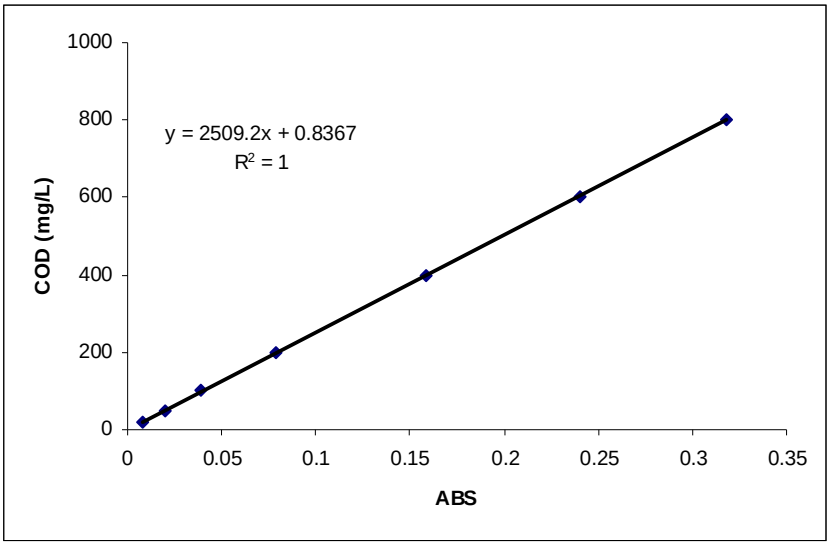


Figure B.2. Calibration curve 2 for COD.

APPENDIX C

Settling Velocity Variations

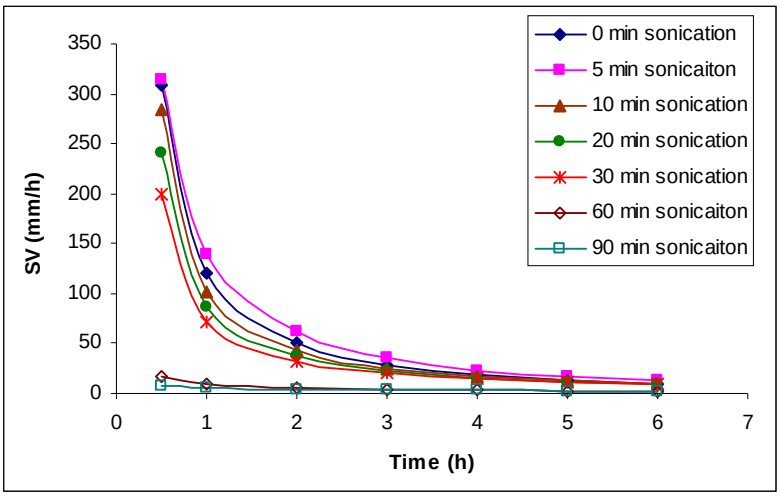


Figure C.1. Variation of sludge settling velocity with sonication time at power density of 0.059 W/mL.

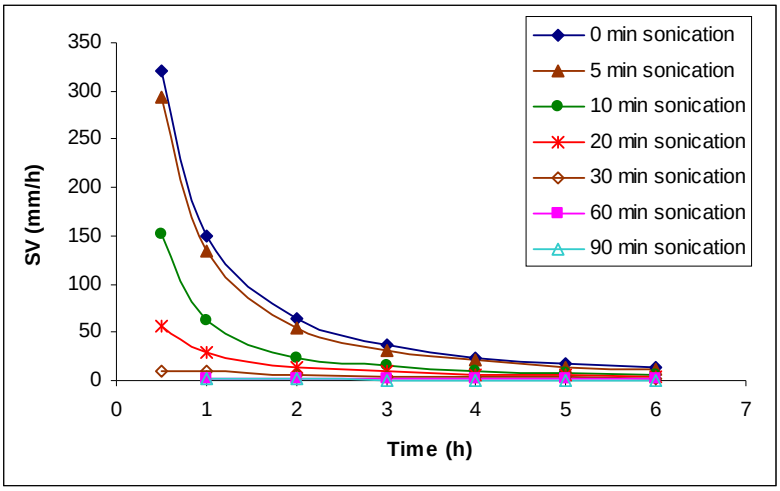


Figure C.2. Variation of sludge settling velocity with sonication time at power density of 0.114 W/mL.

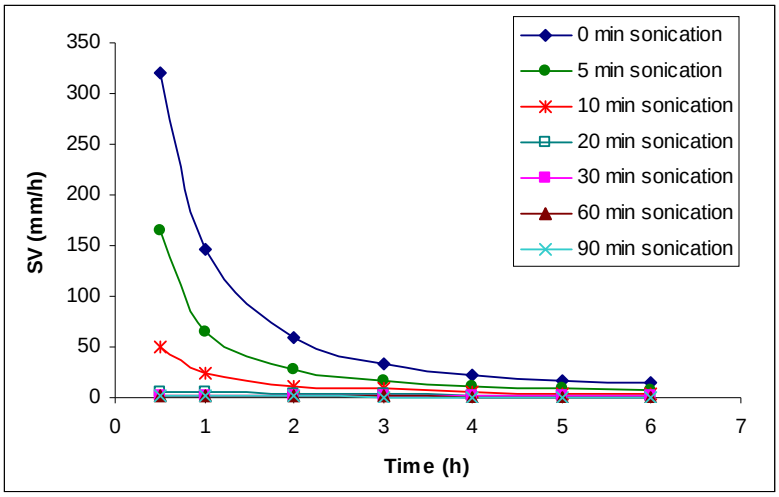


Figure C.3. Variation of sludge settling velocity with sonication time at power density of 0.228 W/mL.