CONTENTS

Figure list

Table list

Abbreviation

Abbr.	Description	Unit
BNR	Biological nutrient removal	--
COD	Chemical oxygen demand	mg/L
DO	Dissolved oxygen	mg/L
E^0	Standard ORP for the given oxidation reduction process	mV
F	Faraday constant	--
K_N	Nitrification rate	mg-NH_4^+-N/L-hr
K_{DN}	Denitrification rate	mg-NO_3^--N/L-hr
MDL	Method detection limit	mg/L
MLSS	Mixed liquid suspended solids	mg/L
ND	Not detectable	--
ORP	Oxidation-Reduction Potential	mV
R	Gas constant	9.314 $Jmol^{-1}K^{-1}$
SBR	Sequencing batch reactor	--
SBBR	Sequencing batch biofilm reactor	--
SEM	Scanning Electron Microscope	--
WAS	Waste activated sludge	--
E_{SND}	SND efficiency	%
DOE	Design of experiment	--
F/M		Kg COD/ kg MLSS- day
C/N	Carbon nitrogen rate	--

CHAPTER 1 INTRODUCTION

1.1 Background Information

In our countrymore than280,000 tonsof wasted activated sludge (WAS) was produced daily from secondary wastewater treatment plants (Huang *et al.*, 2005). The price of disposal WAS were hiked to US$80-150/ton recently, due to the lack of dumping land and might be doubled in the coming two tothree years. Unfortunately, the huge amount of domestic sludge was still booming with the rapid expansion of municipal wastewater treatment plants. To find out an applicable method to reduce the WAS is the urgent issues for the local governments in Taiwan.

In the other hand, the main composition in domestic wastewater contains carbon, nitrogen and phosphorus compounds. When the excess discharge of wastewater treatment plant, the effluent nitrogen and phosphate compounds will cause eutrophication (Blaney*et al.*, 2007). Besides eutrophication, the high content of ammonia nitrogen will cause toxic problems to aquatic organisms. To reduce the toxicity of ammonia can be used to remove both organic and nitrogen at one unit, the biological nutrient removal (BNR). After microbial metabolism, the system usually produce large amount of the WAS,which can be reused as immobilized pellets to allow biofilm growth,due to contains of inorganic and trace elements.

Some studies apply high-temperature sinter to recycle the wasted activated sludge to rebuild useful materials such as tiles and lightweight aggregate (Wang, *et al*., 2009). However, the wasted lightweight aggregate is difficult to disposal and can cause secondary pollution. Apply the calcination unit to rebuild the wasted sludge under a lower temperature to save energy and obtain prorous mediawith less hardness that could be easier for disposal and would have less impact to the natural environment (Su, 2008). Therefore, this study had attempted to investigate a way to recycle the WAS and upgrade the nutrient removal efficiency of the convention treatment plants.

The design of experiment (DOE) is method to conduct minimal tests to find out the critical elements that affect the results and can reduce the cost and time of trial and error (Ivanova & Malone, 1999; May *et al*., 1991; Silva *et al*., 2001). Hence, this approach had been used to gain an optimal formula to assemble the porous WAS pellets with the characteristics of sufficient porous and strength to reuse as immobilized material.

The application of the immobilized media could increase the removal efficiencies and improve sludge quality in theSBR system (Sirianuntapiboon*et al*., 2005). Traditionally, the nitrification and denitrification biological system used to remove the nitrogenous compounds in wastewater will generatea lot of sludge (Sirianuntapiboon*et al*., 2000; Keller *et al*., 1997). The simultaneous nitrification and denitrification (SND) removal mechanisms will taking place in the SBBR system with the rebuilt WAS pellets because it contains various kinds of anaerobic and aerobic biomass, which can decompose both carbon and nitrogen simultaneously in a single biological system (Jun *et al*., 2003).

In order to upgrade the removal mechanisms, the reused pellets were put into a sequencing batch reactor (SBR) and the pellets were immobilized the microorganism, deep pores and contain rich inorganic elements can attach biofilm (becomes SBBR) to growth and enhance the nutrient removal efficiency. This study add reused WAS porous carrier to a SBBR system, which can increase the biomass in reactor and conduct simultaneous nitrification and denitrification (SND) effectively. The excess trace inorganic nutrient added to the reused WAS pellets can also provide extra nutrients for the biofilm attached on the surface of the immobilized cells. The proposed rebuilt pellets contain 50% dry sludge and can significantly reduce the amount of disposal WAS.

1.2 Objective of Research

This study utilized the WASand other chemical additions to make porous pellets (with and without nutrients) then reuse them in the SBBR system to remove nutrient from municipalwastewaters. This study had attempted to apply the pellets to increase the biological nutrient removal (BNR) efficiency.

The objectives of this study included:

1. To find out the optimal formula of rebuild the WAS pellets and nutrients additive.
2. Apply the pellets in SBBR system and accomplish SND.
3. Compare treatment performances for the SBBR system using two different pellets (with and without nutrients).
4. To develop a model in SBBR/ SND system.

CHAPTER 2 LITERATURE REVIEW

2.1 Characteristic of Wasted Activated Sludge (WAS)

2.1.1 Sources and Types of Wasted Activated Sludge (WAS)

In order to achieve the pollutants removal, the microbe uptake of pollutants in wastewater as a nutrient for cell growth and cause the microbe proliferation, excessive biomass will be death and discharge become the WAS. The WAS was divided into three types according to the treatment wastewater characteristic: domestic sludge, animal husbandry sludge and industrial sludge.

The domestic sludge was primary from the municipal wastewater treatment plant. In recent years, due to the industrial and commercial development, population to the urban concentration and the quality of life was improved to induce the rapid growth of wastewater output, and that is increasing very fast. The municipal wasted sludge is an inevitable by-product of the treatment process of wastewater, following the

Ouyang*et al.* (1998) research, the wasted sludge output of up to 14,600 million tons per year in Taiwan. The animal husbandry sludge was produced from the animal excreta after the biological treatment, and the purpose of this type of the sludge was compost. Most of industrial sludge from food processing, paper manufacturing, chemicals, plastics and other raw materials industry and electroplating industry wastewater treatment plant. The characteristic of wasted sludge was discrepancy from different industry. There are many high concentrations of pollutants, toxic substances or heavy metals in the industrial wasted sludge. Therefore, it was difficult to improve the treatment of industrial sludge and industrial sludge reduced substantially recycled the viability of organic matter.

In the three types of wasted sludge, domestic wasted sludge characteristic with more simple and high content of organic carbon, and therefore most suitable for domestic wasted sludge treatment and recovery resources of the organic matter.

2.1.2 Composition of Wasted Activated Sludge (WAS)

In Lin's (2006) study indicated much kind of sludge contents had more 50% ash. The high content of ash could reused and low heavy metal was general industrial waste, which was positive help for the material sintering. The sludge characteristics of the wastewater treatment were different by raw water quality, coagulant type and operation mode. The type of pressure filter was the most efficient of the mechanical dewatering; the water content could be under 60%. The water content of the other types of dewatering was higher 80% - 85%, which had to additional drying equipment to reduce the water content. That was poor efficiency and energy consumption. In the study says the sludge cake dewater by dewatering type of pressure filter had lowest water content, the ash content up to 50% or more. The main components include suspended solids, Fe-Al hydroxide, coagulant, water and small amount of organic matter. The chemical composition based mainly SiO_2, SiO_2 in the raw water comes from mineral sands, while Al_2O_3, Fe_2O_3 mainly from clay minerals and aluminum coagulation dosing system or ferric coagulant, other chemical substances including K_2O , MgO, Na_2O, and other types of sulfur salts.

Taiwan sewer sludge composition analysis shows that under a fixed water source, its chemical composition changes of sludge are not large (Chen, 2006).Therefore, the WAS recycling products, the stability of product quality should be good.

2.1.3 Wasted Activated Sludge Recycling

In recent years, there are many wastewater treatment plants designing and constructing in Taiwan area. Those plants are belonging to secondary treatment process, i.e., which treating wastewater by using activated sludge process mainly. The secondary treatment process usually generates excess amount of WAS (Wojciechowska, 2005). Traditionally, there are two major WAS disposal processes i.e., landfill and incineration in Europe and Japan. Landfill, needs large area of land to carry it out, which is not suitable to the overcrowded Taiwan. The incineration can significantly reduce the volume of WAS, but it needs the supplementary fuel to make sure the WAS can burn completely (Dewil*et al.*, 2005). Therefore, the optimal WAS treatment strategy are minimization and reutilization. Some investigations related to the WAS minimization and reutilization were shown in Table 2-1. This study attempts to use the WAS as the main material to bake porous media and reuse them in a biological process to reduce nutrients in wastewater. The reused porous media in bioreactor can enhance the wastewater removal efficiency by increasing the biomass in a sequencing biofilm batch reactor (SBBR).

Table 2-1 Application of wasted activated sludge (WAS)

The aspect of reutilization	References
Biofilm carriers	This study, 2010
Immobilized pellets,	Su, 2008
Air diffuser	
Lightweight aggregate	Cheeseman and Virdi, 2005
Lightweight aggregate	Wang *et al.*, 2009
Micro-media	Kim *et al.*, 2003
Growing-media	Lourdes *et al.*, 2005
Cement	Monzó*et al.*, 2003
Adsorbent for dye	Gulnaz*et al.*, 2006
Ceramic materials	Cheeseman*et al.*, 2003
Immobilized cells	Chen, 2005
Immobilized pellets	Chung, 2007

2.2 The WAS sintering

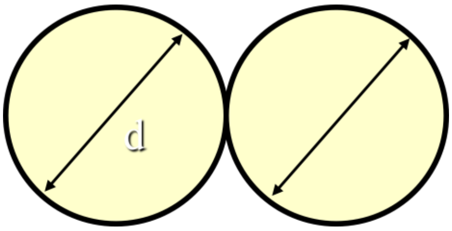
The sintering reaction was heated under the melting point of the material, and rearrange the internal structure of materials up to high intensity, high-density sintered body (Li*et al.*, 2009).

1. Solid-state sintering: applied surface diffusion, lattice diffusion, boundary diffusion or solid material with the surrounding atmosphere was reaction of the mass transfer.
2. Sintering by viscous flow: Sintering was caused by the flow of non-crystalline material, viscous flow sintering mechanism of sintering as the main environment in silicate (skrifivars, 1994).
3. Liquid phase sintering: applied the melting and solid precipitation to increasing particle size and density.

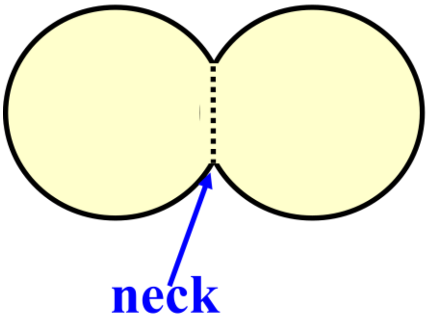
The factors of affect sintering were sintering temperature, the time of heating, chemical composition, forming pressure and size distribution.

German was built the sintering model. The theory was divided into four steps sintering in Figure 2-1. The reaction was started the point contact, and then the neck growth. The new grain boundary was formed between the particles. Two particles was be a single particle (German, 1996).

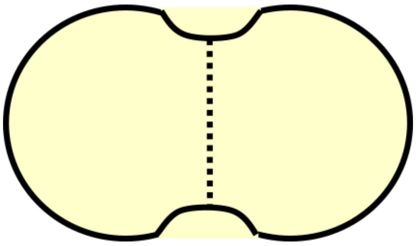
1. Initial point contact



2. Early stage neck growth



3. Last stage neck growth



4. Terminal condition fully coalesced spheres

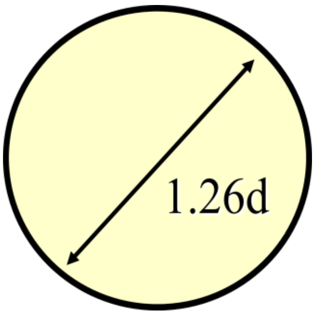


Figure 2-1The granule agglutination response schematic drawing (German, 1996).

Material without viscosity and gas production conditions, the material had to use additives to make the above two points occurs. But the heated gases may not be enough, not a swelling effect. Table 2-2 for the various types of chemical composition and temperature of escaping gases list.

Table 2-2 Various types and temperatures of overflowed gases with all kinds of chemical compounds.

Chemical equation	Reaction temperature (^oC)
$FeS_2+O_2 \rightarrow FeS + SO_2\uparrow$	350 - 450
$4FeS + 7O_2 \rightarrow 2Fe_2O_3 + 4SO_2\uparrow$	500 - 800
$Fe_2(SO_4)_3 \rightarrow Fe_2O_3 + 3SO_3\uparrow$	560 - 775
$MgCO_3 \rightarrow MgO + CO_2\uparrow$	400 - 900
$Na_2CO_3 \rightarrow Na_2O + CO_2\uparrow$	>400
$CaCO_3 \rightarrow CaO+CO_2\uparrow$	600 - 1,050
$CaSO_4 \rightarrow CaO + SO_3\uparrow$	1,250 - 1,300
$6Fe_2O_3 \rightarrow 4Fe_3O_4 + O_2\uparrow$	1,000 - 1,550

2.3 Mechanism of immobilized system

2.3.1 Formation of attached biofilm on the bio-carriers

Traditionally, the porous materials (bio-carriers) were applied to immobilized bioreactor system, such as trickling filters, fluidized bed reactor and rotating biological contactors (RBCs). In this study, the rebuilt WAS pellets were utilized as bio-carriers in the activated sludge reactor. The immobilized system in the bioreactor is composed of support material and attached growth biomass. The formation of immobilized system is complex (Figure 2-2) as a result of a combination of factors, such as bacterial growth, substrate consumption, attachment, external-internal mass transfer of substrates and products, cell death, shear loss (biofilm loss because of erosion), sloughing (fragments disrupting from the biofilm), structure of the support material, competition between bacterial species, and effects of predators (Wijffels. and Tramper., 1995).

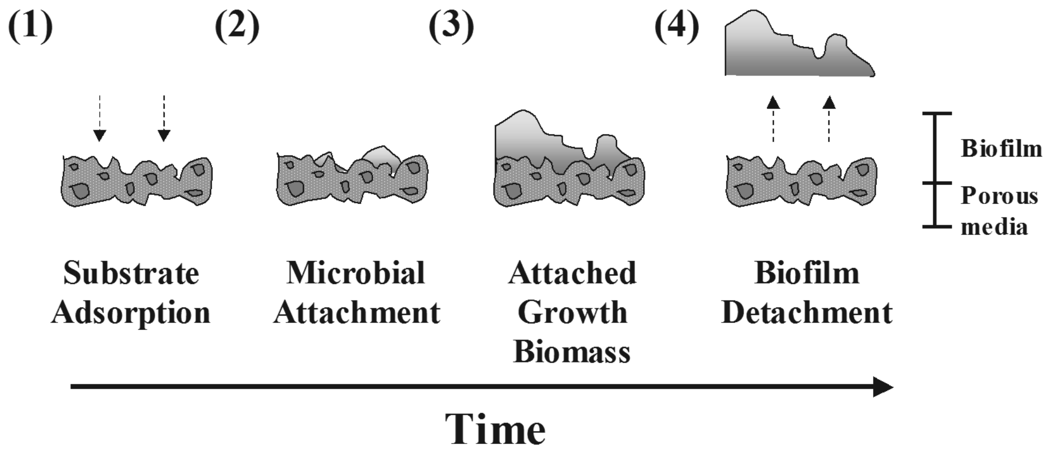


Figure 2-2 Schematic presentation of the formation of a biofilm.

The biofilm formation process included as follows:

(1) Substrate adsorption: Substrate adsorbed into the support material.

(2) Microbial attachment: Microbial in the bulk were attached onto the surface of support material (bio-carriers).

(3) Attached growth Biomass: Substrate utilized with the attached growth biomass. The biofilm thickness can be increased and become matured gradually.

(4) Biofilm detachment: When the biofilm become matured for a long time, the biofilm was peeled out owing to the inner starvation zone formed.

Other conditions such as concentration of substrate, temperature, pH, turbulence and diffusion rate all will influence the biofilm formation (Wijffels. and Tramper., 1995).

2.3.2 The biofilm system

In wastewater treatment process, the biological process is widely be used to decompose organic substances. Among all biological processes, the activated sludge process has the disadvantage of producing excess waste sludge cake and high sludge volume index (Sirianuntapiboon*et al.*, 2005). Due to the difficult settlement of sludge, several biofilm processes were proposed to overcome the problems. Besides, the biofilm can bear a great potential for the simultaneous remove of organic carbon and nutrients, such as nitrogen and phosphate, in wastewater treatment (Gieseke*et al.*, 2002). The biofilm system can reduce the excess bio-sludge in the reactor (Sirianuntapiboon*et al.*, 2005). In this study, the pellets used as carrier for bacteria growth were made from recycled wasted activated sludge. According to literature, the bacteria grow on carrier have five processes (Molin and Tim, 2003):

(1) Adsorption: The substance adsorbed on the carrier.

(2) Transportation: The bacteria approach to the carrier in the aquatic

condition.

(3) Contact: The bacteria adsorbed on the carrier.

(4) Growth: Bacteria formed the biofilm on the carrier and treated the nutrient in the substance.

(5) Detachment: When the biofilm getting too thick, the substance cannot transmit into the deepness of biofilm. The bacteria in the deep biofilm start autolysis in endogenous phase.

2.4 Biological nutrient removal (BNR)

The microbial decomposed and oxidized organic pollutants of wastewater affected by the following conditions; (1) maintain the F/M in the aeration tank; (2) maintain the DO; (3) separate from the sludge sedimentation tank should be concentrated higher than activated sludge concentration in aeration tank; (4) removal SS and the proliferation of microbial decomposition of BOD, treated water should be discharged to maintain a sludge retention time.

2.4.1 Nitrification

Normally, the nitrogen compound removal can be divided into two processes, nitrification and denitrification (Ahn, 2006). Nitrification is converting ammonia nitrogen to nitrite or nitrate nitrogen. The reaction is shown as:

$$NH_4^+ + \frac{3}{2}O_2 \rightarrow NO_2^- + 2H^+ + H_2O \text{..} Eq.(2-1)$$

and

$$2NO_2^- + O_2 \rightarrow 2NO_3^- \text{..} Eq.(2-2)$$

The overall nitrification combined *Eq. (2-1)* and *Eq. (2-2)* and shown as:

$$NH_4^+ + 2O_2 \rightarrow NO_3^- + 2H^+ + H_2O \text{..........} Eq.(2-3)$$

Traditionally, nitrification require oxygen as electron acceptor and under aerobic condition with the autotrophic bacteria, e.g. *Nitrosomonas* and *Nitrobacter* (Siripong and Rittmann, 2007; Michaud *et al.*, 2006). There are some parameters will inhibit nitrification, i.e., pH, DO, temperature, etc. When nitrification proceeded, the alkalinity was consumed by hydrogen ion released. The optimal pH values are between 7 to 8 (Gou*et al.*, 2009). The sludge retention time (SRT) is also an important parameter for nitrification. Because the nitrifiers growth rate are slow, it needs long SRT in nitrification for growth of nitrifiers (Juliatuti*et al.*, 2003).

2.4.2Denitrification

Generally, ammonia nitrogen in aerobic condition is converted to nitrite and nitrate nitrogen by nitrification process. Denitrification can convert those two compounds into nitrogen gas. But, the contamination of groundwater and drinking water sources with nitrate (NO_3^-) and nitrite

(NO_2^-) is a serious problemin recent years because $NO_3^-$and $NO_2^-$are responsibleformethemoglobinemia (Blue baby syndrome) ininfantsandmay cause their carcinogenic diseases.

$$NO_3^- \rightarrow NO_2^- \rightarrow N_2O_{(g)} \rightarrow N_2 \ldots\ldots\ldots Eq.(2-4)$$

2.4.3 Simultaneous nitrification and denitrification (SND)

Many studies showed that the nitrification and denitrification can occur in the same reactor at the same time, which is defined as simultaneous nitrification and denitrification (SND) (Chiu *et al.*, 2007; Holakoo*et al.*, 2007; Zhang *et al.*, 2007; Weissenbacher*et al.*, 2007). The SND process can divide into two categories; pure-culture and mixed-culture systems. Pure-culture system implies the SND reactor has only one bacterium which can achieve nitrification and denitrification consecutively. Some bacteria can accomplish this process like *Nitrosomonaseuropaea* and *Paracoccusdenitrificans* (Stüven and Bock, 2001). Although the pure-culture have a good performance in nitrogen compounds removal, but pure-culture is strictly limited to use in the small-scale and is difficult to apply in the field. Therefore, mixed-culture system is focused on improving the field SND treatment process. Two parameters: dissolved oxygen and carbon source, need to be considered in mixed-culture system. The dissolved oxygen limitation is 0.5 to 2.0 mg/L for SND process (Chu *et al.*, 2006; Chiu *et al.*, 2007; Holakoo*et al.*, 2007). Due to the rate of nitrification is much faster than that of denitrification, therefore, a balance controlled reaction system will insure the consecutively processes work smoothly. Low DO condition can decrease nitrification reaction rate and inhibit it close to that of denitrification process. Nitrification in SND can consume 8.64 g-$CaCO_3$/g-NH_4^+-N alkalinity in the reactor; while the denitrification reaction will produce 3.6 g-$CaCO_3$/g-NO_3^--N alkalinity and maintain pH in the solution. Therefore, the biological system will not require extra alkalinity to adjust pH value during the denitrification stage. The sufficient carbon sources, i.e., more than C/N 10 will allow denitrification proceed well (Guo*et al.*, 2005; Chiu *et al.*, 2007).

The immobilized system compared with traditional activated sludge system has main advantages as follows (Loukidou and Zouboulis., 2001):

1. Higher biomass concentrations in the aeration tank, which correspond to lower wastage of biomass；
2. Elimination of long sludge-settling periods；

3. Co-existence of aerobic and anoxic metabolic (simultaneous nitrification and denitrificaion)activity within the same biomass ecosystem.
4. Up-grading of existing wastewater treatment plants at a minimum cost.
5. Lower sensitivity to toxicity effects, as well as to other adverse environmental conditions.

The main disadvantage of this system is however, the need for operation at higher concentrations of dissolved oxygen (above DO of 2-3 mg/L) in order to maintain the biofilm activity and high nitrification rats (Holman *et al.*, 2005; Guo*et al.*, 2009).

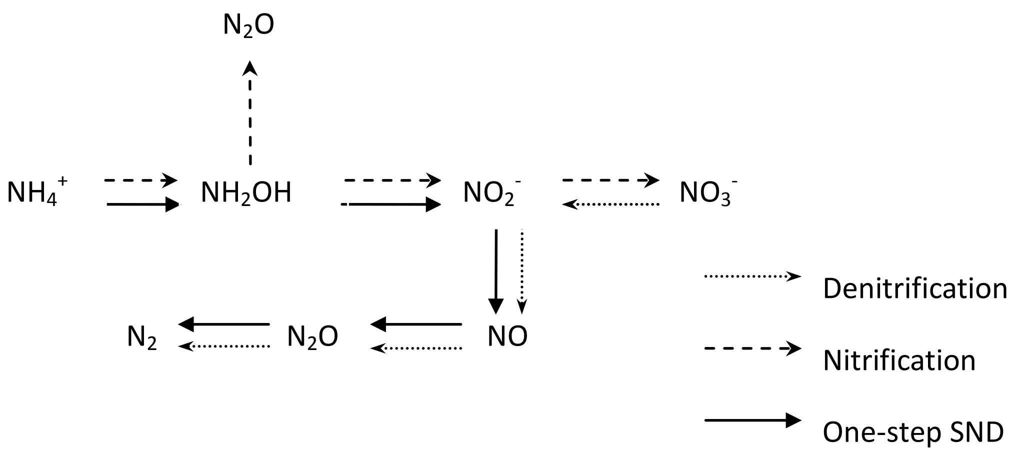


Figure 2-3Flow chart of conventional nitrification/denitrification and one-step SND via nitrite pathway. (Wrage*et al.*, 2001)

2.4 Sequencing batch biofilm reactor (SBBR)

The sequencing batch reactor (SBR) is a process for wastewater treatment system that all the processes are conducted in the same reactor by changing different operating patterns. The conventional operating steps in sequence listed as followed: fill, react, settle and decant (Kishida*et al.*, 2007). The SBR systems have proven advantages to the conventional biological treatment system for both domestic and industrial wastewaters (He *et al.*, 2007). Antileo*et al.* (2006) indicated that the continuous flow system for nitrification is difficult to maintain nitrite oxidizing bacteria but not for the SBR. Li *et al.* (2003) indicated the SBBR can govern the biomass level and enhance removal efficiency by biofilm. While the SBBR system can control the biofilm thickness by adjusting the filling and decanting stages. Mohan *et al.* (2005) mentioned the SBR system can enhance phosphorus removal due to phosphate accumulation during the anaerobic process and utilization in the subsequent aerobic process. Table 2-3 showed the treatment performance by using the SBR and the SBBR systems from references. There are different kinds of wastewater treated by using SBR and SBBR systems provide a satisfactory effluent water quality.

Table 2-3 The treatment performance by using SBR and SBBR systems from references.

Reactor	Wastewater source	Removal efficiency	Authors
SBBR	Landfill leachate	COD : 81% BOD_5: 91%	Loukidou and Zouboulis, 2001
SBBR	Synthetic (domestic)	COD : 90%	Li *et al.*, 2003
SBBR	Synthetic (industrial)	COD :>90%	Hu *et al.*, 2005
SBR	Industrial	BOD_5: 92.2%	Mohan *et al.*, 2005
SBBR	Milk Industrial	COD :89.3-98.6% BOD_5:83.0-97.7% TKN :59.4-87.0%	Sirianuntapiboon*et al.*, 2005
SBBR	Synthetic (domestic)	TN :100-53%	Lemaire*et al.*, 2006
SBBR	Synthetic (domestic)	COD:> 99% NH_4^+-N:> 98% PO_4^{3+}-P:> 90%	Kishida*et al.*, 2006
SBR (SND)	Synthetic (domestic)	COD: 79% N: 88% NH_3-N: 75%	Holman *et al.*, 2009

2.5 Nernst Equation

The biological nitrogen removal, i.e., nitrification and denitrification, processes is an oxidation-reduction reactions during which electron are transferred from the reducing agent to oxidizing agent until the reaction reaches equilibrium. The electrochemical potential between the reducing and the oxidizing agents is known as the oxidation-reduction potential (ORP), which measures the net potential of the system (Weissenbacher*et al.*, 2007). The reactor can measure nitrification and denitrfication processes the changing of ORP value from an on-line system (Li and Irvin, 2007). The measured ORP value in biological system is correlated to the concentration changing of the reductive and the oxidative species represented in a Nernst equation (Chiang *et al.*, 2006). The general Nernst equation is shown in Eq. (2-5):

$$E = E^0 + (RT / nF)\ln([Oxi]/[Red]) \qquad Eq.(2-5)$$

E =ORP (mV)

E^0=standard ORP for the given oxidation-reduction process (mV)

R= gas constant (8.314Jmol^{-1}K^{-1})

T=absolute temperature (K)

N=number of electrons transferred in the reaction

F= Faraday constant (96,500$Cmol^{-1}$)

[Oxi] = Concentration of oxidizing agent

[Red] = Concentration of reducing agent

Chang *et al.* (2004) used on-line measured data (e.g. ORP) and nitrogen compounds (e.g. ammonia, nitrite and nitrate) in the reactor to substitute terms in Nernst equation. Deduced Nernst equation can be used to understand the nitrification and denitrificaion conditions in reactior. ORP value of any degree conversion of reactants to products in reactor can be precisely calculated. Thus, on-line control strategy can effectively utilize in a SBR system.

2.6 Design of Experiments (DOE)

MINITAB® 14 is a statistical software was used to analysis the optimal analysis of operation factors (Gunawan*et al.*, 2005). Choose the major factors and after the test, a response surface and contour plot was obtained, which will display the optimal operation zone for the following study.

In order to correlate the dependent variables and independent variables with the minimum possible number of experiments, a central composite design for two factors has been used. The total number of experiments (N) required for two independent variables was determined by *Eq. (3-6)*(Lu *et al.*, 2008).

$$N = 2^K + 2K + n_c \quad \text{……………………………………………………} \textit{Eq. (3-6)}$$

Where K represents the number of independent variables, in the present study, K=2 and n_c is the center point. The data were analyzed using MINITAB® 14 statistical software. Three replications of center point were selected for center composition design and totally, 11 experiments were predicted by the software.

CHAPTER 3　MATERIAL AND METHODS

3.1 Experimental design and flow chart

The purpose of this study is to reduce the excess wasted sludge, to utilize the sinter, to recycle the wasted sludge, and to apply the recycled sludgein the wastewater treatment processes.

The flow chart of this study could be divided into three parts (Figure 3-1).　Sequence (A): Rebuilt the WAS pellets as a biofilm carrier based upon the DOE method and applied in the activated sludge reactor. Sequence (B): Develop the SBBR system to conduct SND reactor. Sequence (C): Establish the Nernst equation with the SND process.

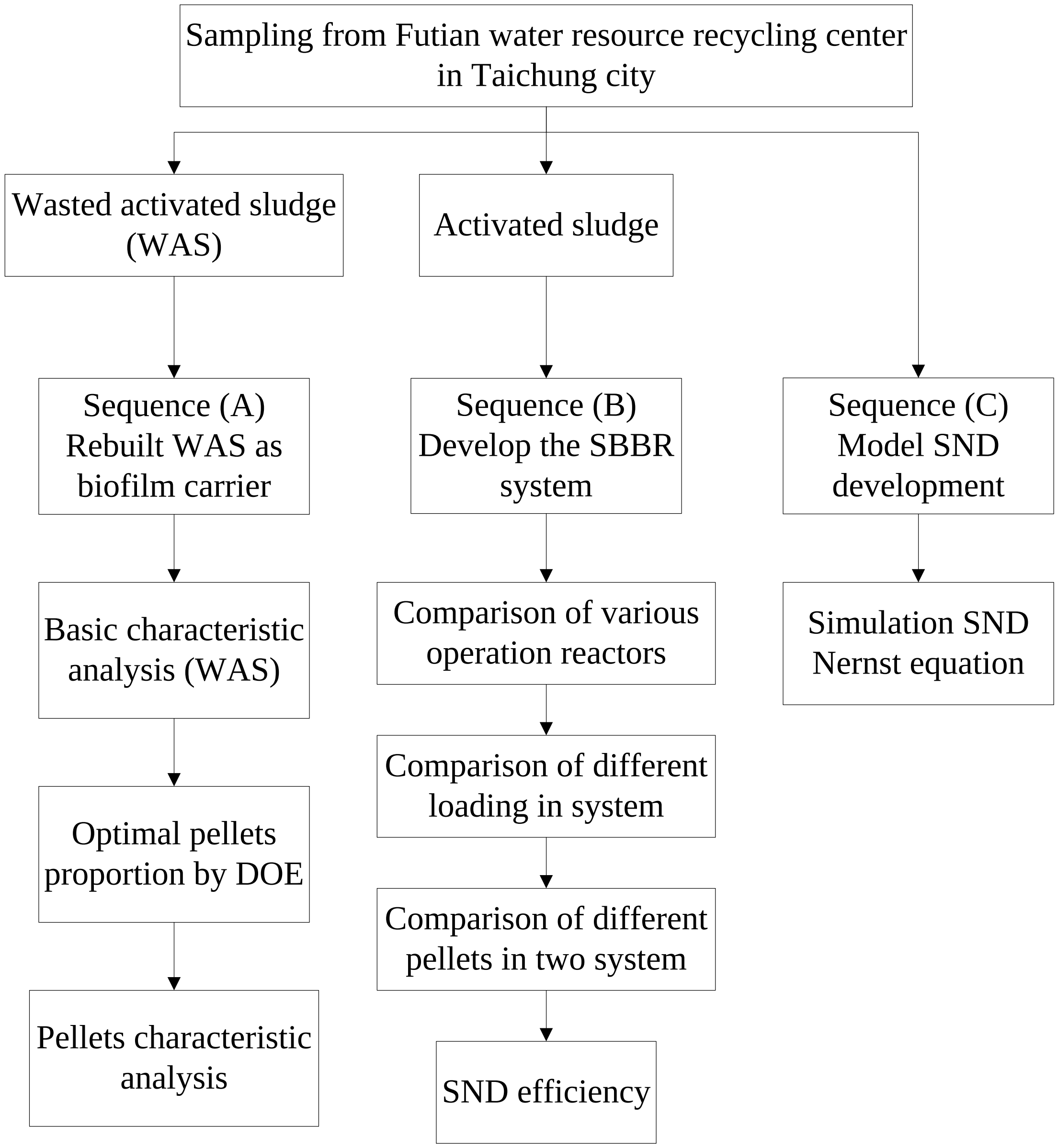


Figure 3-1 The flow chart of this study included three parts. Part (A): Rebuilt the WAS pellets as a biofilm carrier. Part (B): Develop the SBBR system. Part (C): Establish the Nernst equation with the SND process.

3.2 Rebuilt WAS as biofilm carrier

3.2.1 The procedure of manufacture the WAS pellets

The process of rebuild the WAS to the biofilm carrier were to mix the WAS, laterite and chemical additive and leave it on the 105°C oven drying for three days. The laterite was help the pellets to shaping and the chemical additive is to the generate foam agent and can increase the hardness.

The dried WAS and laterite was milled by ball mill machine and then sifts to the grind size of the powder close to 10 μm. The smaller the powder size, the smaller space between particles could be obtained, that was making the structure more closely to increase the rigidness.

The mix formula of WAS and laterite was design by MINITAB®, DOE method, which use test every design proportion then try and error to found out the optimal formula.

Thereafter, the pellets were baked in an oven under the temperature raised from 200 °C to the final temperature of 950 °C. The raising programs are: baked for an hour for raising every one hundred centigrade degree and finally baked at 950 ^{o}C for three hours. The overall time to bake the pellets is 7.6 hours (430min). After baking, those pellets were cooled down to the room temperature (25 ^{o}C). After many trial tests, this study obtained the optimal formula ratio of WAS: laterite: chemical additive is 6: 7: 1. The pellet's WAS reuse ratio was 50% at least and their diameters were between 12-14 mm.

3.2.2 The addition of nutrients

The nutrients, i.e. nitrogen, phosphorous and potassium, were the trace elements which can be used by microbial. Many compounds, monopotassium phosphate, was selected by BOD test method and used to cultivate bacteria in the dilution water while potassium nitrate was usually added to fertilizers. In the process of pellets preparation, added 0.1M KH_2PO_4 and 0.1M KNO_3, and two compounds mixed together in order to replace the raw particles produced by adding water.

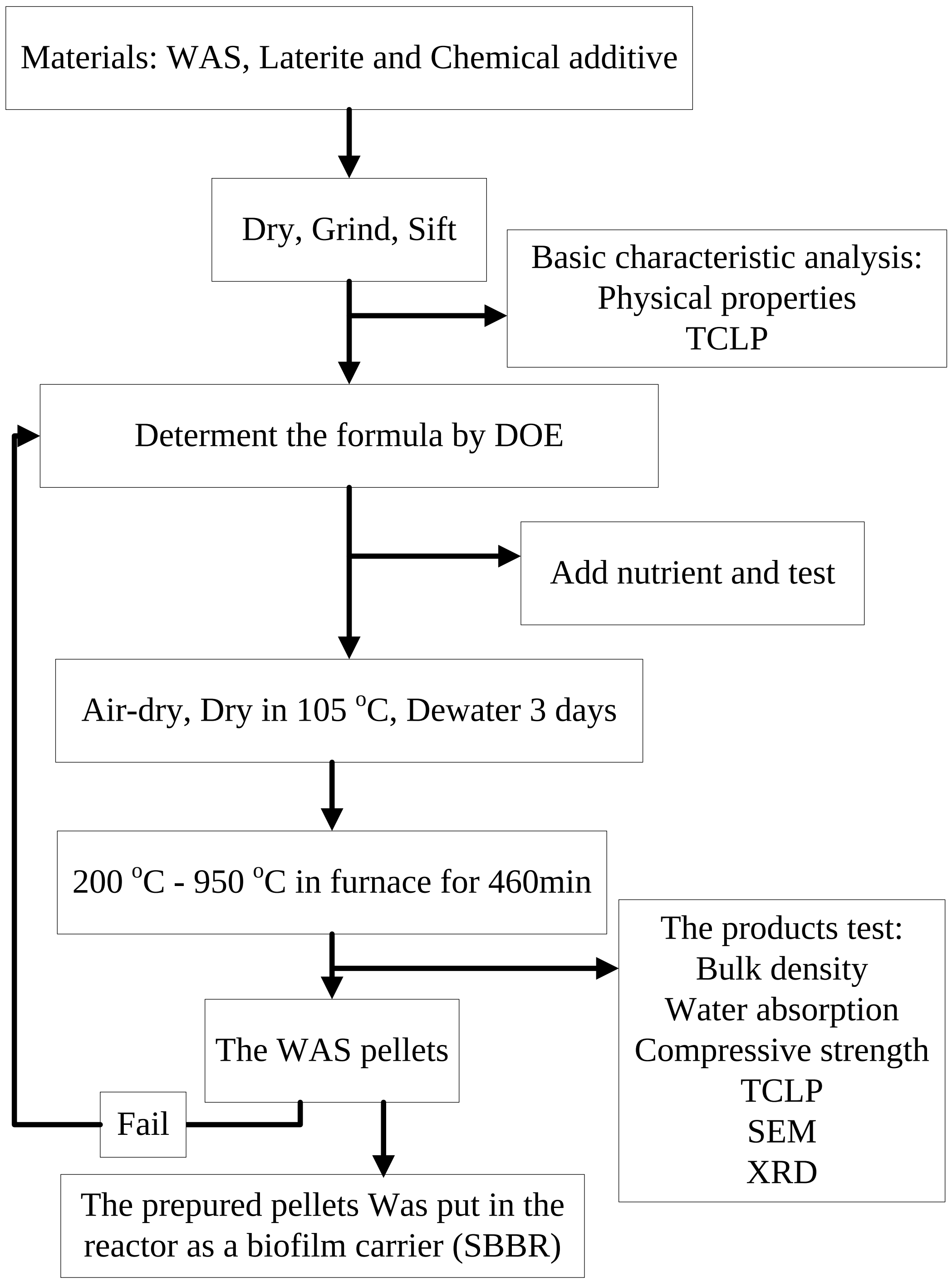


Figure 3-2 The flowchart showing a method of manufacture the biofilm carrier pellet.

3.3 The WAS sampling and basic characteristics analysis

The WAS was collected from Futian water resource recycling center in Taichung City. It treats 55,000 CMD domestic wastewater from downtown Taichung City and septic sewer treatment center of Taichung City (Chung, 2004) and produced 60,000 tons dry sludge cake per month. Currently the WAS is disposed by landfill. The WAS was dried at 105 ^{o}C for three days before conducting rebuild pellets process. The ash content is measured after baked under 800 ^{o}C for three hours (NIEA R205.01C). The formula of the moisture, ash and flammable contents in WAS are shown in *Eqs. (3-1)*, *(3-2)* and *(3-3)*

Moisture content = $(W_1-W_2) / W_1 \times 100\%$ ……..…..…...*Eq.(3-1)*

Ash content = $W_3 / W_1 \times 100\%$.................................*Eq.(3-2)*

Flammable content=100% - Moisture content % - Ash content % .*Eq.(3-3)*

W_1= weight of sample before burned in oven (25 ^{o}C)

W_2= weight of sample dried by 105 ^{o}C

W_3= weight of sample baked by 800 ^{o}C

3.3.1 Compressive strength

Strength of various materials often measured by the compressive strength, which testing method is adopted Chinese national standard (CNS1010 R3032). The sample was prepare to made into a 3cm x 3cm cubes and testing the value of uniaxial compressive stress.

3.3.2 Water absorption

In physical characteristics of the porous carrier, the water absorption is the reference density measurement (Cheeseman*et al.*, 2003). The water absorption testing was following the Chinese National Standard (CNS-487). The sample was immersed in 23°C water for 24 hours, and then removes out of the water. Dried the surface water of sample, measured the sample weight that was the weight of the water saturation.

water absorption (%) = $(W_s - W_d) / W_d \times 100\ \%$ …………...….*Eq. (3-4)*

W_s : the weight of dry sample (g)

W_d : the weight of dry surface and water saturation(g)

3.3.3 Bulk density

According the Archimedes principle, obtained after the sample volume, and then divided by the weight of sample.

Volume: V_S (cm^3) = $V_W - (W_b - W_a) / \rho_W$……………………….*Eq. (3-5)*

Bulk density: ρ_S (g · cm^{-3}) = W_S / V_S…………………………….*Eq. (3-6)*

W_S: Weight of dry sample (g)

W_a: Weight of graduated cylinder + W_S (g)

W_b: Weight of quantitatively to the 100ml water + W_a (g)

V_W: Volume of quantitatively water (cm^3)

ρ_S: Density of water (g · cm^{-3})

3.3.4 Specific external surface area

The easiest method for the determination of the specific external surface area, a_s, is the counting-weighing method. Here, a representative sample of dry particles is counted after the weighing (Andreasen, 1928). Using the particle density, the volume of the average spheres having the same volume, d_v, is calculated. The specific external surface area is related to d_v by this expression:

$$a_s = \frac{6}{\rho_p d_v} \quad \text{..} \textit{Eq. (3-7)}$$

a_s: Specific external surface area

ρ_p: Particle density

d_v: equivalent diameter from the same volume

3.3.5 Toxicity characteristics leaching procedure (TCLP)

Toxicity characteristic leaching procedure (TCLP) (NIEA R201.13C) is a soil sample extraction method for chemical analysis employed as an analytical method to simulate leaching through a landfill. The leachate is analyzed for substances appropriate to the protocol.

TCLP comprises four fundamental procedures:

- Sample preparation for leaching
- Sample leaching
- Preparation of leachate for analysis
- Leachate analysis

A concern has arisen in recent years regarding TCLP analysis in that the test is based on the assumption that the waste material will be buried in landfill along with organic material, however organic matter is rarely buried with other waste anymore (composting usually applies). In light of this issue other leachate techniques may be more appropriate.

In the TCLP procedure the pH of the sample material is first established, and then leached with an acetic acid / sodium hydroxide solution at a 1:20 mix of sample to solvent. The leachate solution is sealed in extraction vessel for general analytes, or possibly pressure sealed as in zero-headspace extractions (ZHE) for volatile organic compounds and tumbled for 18 hours to simulate an extended leaching time in the ground.

WAS from domestic wastewater plant may contain trace amounts of heavy metals, therefore, this study conducted the toxic characteristics leaching procedure (TCLP) test to identify the heavy metals from it. Five toxic heavy metals (Cd, Cr, Cu, Pb and Zn) were analyzed.

Table 3-1 The Regulation of heavy metal concentration of leaching.

Element	Regulation of TCLP from HM (mg/L)*
Cu	15
Pb	5
Cr	10
Zn	25
Cd	0.5

*Heavy metals regulation was obtained from Waste Material Cleanup, EPA, Taiwan

3.4 The sequencing batch biofilm reactor (SBBR) system

3.4.1 Experiment setup

(1) The rectangular biological reactor was made by acrylic fiber glass with the height of 45.0 cm and the inside depth, width and length of 25.0 cm and 25.0 cm, respectively. The effective working volume was 25 L.

(2) Mixer: Oriental Motor, Japan, operated at 150 rpm.

(3) Aeration pump: Serial No. 1030114, Medo Co., Japan. The maximum air flow rate is 12 L/min.

Two pilot-scale sequencing batch reactors (one SBBR and one SBBR-Nutrient) are shown in Figure 3-3, both systems were added the rebuilt pellets and nutrient pellets in reactors with the volumetric packing ratio of 15%, respectively.

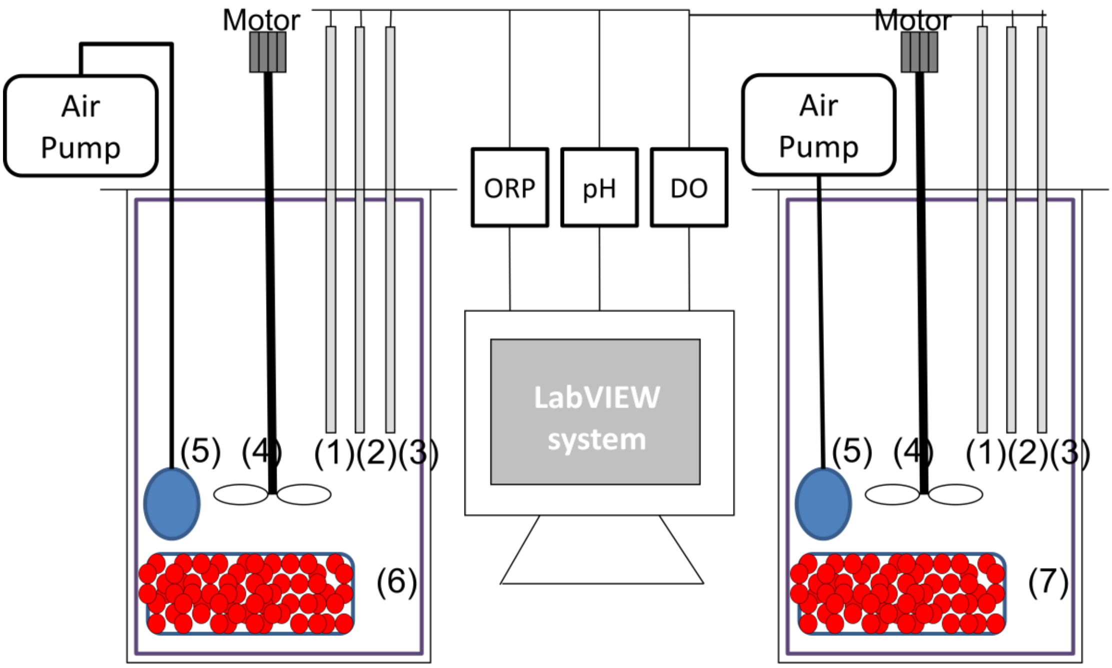


Figure 3-3 Schematic diagram of the system, (1) (2) (3) ORP, pH, DO electroed, (4) Mixer, (5) Air aeration diffuser, (6)Biofilm carrier (raw pellets, suspended), (7)Biofilm carrier (nutrient pellets, suspended).

3.4.2 The real-time monitor

(1) pH: pH meter (SUNTEX PC-310, Taiwan, R.O.C.) equipped with glass membrane sensor (Mettler-Toledo pH 114053134, Switzerland) is adjusted by using the pH 4.0 and pH 7.0 standard solutions.

(2) ORP: ORP meter (SUNTEX PC-310, Taiwan, R.O.C.) equipped with ORP probe (AgCl, Mettler-Toledo ORP 115053020,

Switzerland) is adjusted by using the zero-point (0 mV) and 220 mV standard solutions.

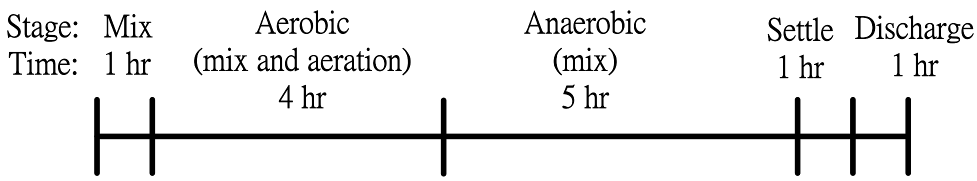
(3) DO: DO meter (SUNTEX DC-5100) equipped with DO electrode (WTW TriOxmatic 690, Germany) adjusted by using internal aero-correction process.

(4) Computerized monitoring system: computer (Microsoft OS Windows 2000); LabVIEW (Laboratory Virtual Instrument Engineering Workbench 5.1) monitor software and AD/DA card (AT-MIO-16E-10).

3.4.3 Experiment operation

This study conducted the nitrification and denitrification reactions by adjusting different levels of airflow in the systems. In order to achieve the SND reaction, the airflow was controlled under low level, i.e., low DO case with air 1.0 L/min, DO = 0.4-0.8 mg/L (Peng, 2009). The two systems were operated with a cycle time of 12 hours in an air conditioned room (with temperature under 25±2 ^{o}C). The operation cycle of each period in two systems are shown in Figure 3-4. In the cyclical operation stages of cultured the sludge is (a); the first stage, the reactors only mixed without aerated. While in the second stage, the reactors mixed and aerated, then in the third stage, the reactors only mixed. Finally, in the fourth stage, the reactors were settling without mixing and aeration, then, the supernatant was drawn. After cultured the sludge, the cyclical operation stages was only mixed and aeration without the anoxic stage shows in Figure 3-4 (b). The daily effluent samples were analyzed right after filtered by a 0.45 μm glass fiber membrane filter.

(a) The cyclical operations stage of cultured the sludge in two systems.



(b) The cyclical operation stage of SND process in two systems.

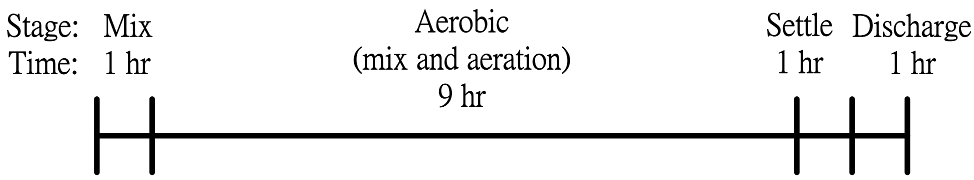


Figure 3-4 Operation cycle in (a) The operation stage of cultured sludge.

(b) The operation stage of SND process.

3.4.4 Composition of the synthetic wastewater

The formula of the stock synthetic wastewater is shown in Table 3-2.

The synthetic wastewater contains carbon source (glucose), nitrogen source (urea), phosphate buffer (KH_2PO_4) and trace nutrients. The general nutrients include acetic acid, glucose, urea, ammonium chloride and phosphate. The organic and inorganic nitrogen in the synthetic wastewater are urea and ammonium chloride. The milk powder in the formula contains protein, lactose, mineral and fat. The stock synthetic wastewater is maintained in a 4 ^{o}C refrigerator to avoid spoilage. The characteristics of the influent synthetic wastewater are shown in Table 3-3. The high nutrient contented synthetic wastewater with the average ratios of COD: NH_4^+-N: PO_4^{3-}-P (100: 12.6: 2.2) is used in this study.

Table 3-2 The composition of the stock synthetic wastewater in this study (Chung, 2007; Su, 2008).

Content	Doses (in 3 L distilled water)
KH_2PO_4	20 g
Glucose: Peptone	18 g: 9 g
Urea	60 g
NH_4Cl	125 g
whole milk powder	150 g
low-fat milk powder	122 g
$FeCl_3$ (10%)	2 mL
CH_3COOH (99.8%)	58 mL
$NaHCO_3$	256 g

Table 3-3 The water quality of influent synthetic wastewater.

Components	Concentration (mg/L)		
	Loading I	Loading II	Loading III
COD	329 ± 9.5**	458 ± 18.3**	675 ± 11.7**
NH_4^+-N	41 ±4.7**	57 ±7.2**	83 ± 3.1**
NO_x-N	N. D. *	N. D. *	N. D. *
PO_4^{3-}-P	7 ± 0.4**	10 ± 1.3**	16 ± 3.8**
COD: NH_4^+-N: PO_4^{3-}-P	100: 12.5: 2.1	100: 12.4: 2.2	100: 12.3: 2.3

*N. D. : Not detected

**n = 3

3.5 The methods of analysis

3.5.1 The methods of water analysis

Corresponding analysis method in this study is listed in Table 3-4. All the major parameters were analyzed according to Standard Method (APHA, 2005). Before conduct analysis, nitrite and nitrate samples must be filtered with 0.2μm membrane to prevent jamming. Hydroxylamine (NH_2OH) was determined by calorimetrical method (Peng, 2002). A 2 mL sample was filtered with 0.45μm membrane, then adding 1 ml 1% alcoholic 8-hydroxyquinolin (32502, R.D.H., Germany) and 1 ml 2N Na_2CO_3 (A356892, Merk, Germany). After mixing, obtained indoxine of indigo product then placed samples for 2 hours in room temperature. Sample was measured with a spectrophotometer (U-2000, HITACHI, Japan) with absorbance of UV light at 705 nm. The hydroxylamine standard solution (379921, Aldrich, USA, 99.9999%) was adjusted between 0.0 to 1.0 mg/L OF hydroxylamine and had a linear regression $R^2 > 0.995$.

Table 3-4 The analytic methods and instruments used in this study

Item	The Analysis Method and Instrument
COD	Methods 5220B*
BOD_5	Method 5210B
NH_4^+-N	Method 4500F*
NO_2^--N	Method 4500 NO_2^-B*
NO_3^--N	Method 4500 NO_3^-B*
pH	pH meter, Method 4500-H^+B* (SUNTEX PC-310)
ORP	ORP meter, Method 2580B*(SUNTEX PC-310)
DO	DO meter, Method 4500-O G* (SUNTEX DC-5100)
Temp	Temperature meter, NIEA W217.50A
MLSS	Method 2540 D*

* Standard Methods for the Examination of Water and Wastewater 21st Edition (APHA *et al.*, 2005)

3.5.2 Field Emission Gun Scanning Electron Microscopy (FESEM)

By FESEM analysis, it was a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography. Cold Field Emission Scanning Electron Microscope and Energy Dispersive Spectrometer (FESEM), JEOL JSM-6330F (Japan) in National TsingHua University and JEOL JSM-6500F (Japan) in National Chiao Tung University, were used to observe the micro morphologies and structure of original. First of all, the samples were placed on an aluminium holder and coated with a thin layer of Platinum (coating 5 min.) by sputtering to improve the electric conductivity.

3.5.3 Energy Dispersive X-ray analysis (EDX) analyses

Energy dispersive X-ray spectroscopy (EDX), JEOL JSM-6500F (Japan) in National Chiao Tung University, was an analytical technique used for the elemental analysis or chemical characterization of a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing X-rays that are characteristic of an element's atomic structure to be identified uniquely from one another.

First of all, the samples were placed on an aluminium holder and coated with a thin layer of platinum (coating 10 sec.) by sputtering to improve the electric conductivity.

3.5.4 The particle size analysis

The laser particle size analyzer, BECKMAN COULTER LS230 (USA) in Cheng Kung University, was the most widely used technique for particle size analysis. Instruments employing this technique are considered easy to use and particularly attractive for their capability to analyze over a broad size range in a variety of dispersion media.

Light scattered by particles forms a series of concentric rings of alternating maximum and minimum intensities, often called the Airy disk. The first minimum (closest to the centre of the Airy disk) provides information to determine the mean size of the distribution. Subsequent maxima and minima contain information on the shape and width of the distribution, including any shoulders and tails. It is this series of maxima and minima that needs to be accurately measured in order to report the true shape of the particle size distribution.

3.5.5 X-ray powder diffractometer (XRD)

X-ray powder diffraction (XRD), Rigaku TTRAX Ⅲ (Japan) in Cheng Kung University, is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined.

CHAPTER 4　RESULTS AND DISCUSSION

4.1 The basic characteristic of the domestic waste activated sludge

The basic physical characteristic of domestic WAS, obtained from the Futian water resource recycling center of Taichung City, are shown in Table 4-1.　After the wasted sludge was dried by belt press filter and baked in 105^{o}C for two days, the WAS has 1.77 % moisture content, and the raw WAS has 46.99 % flammable content and 51.24 % ash content. The flammable content will be burn out in high temperature, therefore, the pellets tend to be high porosity.　The laterite has most component of ash content, which could increase the strength of structure.

Table 4-1 The basic characteristics of wasted sludge sample (after 105°C treatment for 2 days) of Futian water resource recycling center of Taichung City.

Basic characteristic	Dry WAS
Moisture content, %	1.77 ± 0.48*
Flammable content, %	46.99 ± 0.56*
Ash content**, %	51.24 ± 0.76*
pH	4.71 ± 0.16*

*n=3

**800°C, 3hr

Table 4-2 The basic characteristics of laterite sample of Tunghai University (after 105°C treatment for 2 days).

Basic characteristic	Laterite
Moisture content, %	1.02 ± 0.15*
Flammable content, %	3.06 ± 0.34*
Ash content**, %	95.92 ± 0.36*
pH	4.80 ± 0.26*

*n=3

**800°C, 3hr

The material was ground into powder by ball mill machine. Figure 4-1 indicated the particle size distribution and the average diameter were mainly in the 12μm. The smaller the powder and space between particles are the more tightand hardness the porous WAS pellets would be.

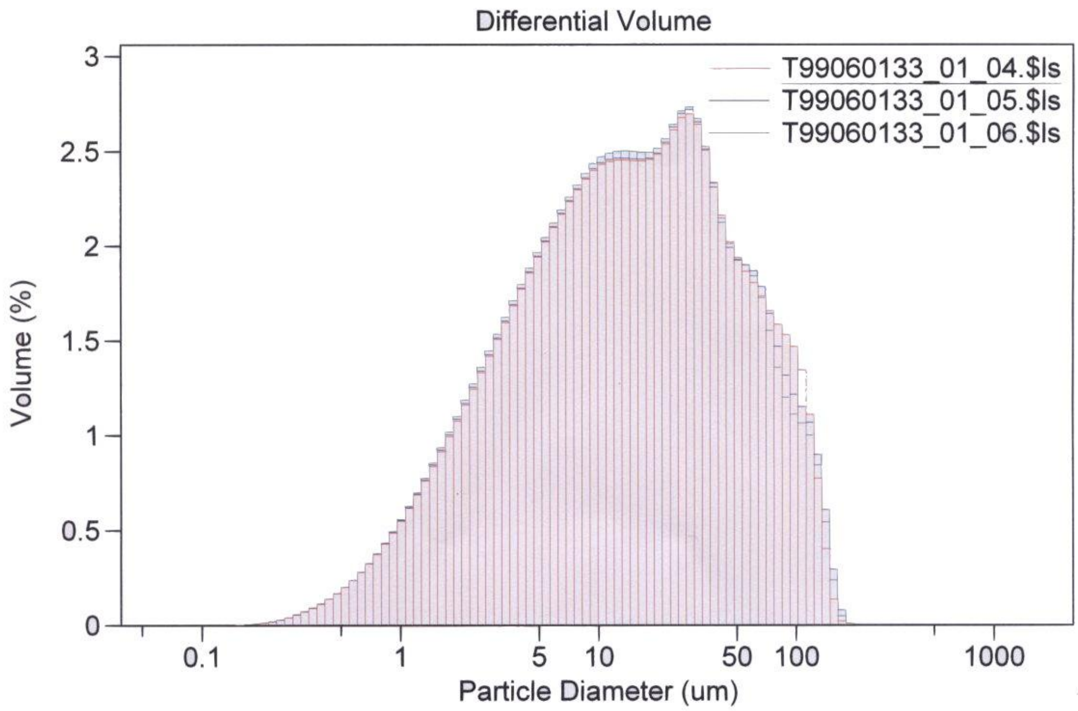


Figure 4-1 The particles size distribution analysis of the WAS powder.

Toxicity characteristics leaching procedure (TCLP) test for the WAS from the domestic waste treatment plant was tested. Table 4-3 indicated the leaching of heavy metals from WAS were all below the limitation regulations and standards.

Table 4-3 The TCLP test for heavy metal concentration of Futian WAS and laterite.

Element	Concentration of WAS (mg/L)	Concentration of laterite (mg/L)	Regulation of TCLP from HM (mg/L)***
Cu	0.123*	0.019*	15
Pb	0.030*	0.010*	5
Cr	0.005*	0.003*	10
Zn	3.330*	0.090*	25
Cd	ND**	ND**	0.5

*n=3

**ND: Not detected

***Heavy metals regulation was obtained from Waste Material Cleanup, EPA, Taiwan

4.2 The characteristic of the biofilm carrier pellets

The purpose of this study is to build WAS pellets as immobilized media to enhance wastewater treatment efficiency. This study was added the nutrients in the WAS carriers and tried to encapsulated the nutrients in the particles. In the experiment, the nutrients were not leached in the water; the data were all lower than the detection limit (DL). Normally, it is difficult to reuse WAS directly to build material due to its weakness. After repeatedly tests, the optimal formula ratios of rebuilt pellets are 50% sludge. Table 4-4 shows the compressive strength, density, water absorption and specific external surface area under the optimal ratio of rebuilt WAS pellets and other references. The 50% sample was the best formula. Although the compressive strength was lower than Su's study, it was lower than the regulations. The standard regulation of the industrial waste compressive strength was lower than 10kgf/cm^2. The plot of DOE by MINITAB® was shows in Figure 4-2. The result of DOE indicated that the best formula was at the WAS about 50%, the surface area about 2 cm^2/g and compressive strength about 11 kgf/cm^2.

Table 4-4 The bulk density, water absorption, compressive strength and specific external surface area of various ratio of rebuilt WAS pallets.

Pellets	Bulk density (g/cm^3)	Water absorption (%)	Compressive strength (kgf/cm^2)	Specific external surface area (cm^2/g)
50%[1]	1.54 ± 0.08*	41.75 ± 2.59*	11.30 ± 2.52*	2.23 ± 0.11*
63%[1]	1.51 ± 0.07*	54.37 ± 0.37*	6.43 ± 1.30*	2.27 ± 0.10*
73%[1]	1.28 ± 0.13*	60.33 ± 1.75*	2.96 ± 0.72*	2.70 ± 0.26*
50%[2]	1.47 ± 0.31*	45.07 ± 1.12*	10.48 ± 1.82*	2.33 ± 0.31*
Su, 2008	7.10	32.99	36.70	
Chung, 2007	1.10	41.02	6.40	
Chen, 2005	0.67	60.49	2.44	

[1] Percentage of sludge

[2] Pellets of add nutrient

* n = 3

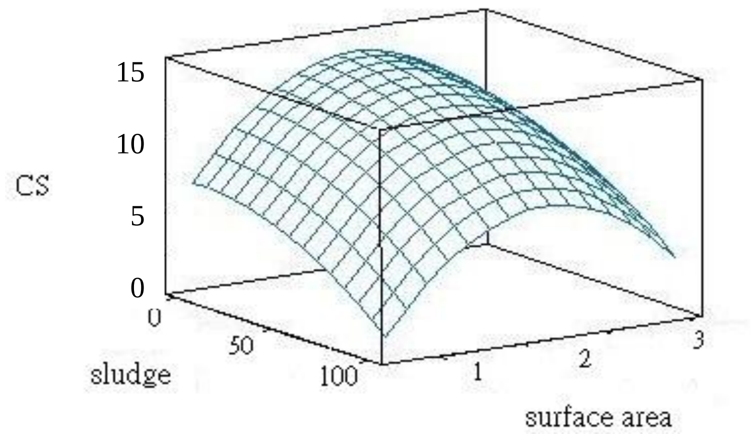


Figure 4-2 Surface plot of compressive strength (CS) (kgf/cm^2), dry sludge (%) and surface area (cm^2/g).

4.2.1 The TCLP test

After obtaining the optimal ratio of pellets, the toxicity characteristic leaching procedure (TCLP) test was conducted to detect the potential of toxic heavy metals leached. After the TCLP test, the leached toxic heavy metals are far below the Taiwan's limitations and the result is shown in Table 4-5.

Table 4-5 The TCLP test for heavy metal concentration of the porous WAS pallets.

Element	Concentration of pellet samples (mg/L)	Regulation of TCLP from HM (mg/L)***
Cu	0.186*	15
Pb	0.122*	5
Cr	0.003*	10
Zn	0.970*	25
Cd	ND**	0.5

*n=3

**ND: None detected

***Heavy metals regulation was obtained from Waste Material Cleanup, EPA, Taiwan

4.2.2 The pellets surface image and composition

Figure 4-3 shows the pictures of external shape of the rebuilt WAS pellets. There are no difference in the appearance of raw pellets and nutrient pellets. The rebuilt pellet diameters are between 12 to 14 mm. Figure 4-4 shows the porous image of rebuilt WAS pellet under SEM, which shows there are many pores on the rebuilt pellet. And compared with previous studies of particles in this study contains more pores. The porous surface is suitable for the bacteria to growth and attach on it.

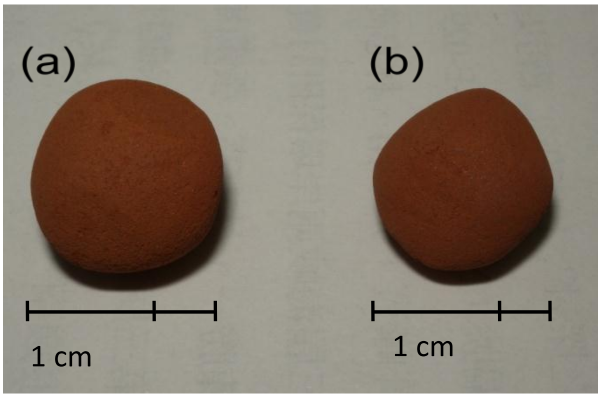
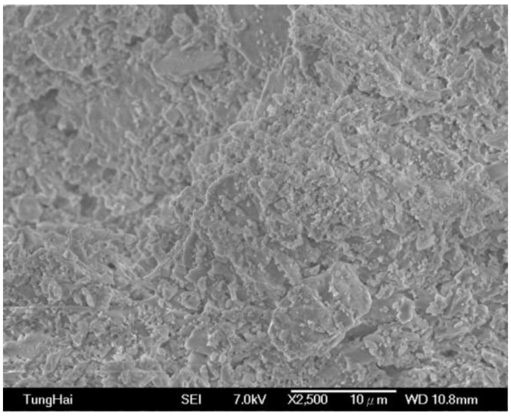
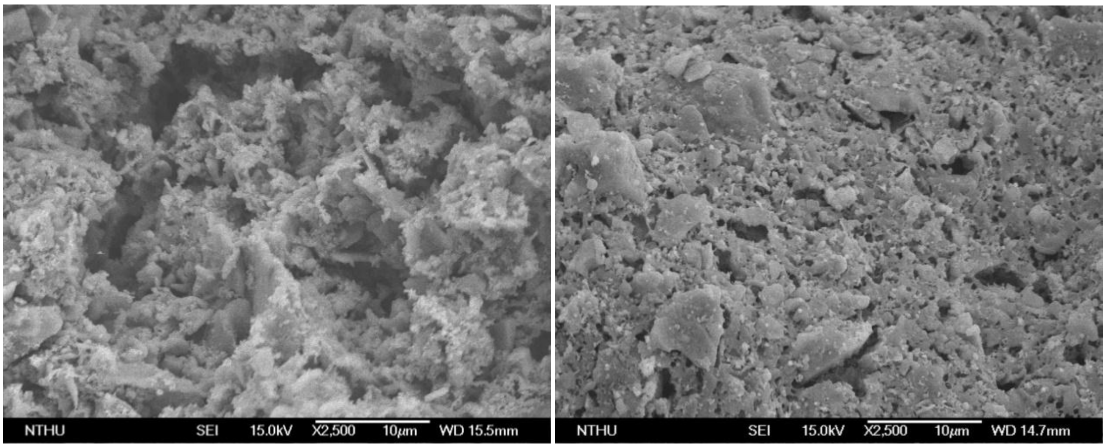


Figure 4-3 (a) The raw pellet. (b) The nutrient pellet.



(a) Su, 2008, the fine WAS pellets



(b) This study, 2010 (Left is raw pellets, Right is nutrient pellets)

Figure 4-4 The SEM image of the pellets surface.

The elemental analysis of raw pellet and pellet with nutrient addition are measured by EDS spectrum as shown in Figure 4-5 and Figure 4-6, respectively.The EDS result of raw pellet in Figure 4-5 (b) indicates the presence of silicon, aluminum and oxygen. On the other hand, the result of nutrient additive pellet in Figure 4-6 (b) indicates the presence of ferric, silicon, aluminum and oxygen.

(a)

(b)

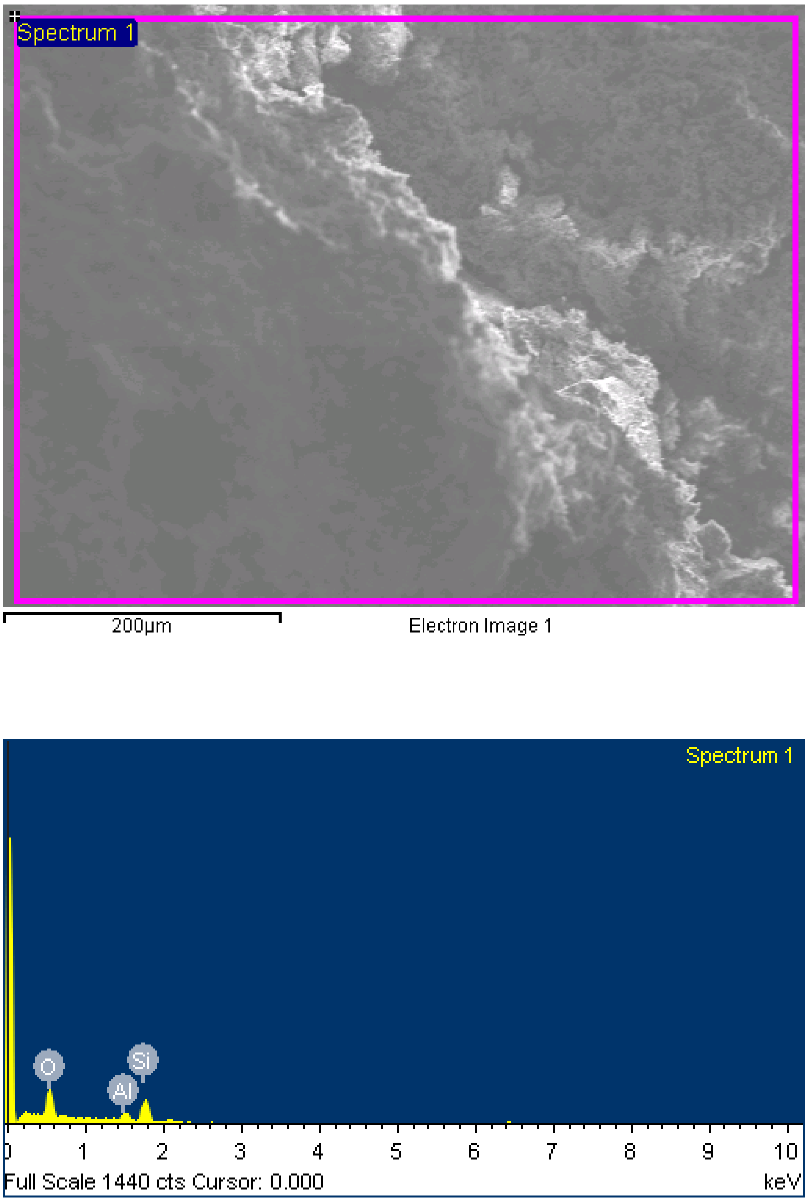


Figure 4-5(a) SEM and (b) EDS analysis of raw pellet.

(a)
(b)

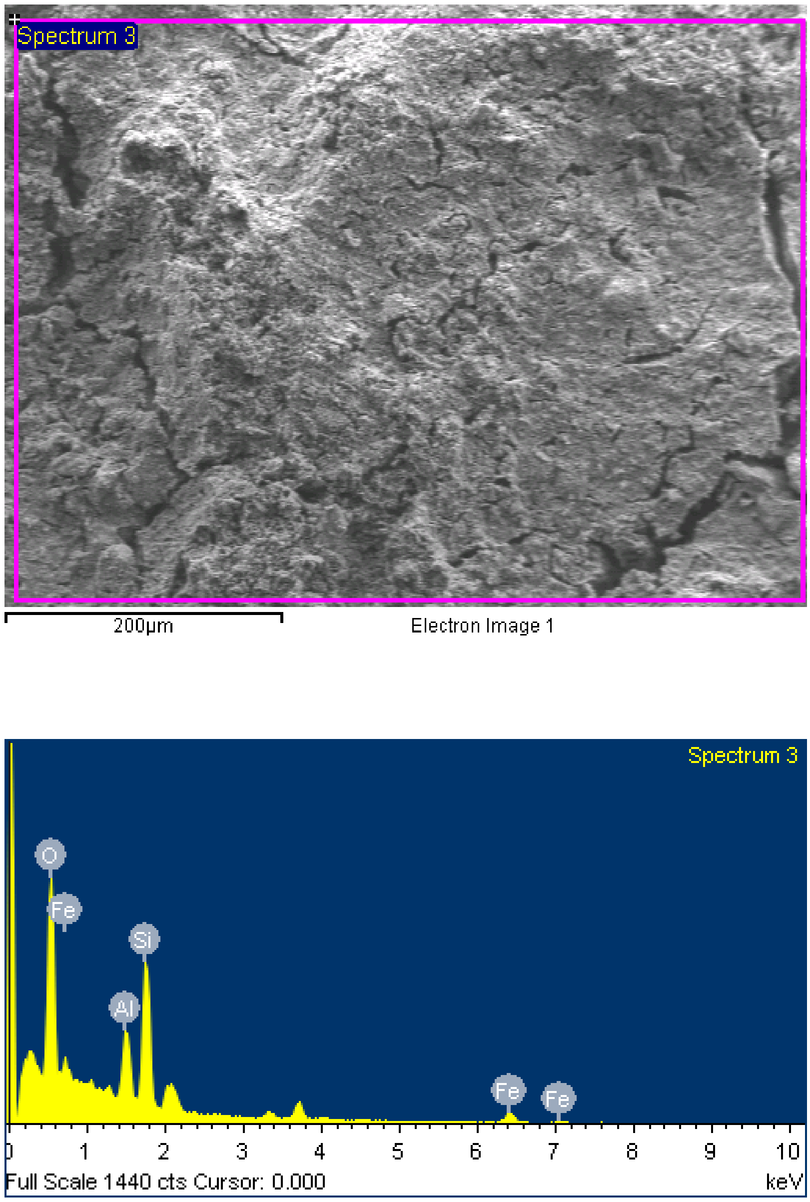
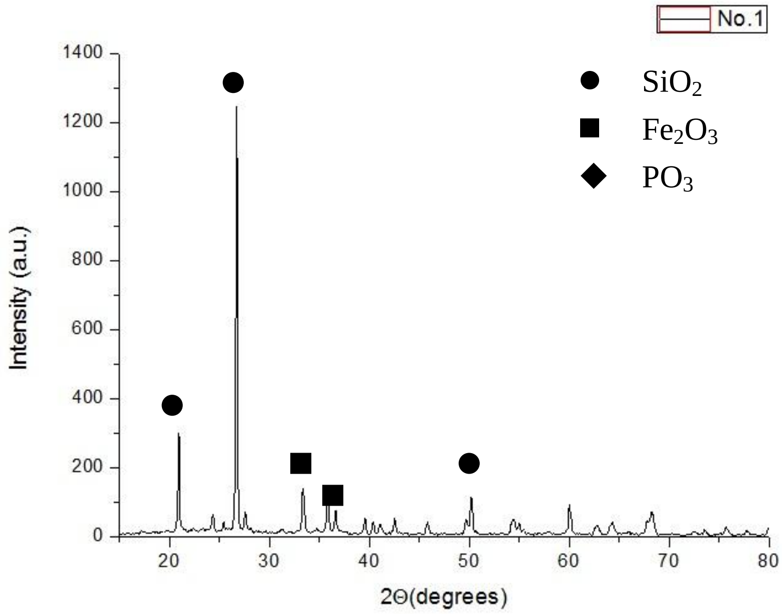


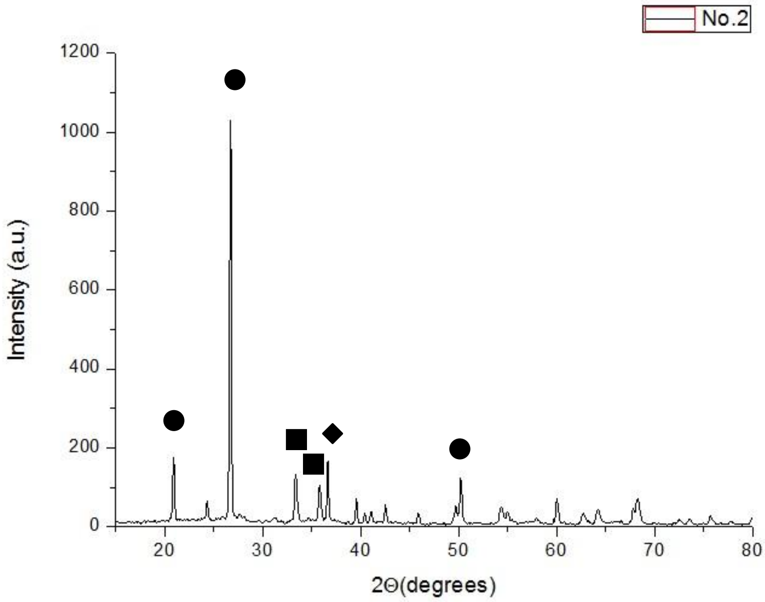
Figure 4-6(a) SEM and (b) EDS analysis of nutrient additive pellet.

4.2.3 The XRD patterns of the pellets

The XRD patterns of two pellets are illustrated in Figure 4-5. The compound was found out by the Jade 5. Jade 5 is the software to find out the possible componentsin the analysis data of XRD. The peaks primarily represent Si, Al and Fe, but Figure 4-7 shows the phosphorus content. But in the materials, also added the potassium and nitrogen, it was not shows in the XRD pattern. The contents were low level to not detect.



(a)



(b)

Figure 4-7 XRD patterns with the (a) raw pellets and (b) nutrient additive pellets.

4.3 Apply the rebuilt WAS pellets in SBBR systems

4.3.1 The daily monitor profiles in system

The two reactors (The reactors were the SBBR system with the raw pellets and the SBBR-Nutrient system with added nutrient pellets)were operatedfor 140 days under three loading stages: Loading Ⅰ (F/M: 0.309 kg COD/ kg MLSS-day, 0.039 kg NH_4^+-N/ kg MLSS-day) for 70 days, Loading Ⅱ(F/M: 0.430 kg COD/ kg MLSS-day, 0.053 kg NH_4^+-N/ kg MLSS-day) for 30 days and followed by Loading Ⅲ(F/M: 0.627 kg COD/ kg MLSS-day, 0.078 kg NH_4^+-N/ kg MLSS-day) for 40 days.

Both systems were added the rebuilt pellets and nutrient pellets in reactors with the volumetric packing ratio of 15 %, respectively

Figure 4-8 shows the daily influent and effluent profiles of COD in two reactors. The effluents of COD were always below 20 mg/L.

Figure 4-9 shows the daily influent and effluent profiles of ammounium in two reactors. The three loading operatedammounium were 41 mg/L, 57 mg/L and 83 mg/L for Loading Ⅰ, Ⅱ andⅢ phase, respectively. At steady state of each operation phase, the effluentammonium were always below 1 mg/L. The DO level had to rise under LoadingⅢ, because the ammonium concentration was too high to treated by the system.

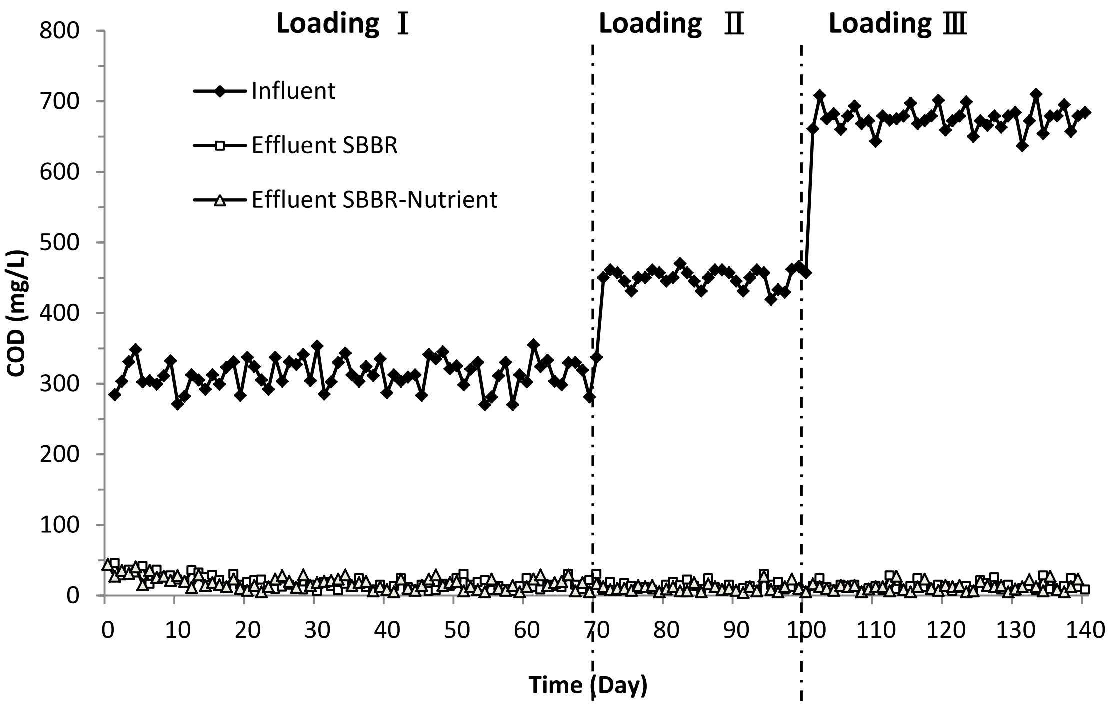


Figure 4-8 The daily profiles of COD in two types of system (SBBR and SBBR-Nutrient). The period of Loading Ⅰ (F/M: 0.309 kg COD/ kg MLSS-day) under 70 days, Loading Ⅱ(F/M: 0.430 kg COD/ kg MLSS-day) under 30 days and highest concentration was Loading Ⅲ(F/M: 0.627 kg COD/ kg MLSS-day) under 40 days, respectively.

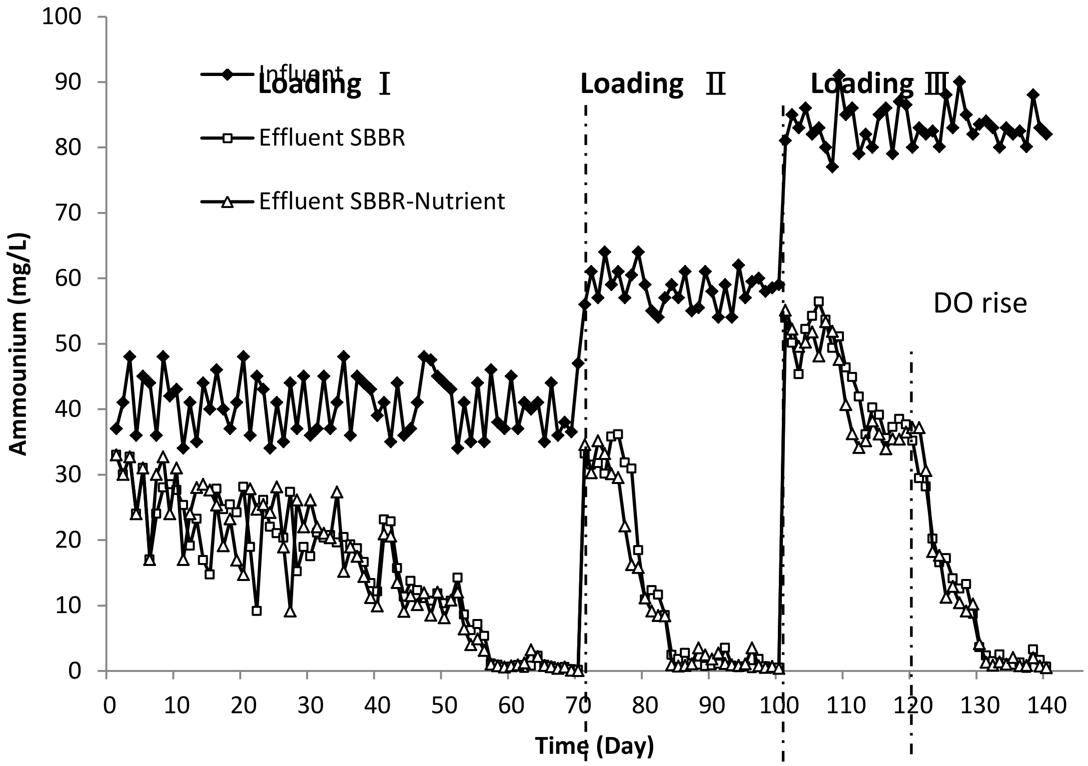


Figure 4-9 The daily profiles of NH_4^+-N in two types of system (SBBR and SBBR-Nutrient). The operation period of Loading stage I, II and III are 70 days, 30 days and 40 days, respectively.Loading Ⅰ (F/M: 0.039 kg NH_4^+-N/ kg MLSS-day), Loading Ⅱ(F/M: 0.053 kg NH_4^+-N/ kg MLSS-day) and Loading Ⅲ(F/M: 0.078 kg NH_4^+-N/ kg MLSS-day), respectively.

4.3.2 The profiles of a cycle in the two SBBR systems

To test the added nutrient pellets applied to the feasibility of biological treatment systems. Design two reaction systems; one was design for the use of added nutrient pellets in the SBBR system, another control system to use the raw pellets in the SBBR system. Two systems both used as the same condition in control and experiment. This study not only tests the feasibility of added nutrient pellets, and to compare the two reactor systems for biological nutrient removal (BNR).

1. SBBR system :

Figure 4-10 is the data profiles under Loading Ⅰ (F/M: 0.309 kg COD/ kg MLSS-day, 0.039 kg NH_4^+-N/ kg MLSS-day) in SBBR system. Each test cycle was 12 hours with 30 minutes per sample taken by online real-time monitoring systems to record ORP, DO and pH.

The NH_4^+-N concentration was reduced from 41.0 mg/L reduce to less than MDL (0.01 mg/L). The DO concentration in reactor was suppressed and the nitrification rate was 0.021 kg NH_4^+-N/ kg MLSS-day (calculated by *Eq. 4-1*, proposed by Carrera *et al.*, 2004). In order to achieve the theoretical SND smoothly, nitrification and denitrificationwithin the reactor must keep identically, i.e., no excess by-products would accumulate in the system. Therefore, nitrite and nitrate generation rates were controlled to approach close enough to their consuming rate. Hence, nitrite and nitrate did not accumulate in system at all.

$$r_{nitrification} = \frac{Q_{in}\left(\left[NH_4^+ - N\right]_{in} - \left[NH_4^+ - N\right]_{out}\right)}{V_{reactors}\left[VSS\right]_{reactors}} \quad \ldots\ldots\ldots Eq.(4-1)$$

(Carrera *et al.*, 2004)

The COD and phosphate can almost be removed completely without any interference. Particularly for phosphate, the “luxury uptake” happens in aerobic condition of system in aeration period. The low DO concentration did not interference this overall bio-reaction. The phosphate can remove completely during the aerobic stage at the reaction time of 180 min. The SBBR system was processed simultaneous nitrification and denitrification (SND), the SND process was used to prove the measured the mid-product of NH_2OH.

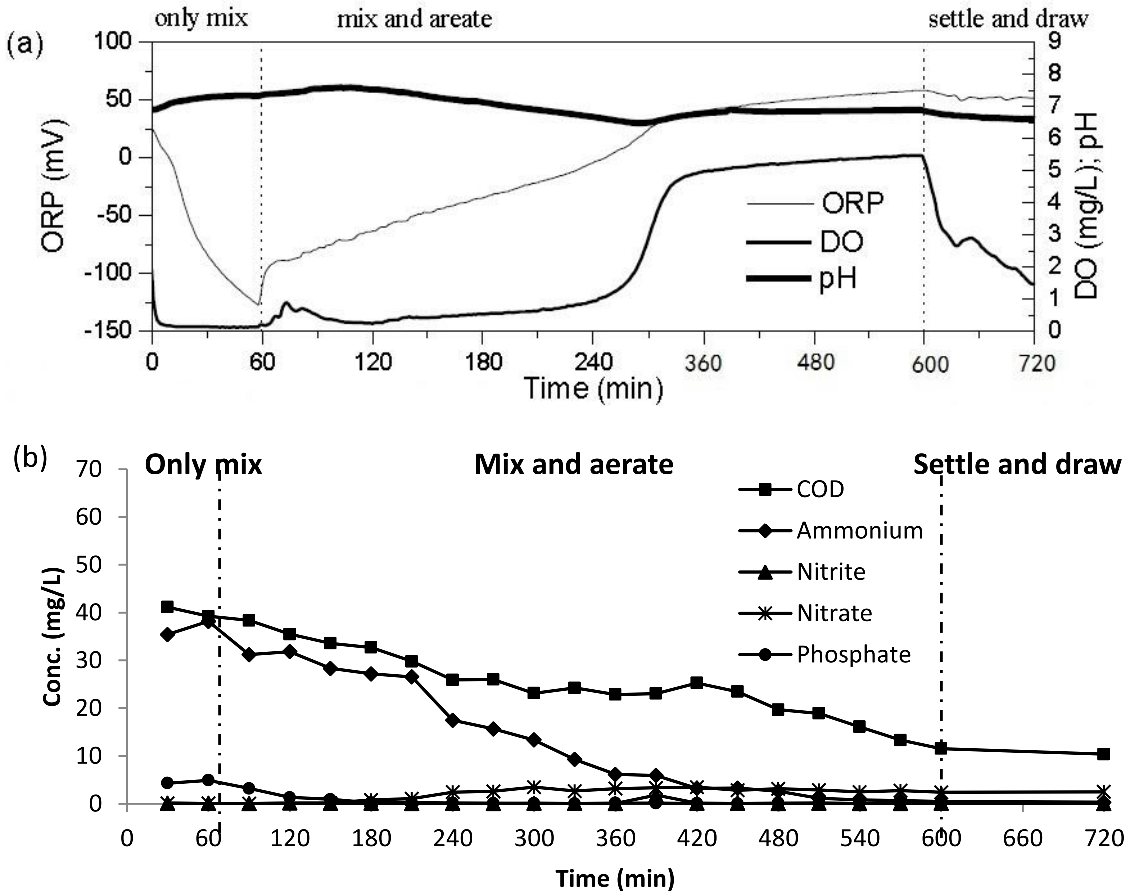


Figure 4-10 The on-line measured parameters (ORP, pH and DO), COD, nitrogen and phosphate concentration in the batch test of the SBBR system (Loading I); (a): the profiles of ORP, pH and DO; (b): NH_4^+-N, NO_2^--N, NO_3^--N, SCOD and PO_4^{3-}-P concentration.

2. SBBR-Nutrient system :

Figure 4-11 shows the batch test of SBBR system with added nutrient pellets in Loading I. Under the same control conditions, the nitrite was almost not detected in the reactor. When ammonium decreased in the system, the nitrite did not increase (below 3 mg/L) and nitrate was not detected. This means the nitrification transfer ammonium to nitrite and then denitrification happened consecutively. The nitrification rate of SBBR-Nutrient system was same with the SBBR system, 0.045 g NH_4^+-N g VSS^{-1} per day. This system was processed simultaneous nitrification and denitrification (SND), while the phosphate was disappeared in the reactor after 120 min of aeration by Luxury uptake.

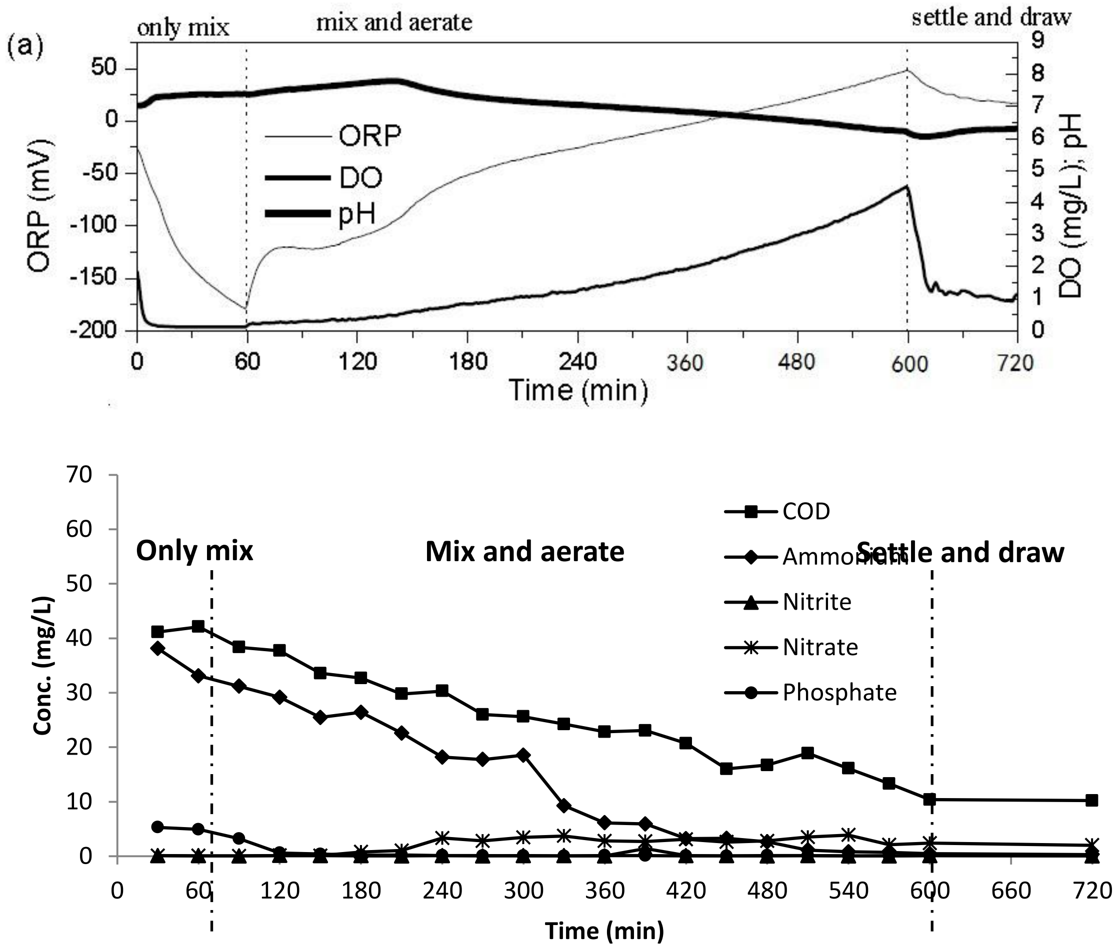


Figure 4-11 The on-line measured parameter (ORP, pH and DO), COD, nitrogen and phosphate concentration in the batch test of the SBBR system with nutrient pellets (Loading I); (a) the profiles of ORP, pH and DO; (b) NH_4^+-N, NO_2^--N, NO_3^--N, SCOD and PO_4^{3-}-P concentration.

4.3.3 The profiles of batch tests

Figure 4-12 shows the performance of traditional SBBR system with raw WAS pellets in Loading Ⅰ (F/M: 0.309 kg COD/ kg MLSS-day, 0.039 kg NH_4^+-N/ kg MLSS-day, C/N: 8.02), Ⅱ (F/M: 0.430 kg COD/ kg MLSS-day, 0.053 kg NH_4^+-N/ kg MLSS-day, C/N: 8.04) and III (F/M: 0.627 kg COD/ kg MLSS-day, 0.078 kg NH_4^+-N/ kg MLSS-day, C/N: 8.13). After the three loadings test reach to a steady state, the reactor was conducted the batch tests. In Figure 4-13 (a), there are not significant differences of COD in three loadings (below 15 mg/L).

In Figure 4-13 (b), the ammounium was removed completed after rise the DO level under Loading Ⅲ. Because of the nitrate was produced between nitrification and denitrification, the nitrate was started to accumulated at 150 min. The condition was indicated that the SND process was not completed (Figure 2-2). In Figures 4-12 (b) and 4-13 (e), when the phosphate was removed, the pH level was drop at 120 min under Loadings Ⅰ and Ⅱ (Lee *et al*., 2001).

Chiu *et al*., (2007) indicated that the SND process was different from the traditional nitrification and denitrification (Figure 2-2). The ammonia was produced the mid-product of hydroxylamine (NH_2OH) in the oxidation, and then oxidized to the nitrite. Finally, the nitrite was reduced to N_2 and N_2O. In this batch reaction, the nitrate was not detected in this test, while the hydroxylamine could prove this was proposed following a proposed SND pathway (Figure 4-14).

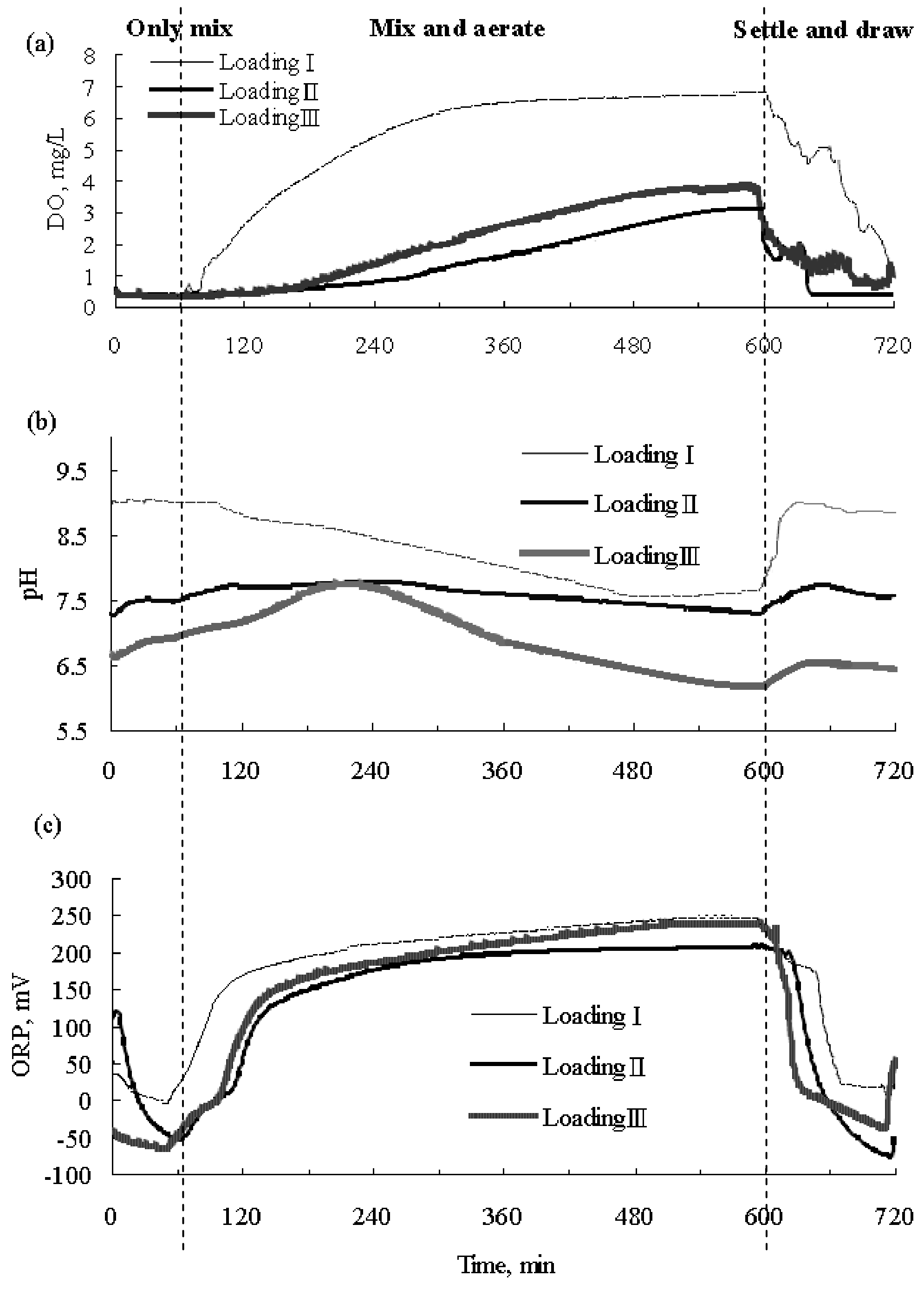


Figure 4-12 The comparison batch tests of various Loadings (I, II and III) in SBBR system with raw pellets (a) DO, (b) pH and (c) ORP.

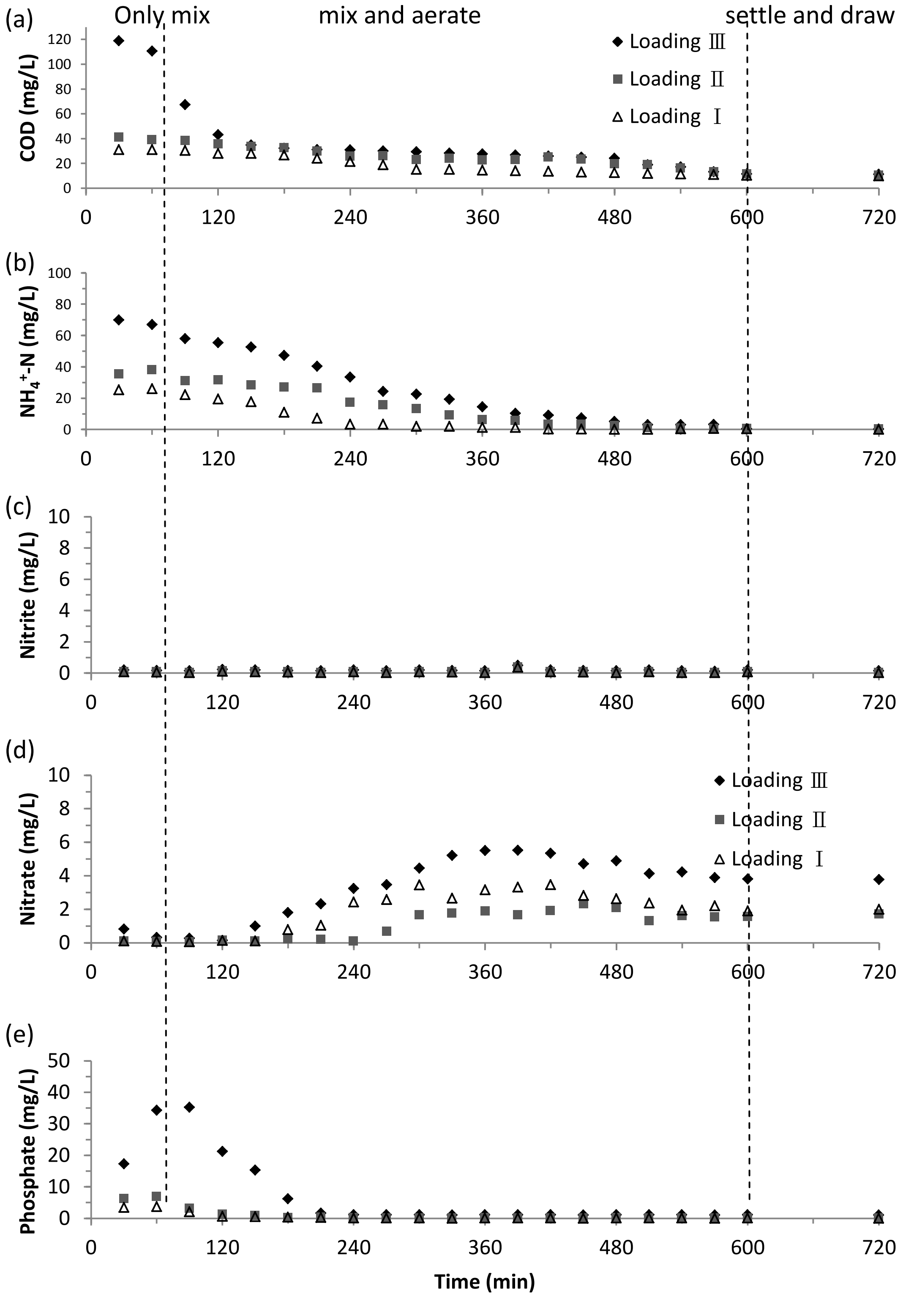


Figure 4-13 The comparison batch tests of various Loadings (I, II and III) in SBBR system with raw pellets (a) COD, (b) NH_4^+-N, (c) NO_2^--N, (d) NO_3^--N and (e) PO_4^{3-}-P.

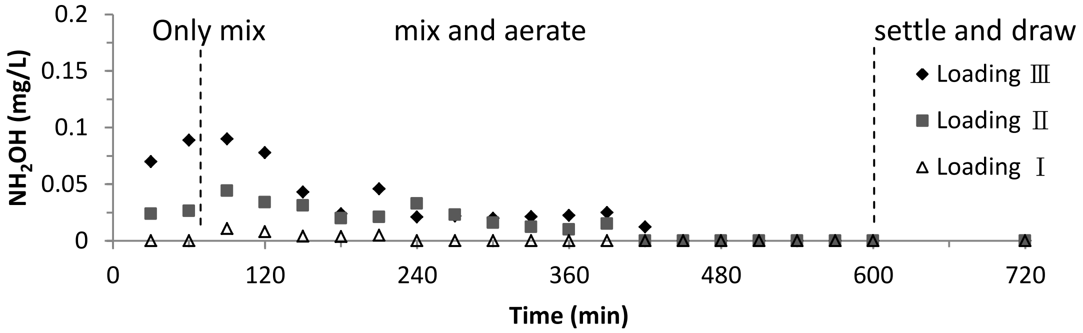


Figure 4-14 The profile of SND mid-product of hydroxylamine (NH_2OH) in SBBR system with raw pellets.

After the SBBR system with added nutrient pellets reaching the steady state, the reactor was conducted tests under three loadings (Loading Ⅰ, Ⅱ and Ⅲ) case. The effluent COD level was all below 15 mg/L, also similar to the SBBR system with raw pellets.

The ammounium was not completely removed, but the level was below 5 mg/L at 420 min (Figure 4-16). In the Figure 4-15 (d), the nitrate was reduced below 5 mg/L under Loading Ⅱ and the nitrite was not oxidized to nitrate, which was also following the proposed SND pathway.

The Guo*et al.*, (2009) indicated that the high level DO may inhabit the SND process. In two systems, the DO level of the Loading Ⅰ (6.8 mg/L) and Ⅲ (3.7 mg/L) was higher than Loading Ⅱ (3.0 mg/L) (Figure 4-12 (a) and Figure 4-15 (a)). While the Figure 4-13 (d) and Figure 4-16 (d) shows that the nitrate of Loading Ⅱ (1.72 mg/L) was lower than other loadings (3.77 mg/L, 2.02 mg/L) for Loading Ⅰ and Ⅲ cases.

In the Figure 4-19, the hydroxylamine of Loading Ⅱ was significant higher than the SBBR system (below 0.05 mg/L), which was identical to the nitrite and DO variation.

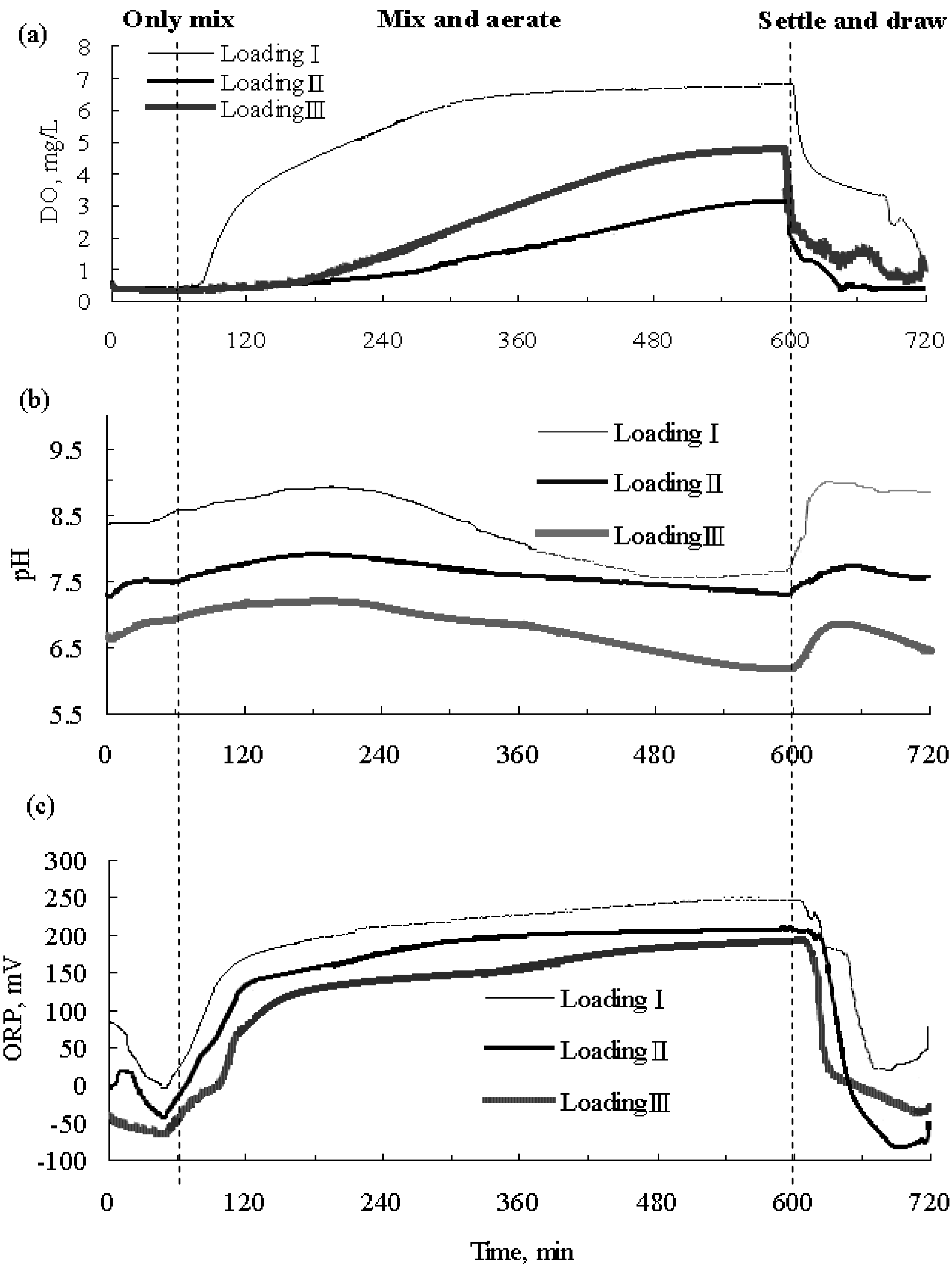


Figure 4-15 The comparison batch tests of various Loadings (I, II and III) in SBBR-Nutrient system (a) DO, (b) pH and (c) ORP.

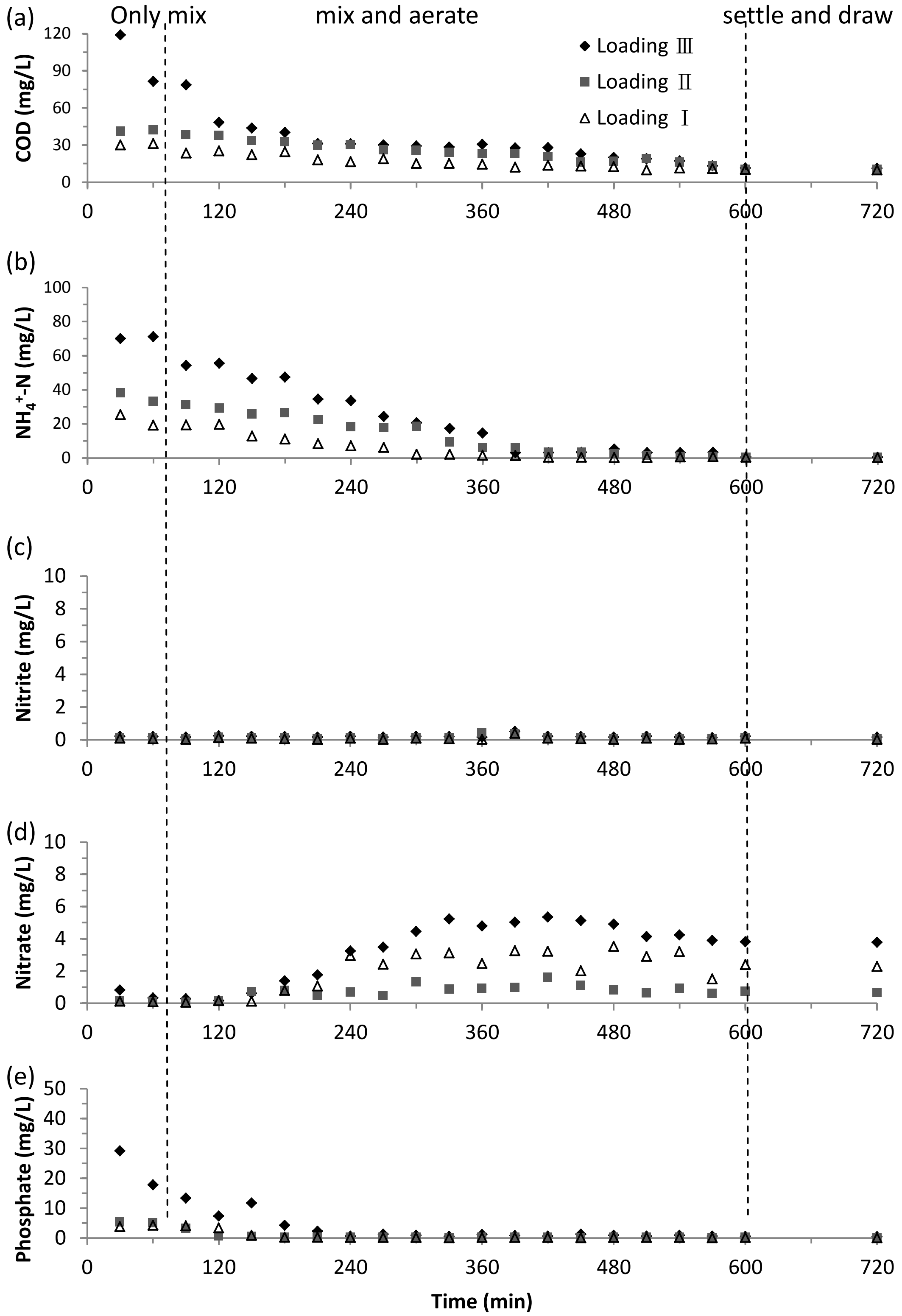


Figure 4-16 The comparison batch tests of various Loadings (I, II and III) in SBBR-Nutrient system (a) COD, (b) NH_4^+-N, (c) NO_2^--N, (d) NO_3^--N and (e) PO_4^{3-}-P.

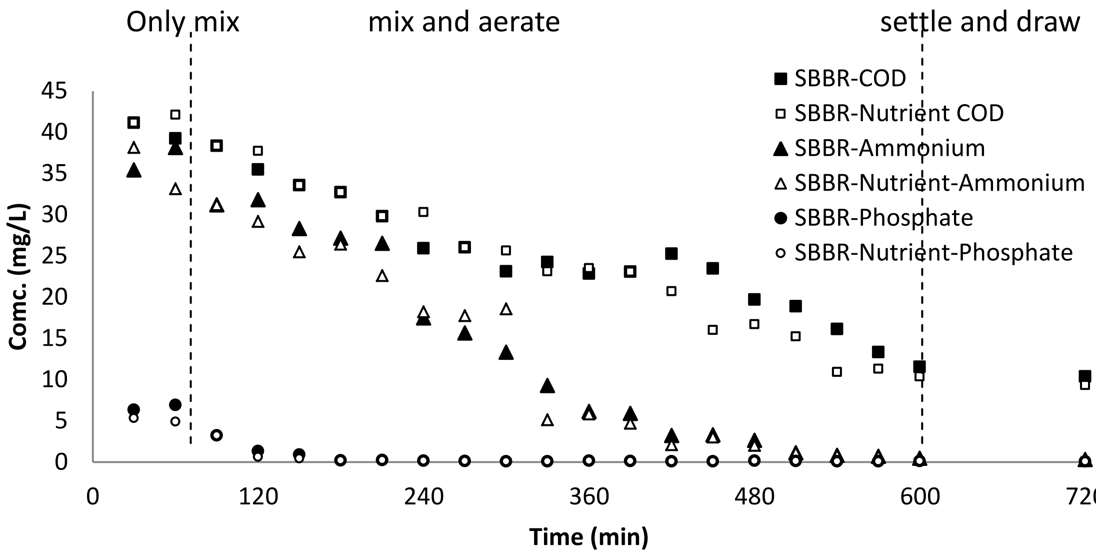


Figure 4-17 The profile of the data (COD, NH_4^+-N and PO_4^{3-}) in two systems under Loading Ⅱ (F/M: 0.430 kg COD/ kg MLSS-day, 0.053 kg NH_4^+-N/ kg MLSS-day)

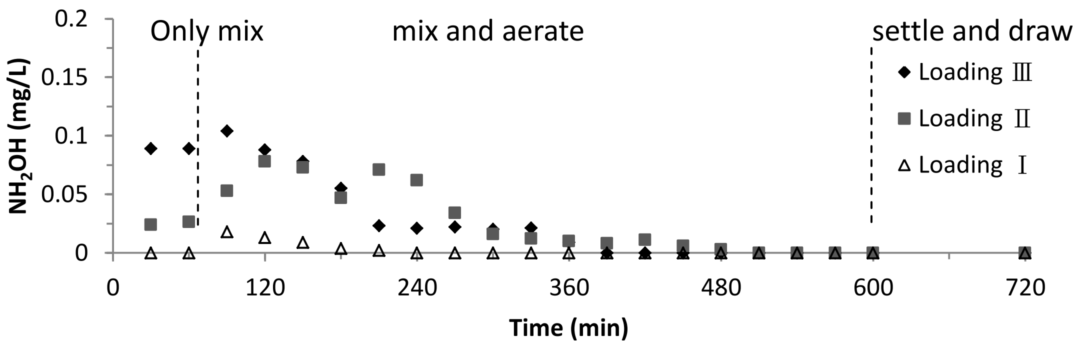


Figure 4-18 The profile of hydroxylamine (NH_2OH) in SBBR-Nutrient system.

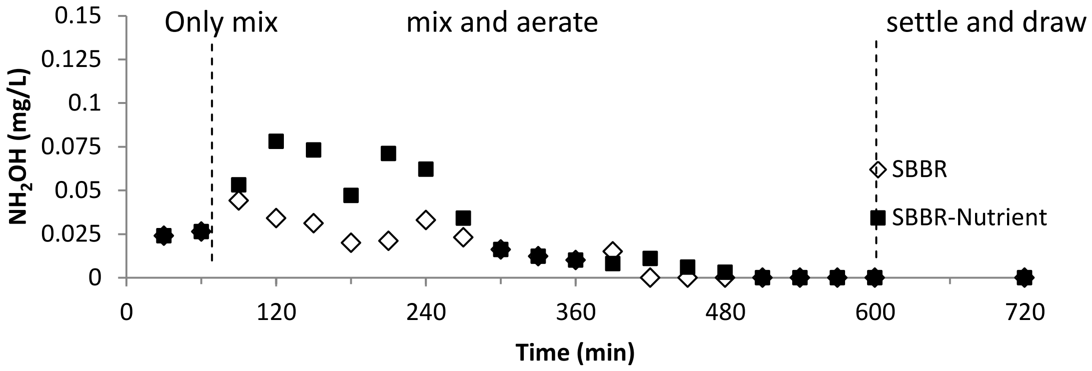


Figure 4-19 The profile of hydroxylamine (NH_2OH) in two systems under Loading Ⅱ (F/M: 0.430 kg COD/ kg MLSS-day, 0.053 kg NH_4^+-N/ kg MLSS-day)

4.3.4 The biomass of two pellets in systems

During the systems were cultured, we took the pellets out once 10 days. Drying them under 105°C and weighting until reach to a constant weight. The Figure 4-20 shows the weight variation of the pellets.

Biomass was the dry weight of the pellet in the reactor subtract the dry weight of the pellets before put in the reactor.

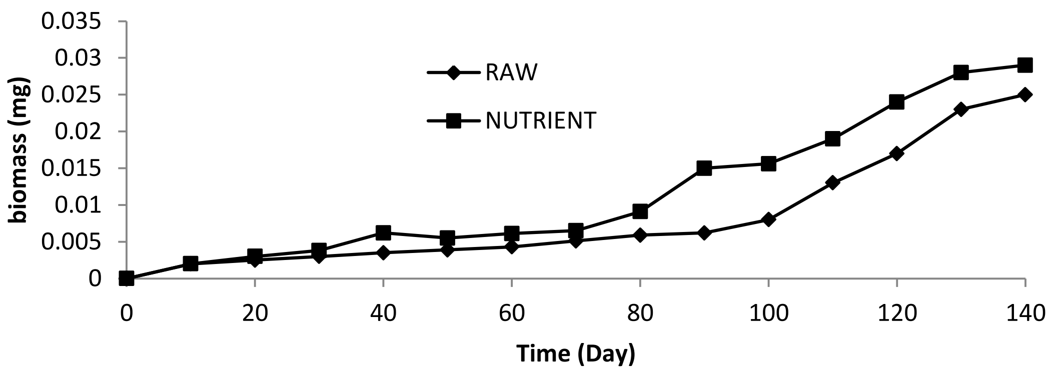


Figure 4-20 The biomass of two pellets. (Raw and added nutrient)

4.4 Comparison the K_N, K_{DN} and SND efficiency of different system

Table 4-6 shows the nitrification rate (K_N), denitrification rate (K_{DN}) and SND efficiency of different systems from this study and other references. Table 4-6 shows the typical SBBR system (with raw pellets), SBBR system with added nutrient pellets. The efficiency of SND is calculated by *Eq. (4-2)* (Zeng*et al.*, 2003):

$$Efficiency_{SND} = \frac{Denitrification}{Nitrification} = \frac{NH_4(tot) - NO_x(acc)}{NH_4(tot)} \text{..........................} Eq.(4-2)$$

Where $NH_4\,(tot)$ is the influent concentration of ammonium; $NO_x(acc)$ is the effluent concentration of NO_x-N.

In Table 4-6, both systems perform well (96-98 % removal) compared to other studies. The results show the K_N and $K_{DN}$are proportional to that of the loadings (from Loading I to II) in SBBR system with rebuilt WAS pellets. The nitrification rate (K_N = 6.0 mg-NH_4^+-N/L-hr) and denitrification rate (K_{DN} = 6.2 mg-NH_4^+-N/L-hr) of SBBR-Nutrient system were higher than the SBBR system (K_N = 5.6 mg-NH_4^+-N/L-hr, K_{DN} = 5.4 mg-NH_4^+-N/L-hr) under phase II with the added nutrient pellets with the 98 % SND removal efficiency. Due to the removal capability in SBBR-Nitrient system reach to the overloading case (Loading III), therefore, rise the DO level to increased the treatment efficiency, the SND efficiency was higher than Loading II.

Table 4-6. The comparison of nitrification rate (K_N), denitrification rate (K_{DN}) and SND efficiency in different systems in this reactor and other references.

Reference		K_N[a]	K_{DN}[b]	$Efficiency_{SND}$
This study, (SBBR, SBBR-Nutrient under Loading Ⅱ)	SBBR	5.6	5.4	96±0.005%
	SBBR-Nutrient	6.0	6.2	98±0.003%
This study, (SBBR-Nutrient system)	Loading Ⅰ	4.2	4.3	93%
	Loading Ⅱ	6.0	6.2	98%
	Loading Ⅲ	7.8	7.5	95%
Guo, 2009 (SBR-1 high DO (above 3 mg/L) SBR-2 low DO (0.4–0.8 mg/L)	(SND)	5.2	4.9	31%
				67%
Su, 2008 (SBBR system with WAS pellets diffusers) (SND)	Loading I	4.2	4.2	95%
	Loading II	5.9	5.8	98%
	Loading III	8.1	7.7	94%
Chung, 2007 (SBBR system with rebuilt WAS pellets, SBBR system with commercial pellets) (SND)		5.5	5.2	94%
		5.5	4.9	89%
Yang, 2005 (Low DO, SND, SBR-Member)		5.1	3.6	75%
		4.7	4.4	95%
Chen, 2005 (SBBR)		3.7	2.4	72%
		5.0	4.7	90%
Li, 2004		9.4	9.2	99%
Gieseke *et al.*, 2002 (SBBR)		4.1	3.7	84%
Park *et al.*, 2002		6.7	5.3	45%
Jun *et al.*, 2000 (SBBR)		3.2	1.5	68%

[a] Express as mg-NH_4^+-N/L-hr (nitrification rate)

[b] Express as mg-NO_3^--N/L-hr (denitrification rate)

4.5Model Development

4.5.1 Overall SND process in immobilized system

From Figure 4-16 (b), the SBBR-Nutrient system can achieve SND process smoothly. Theoretically, balanced SND system has equally nitrification and denitrification reaction rate, i.e., it implies no intermediate with product (i.e. nitrate and nitrite) occurs. Therefore, the statement of the Nernst equation for the complete SND process can combine *Eq. (4-1)* with *Eq. (4-2).* Due to the excessive carbon substrate existed in the denitrification of SND process, the carbon concentration ($C_xH_yO_z$) can assumed to be constant. Therefore, the *Eq. (4-3)* can be obtained as follows:

$$E = E^0 + \frac{RT}{nF}\ln\left(\frac{[NH_4^+](P_{O_2})^2}{[NO_2^-][H^+]}\right) \quad \text{Eq. (4-1)}$$

$$E = i + j\ln([C_xH_yO_z]) + h\ln([NO_2^-]) + k\ln(\frac{1}{[OH^-]}) \quad \text{Eq. (4-2)}$$

$$E = a'' + b''\,pH + c''\log(NO_2^-) + d''\log(C_xH_yO_z) \quad \text{Eq. (4-3)}$$

$$E = a + bpH + c\ln([NH_4^+])([PO_4^{3-}]) \quad \text{Eq. (4-4)}$$

(Lee, 2004)

4.5.2Nernst Equation established in SND Process

1. First stage: phosphate and ammonium removal

According to the reaction model of Nernst equation which covers both anoxic and aerated, therefore, a suitable simulation can be approached. Table 4-7 shows the Nernst equation modeling results of both anoxic and aerobic cases in immobilized SBBR systems dealing with nitrogen and phosphate removal. Both the fitted models indicate well correlation coefficient (R^2) value of 0.94 - 0.99. This indicates that both *Eq. (4-5)* and *Eq. (4-3)* can describe the overall profiles of ORP in the SBBR systems. Figure 4-21 shows the model simulation and the experimental data, which shows a close correlation. The ORP model can be adapted to the further control the phosphate removal end point of SBBR systems.

$$E = a' + b'pH + c'\log\frac{[NH_4^+]}{[NO_2^-]}$$ ……………………………………...*Eq. (4-5)*

Table 4-7. Results of the Nernst equation for "only mix" and "mix and aerate" steps in two SBBR systems

	Nernst equation model constants			
SBBR with Added nutrient pellets	a	b	c	R^2
Only mix	-4986.01	635.20	13.57	0.99
Mix and aerate	27.43	25.39	-2.73	0.96

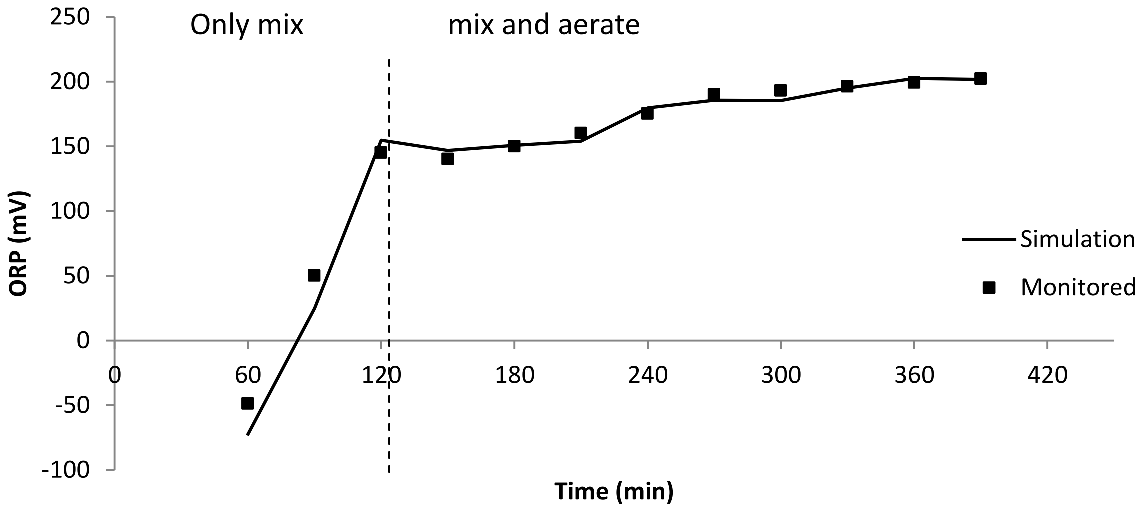


Figure 4-21 The comparison of simulated and experimented ORP profile for "only mix" and "mix and aerate" steps ofSBBR system with nutrients added pellets.

2. Second stage: ammonium removal

Eq. (4-6) is Nernst equation only considered ammonium for excess carbon sources in SND process (Lee, 2004). After phosphate removed completely, this equation can predict the ammonium in reactor.

$$E = a' + b' + c' \ln\left(\left[NH_4^+\right]\right) \ldots\ldots Eq.(4-6)$$

Table 4-8 shows the second stage Nernst equation modeling results of aerobic cases in immobilized SBBR systems with rebuilt WAS pellets. The simulated models indicate well correlation coefficient (R^2) value of 0.96. Figure 4-22 shows the model simulation and the experimental data, which shows a close correlation in second stage reaction.

Table 4-8. Results of the second stage Nernst equation for "mix and aerate" step in SBBR systems with rebuilt WAS pellets.

	Nernst equation model constants			
SBBR with Added nutrient pellets	a	b	c	R^2
Mix and aerate	-360.87	86.98	-38.38	0.96

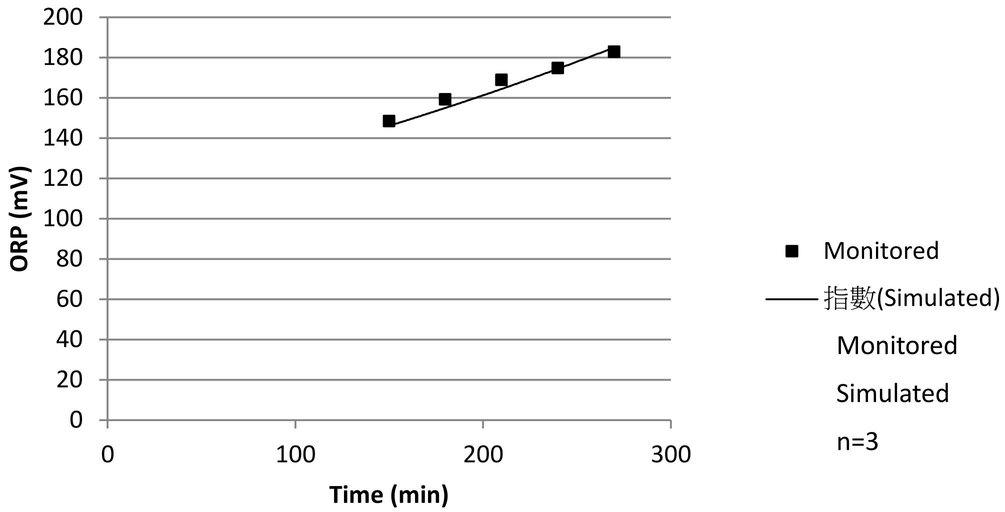


Figure 4-22 The comparison of simulated (second stage model) and experimented ORP profile for “mix and aerate” step of SBBR system with rebuilt WAS pellets

4.5.3Application SBBR SND system

In the SBBR-Nutrient system, according to the Table 4-7 and *Eq. (4-6),* when the ORP value is above 150 mV, (i.e., the reaction time of 150 min) the phosphate can be removed completely. This means the step 1 is finished, and then the reaction entering step 2. According to the Table 4-8 and *Eq. (4-6),* the ammonium can be removed completely when the ORP value above 200 mV, i.e., the SND reaction will be terminated. The sudden drop of pH value is also a good index to realize the end point of phosphate removal. In this study, the pH value is included in the Nernst model, therefore, it can be considered as one of the key parameters to control the end of the first stage reaction.

Figure 4-23 shows the comparison of simulated NH_4^+-N data and real NH_4^+-N data.

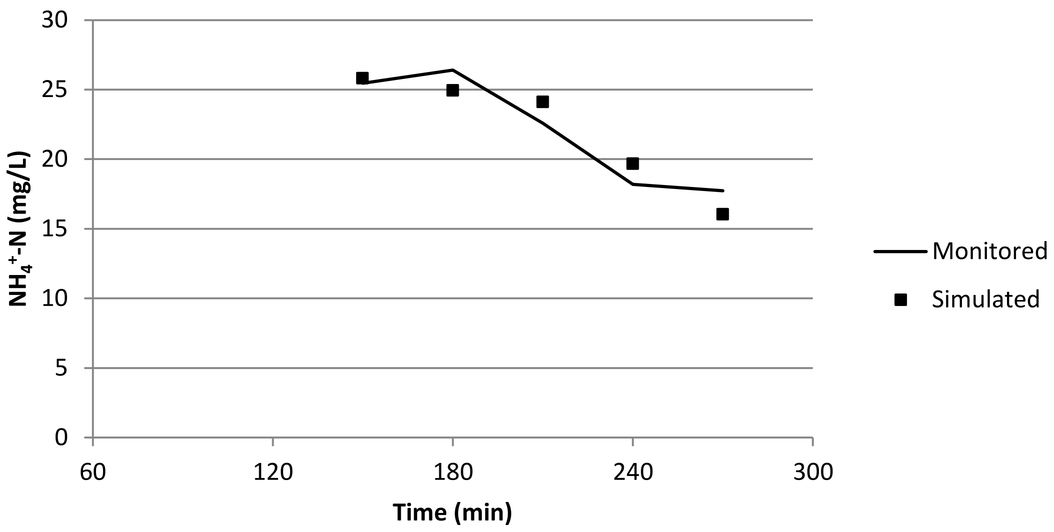


Figure 4-23 The comparison of the simulated ammonium and monitored ammonium value in Loading I.

CHAPTER 5 CONCLUSIONS AND SUGGESTIONS

5.1 Conclusions

In this study, the two systems build with raw pellets and nutrient pellets were tests. Apply the WAS to rebuilt the pellets, which was built for biofilm carrier. All the system was cultured the activated sludge under three loading stages and the added nutrient pellets were achieve more biomass immobilized in SND system. The following conclusions can be made:

1. The raw pellets can be made of WAS, leterite and chemical additive with an optimal formula of 50% of WAS according to DOE. The basic characteristics of rebuilt raw pellets with the water absorption of 41.75 %, bulk density 1.54 g/cm^3 and the compressive strength 11.3 kgf/cm^2.
2. The added nutrient pellets can be made of same optimal formula with raw pellets, and added 0.1M KH_2PO_4 and 0.1M KNO_3, and two compounds mixed together in order to replace the raw particles produced by adding water, the added nutrient was not impact on water

quality.

3. According to the batch tests data, the nutrient added could enhanced the biomass to immobilize, biomass was increased the SND efficiency (Raw 96% ＜ 98% Nutrient).
4. The two systems were cultured to steady state under three different loadings. The SBBR-Nutrient system was not completely removed the ammounium (effluent NH_4^+-N: below 1 mg/L), but the level was below 5 mg/L at 420 min.

5.2 Suggestions

1. The nutrient pellets can be adding higher concentrations to immobilize more biomass. Try to enhance the removal efficiency, but the nutrient concentrations could not impact in the water quality.
2. The added nutrient pellets enhanced wastewater treatment efficiency; thus, could be applied in the biological treatment plant and ecological engineering systems.

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