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Effects of Mixing HTS and FCC Wastwaters on the Quality of A/O Effluent

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Abstract

The effects of mixing of two kinds of wastewaters Hollow Titanium Silicate (HTS) zeolite and Fluid Catalytic Cracking (FCC containing QACs) through various tests in the percentage ratios of 0.50%, 0.65%%, 0.70%, 0.80%, 0.9%, 1.0%, 1.1% and 1.2% to reduce nitrogen species and COD by using A/O system are studied. The domestication has carried out under the following conditions: pool A's DO maintained at 0.5 mg/L while pool O's DO controlled within 2 - 4 mg/L at room temperature. The C/N ratio was regulated to 4.0:1.0 by glucose addition and the hydraulic residence time (HRT) was 70 h. Pool O's pH was controlled at 7.5 - 8.5 by adding NaOH, the wastewater's reflux ratio was controlled to R = 500%, and the sludge reflux ratio was r = 100%. During the experiment, the inlet and outlet water's both COD, ammonia-N and TN as well as SV_{30}, MLSS and SVI were tested and analyzed. The results showed that during process with amounts of mixed HTS wastewater to FCC wastewater of 0.80%, 0.90%, 1.0%, 1.1% to 1.2%, ammonia-N removal was effective. The COD concentration was also reduced to less than 60 mg/L. However, TN elimination did not attain discharge standard. Thereafter, by adjusting the C/N ratio at 5.5:1.0 under temperature at 25˚C, the ammonia-N concentration was 3 - 4 mg/L, the TN concentration was reduced to 37 mg/L and COD concentration was 58 mg/L. With the inlet water COD concentration of 1815 mg/L (including the COD concentration of glucose), and ammonia-N concentration of 330 mg/L, HTS wastewater mixed with FCC at ratio of 1.0%, C/N ratio of 5.5:1.0, temperature between 25˚C - 37˚C, the effluent's ammonia-N concentration remained to 3 mg/L, TN concentration lower than 40 mg/L and COD concentration less than 60 mg/L showing thus the process achieved the nitrogen species removal standard. However, when the temperature of A/O system wastewater was increased to 40˚C or became lower than 25˚C, the sludge was lost and the whole system was destroyed.

Keywords

Ammonia-N, Anoxic/Oxic A/O System, Hollow Titanium Silicate, Fluid Catalytic Cracking, Total Nitrogen

1. Introduction

Nitrogen (N) element is a limiting factor in ecosystems essential for growth of living microorganisms and plants [1]. Ammonia-nitrogen (NH_3-N) is one of the most important nitrogen forms. Since the 1980s, the rapid economic development [2] has increased China's environmental problems due to the spread of excess reactive Nitrogen (Nr). Problems of excess ammonia-nitrogen discharge involved eutrophication, oxygen depletion into water bodies, extinction of some aquatic species, disturb the entire natural food chain, soil acidification, deposition of nitrogen, and atmospheric pollution etc. [3] [4]. Moreover, excess of nitrogenous compounds such as nitrite and nitrate etc. in drinking water results to for serious health problems [5]. This conducted to changing ecosystem functions and degrading ecosystem services in China [6]. In order to pursue the Chinese Government policies of national development of industrial catalysts' production and the improvement of environmental protection, it is a great challenge to resolve the environmental problems caused by these contaminant wastewaters. Therefore, control of ammonia-nitrogen (NH_3-N or NH_4-N) become one of the most important nutrients in wastewater to be controlled before any discharge in natural receiving watercourse. Thus, the reduction of Nr emissions to the environment plus the subsequent Nr pollution are becoming a growing challenge not only in China [7] but worldwide [8]. Therefore, gradual implementation of restricted regulations has applied depending to the type of receiving watercourse reservoirs (lake, river, etc.) of the local government considered. Progressive changes have been realized from the previously China's integrated wastewater discharge standard GB 8978-1996 with discharge concentration of up to 100 mg/L for NH_4^+-N to more strict discharge of 40 mg/L NH_4^+-N for large-scale factories and 70 mg/L for medium-size factories by the Discharge Standard of Water Pollutant for Ammonia Industry (GB 13458-2001) [9]. Then a discharge standard with 25 mg/L NH_4^+-N effluent limit is under studied. Actually, the new industrial wastewater's ammonia-N discharge standards must be less than 5 mg/L. In the same time, the total nitrogen (TN) removal becomes also a major challenge for the municipal WWTP to meet the new standards. In fact, this was due to the overall low nitrogen removal efficiency of the existing municipal WWTPs in China. The discharge standard of TN < 15 mg/L was amended in 2017 by Water Pollution Prevention and Control Law of the People's Republic of China which started on January 1, 2018. These limit values are actually close to the most stringent standards in some developed countries.

In order to achieve these challenges, several studies have revealed the complexity of the biological removal of the ammonia-N and total ammonia (TN) from these wastewaters [10]-[13]. Ren *et al.* [14] and Rosa *et al.* [15], have reported that the SBR nitrification and denitrification, biofilms and other biological treatment processes do not satisfy completely nitrogen species elimination. Moreover, in our previous studies with SBR and A/O biological processes, it was found that the TN content is particularly difficult to remove from such kinds of wastewater [16] [17].

Fluid catalytic cracking (FCC) is a major unit in oil refineries process worldwide. It converts feedstocks such as shale oil, tar sands oil, and cooker gas oils into lighter, high-value products by "cracking" C-C bonds via catalysts' operation. These feedstocks may contain some high levels of organic nitrogen compounds such as indole, carbazole, pyridine, and quinolone etc. which may produce ammonia and cyanide during FCC units operations. The more asphaltic the oil, the higher the nitrogen content found in generated FCC wastewaters.

FCC and HTS zeolite processes produce two kinds of wastewaters from crude oil refining. These wastewaters contain high complex nitrogen and organic contents that are not environmentally friendly [11]-[17].

In this study, the A/O process was used to assess a technical method consisting of gradually mixing FCC wastewater and HTS wastewater through various tests with ratios between 0.5% to 1.2% along with C/N and temperature influences on the process.

2. Material and Methods

2.1. Apparatus

The A/O reactor is that one used in our previous article I. L. Ali *et al.*, 2024 [7] (see Figure 1).

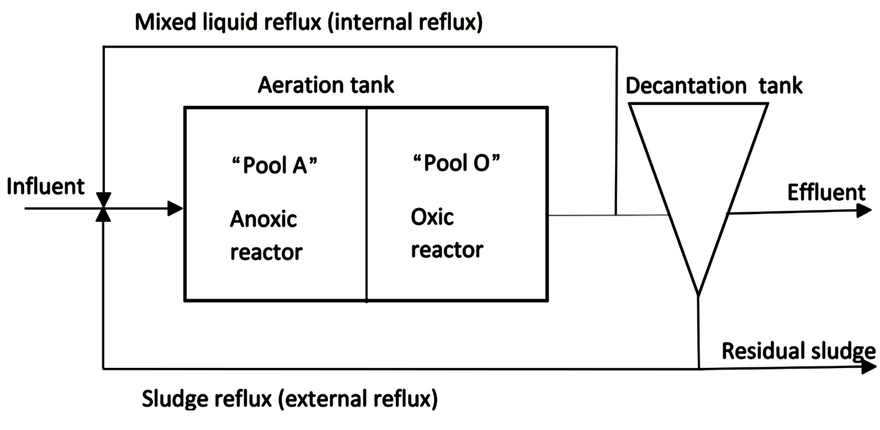

Figure 1. Flow diagram of A/O process.

2.2. Experimental Procedures

During all experiments, the inlet and effluent's water both COD, ammonia-N and TN as well as SV_{30}, MLSS and SVI are tested and analyzed.

The two mixed wastewaters are HTS wastewater (COD~3300 mg/L and ammonia-nitrogen~80 mg/L, and TN~165 mg/L) [8] and FCC (COD~1320 mg/L, ammonia-nitrogen and TN~330 mg/L each one) wastewater. At the beginning, HTS wastewater was added to FCC wastewater by 1.0% ratio in order to domesticate the sludge. Domestication conditions are glucose addition at C/N ratio of 4.0:1.0, the hydraulic residence time HRT of this stage of the A/O process is set to 70 h, pool A's DO adjust within 0.5 mg/L through the electric mixer, pool O's DO control within 2 - 4 mg/L through the aeration device. Pool O's pH is kept at 7.5 - 8.5 by adding NaOH. By adjusting the operating parameters of the peristaltic pump, the reflux ratio of wastewater is controlled to R = 500%, and the sludge reflux ratio is r = 100%. The temperature is maintained at room temperature in experimental conditions. The experimental period lasted 8 days.

But, due to the failure of the above domestication, the amount of HTS wastewater into FCC wastewater has been reduced to 0.5%. This test duration lasted 20 days.

According to the success of domestication, the mixing ratios were therefore increased to 0.65%, 0.75%, 0.80%, 0.90%, 1.0%, 1.1% and 1.2% with previous other operating conditions.

At the end of the test with 1.2% ratio, the effluent's COD, ammonia-N and TN meet the standard. But when continuing the operation, TN cannot meet the removal standard any more, and at this stage the consideration may be that the carbon source is insufficient. Thus, the C/N ratio of inlet water was increased to 5.5:1.0, with the ratio of 1.0% of HTS wastewater into FCC wastewater.

Then, effects of temperature on mixed wastewater in A/O system treatment was also assessed. Considering that the inlet temperature of the FCC wastewater is about 40˚C, and since the experiment was conducted at room temperature, the wastewater temperature is then increased to 30˚C and 35˚C, and the amount of HTS wastewater added to the FCC wastewater is 1.0%. Moreover, effects of temperature of 37˚C and 40˚C on treatment were also tested. On the basis of the previous experiment, considering that the temperature of the original wastewater can be higher than 40˚C, effluent's COD, ammonia-N and TN concentrations can be discharged. Therefore, in the next experiment, the temperature of wastewater can be gradually raised, and the effect of raising temperature on the treatment should be investigated.

3. Results and Discussion

3.1. Sludge Domestication

Sludge from an FCC catalyst petrochemical enterprise produced wastewater has been domesticated during one month from end of July in order to treat a FCC catalyst company wastewater. Thereafter, treatment of the non-diluted a catalyst company catalyst FCC wastewater has been carried out during the A/O process by adjusting the pH value and dissolved oxygen DO parameters so that the concentration of ammonia-N in the effluent was reduced to 3 mg/L - 4 mg/L, the TN removal rate was about 50%, and COD concentration was below 50 mg/L. It is thus considered that this stage of work is effective. In fact, at the beginning of domestication, sludge's microorganisms need certain time to adapt; in general, some of them manifest as a result of changes in wastewater parameters such as nitrogen and COD contents. Then the remaining microorganisms slowly adapt to new environment by using available components of wastewater so that resulting in parameters modification whom analysis can tell whether the wastewater satisfy discharge conditions.

In this stage, the lack of denitrification bacteria in the sludge taken from a petrochemical catalyst enterprise, which caused only the wastewater's TN to drop at 50%, therefore these bacteria still need to be domesticated.

3.2. Effects of Mixing 1.0% HTS Wastewater

The treatment efficiency and the performance of sludge of 1.0% HTS wastewater mixed to FCC wastewater under above experimental conditions are shown in Table 1.

Table 1. Effects of adding 1.0% HTS wastewater to FCC wastewater on effluent parameters.

Serial No.	1	2	3	4	5	6	7	8
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L) (mg/L)	31	37	42	50	56	76	85	87
Ammonia-N removal rate (mg/L) (%)	91	89	87	85	83	77	74	74
Influent TN (mg/L)	330	330	330	330	330	330	330	330
Effluent TN (mg/L)	170	175	180	180	181	186	187	187
TN removal rate (%)	48	47	45	45	45	44	43	43
Influent COD (mg/L)	1320	1320	1320	1320	1320	1320	1320	1320
Effluent COD (mg/L)	46	50	55	55	53	55	56	59
COD removal rate (%)	97	96	96	96	96	96	96	96
SV_{30} (%)	90		89		90		90	
MLSS (mg/L)	7067		7012		7031		7014	
SVI (mL/g)	127		127		128		128	

As it can be seen in Table 1, after adding 1.0% of HTS wastewater to FCC wastewater, the concentration of ammonia-N in the inlet water was basically maintained at 330 mg/L. Through the domestication stage, the concentration of ammonia-N in the effluent began to increase gradually. On Serial No. 1, the ammonia-N concentration in the A/O system rose to 31 mg/L (raw HTS wastewater, after treatment, has its effluent ammonia-N concentration of 3 mg/L - 4 mg/L), and by the fourth day, Serial No. 4, the concentration of ammonia-N in the effluent rose to 50 mg/L. The domestication experiment continued, and by Serial No. 8, the concentration of ammonia-N in the effluent had already reached to 87 mg/L. That is to say, the addition of 1.0% content of HTS wastewater leads to effluent 91% removal rate of ammonia-N at the beginning, then it down gradually to 85% until

74% at Serial No. 8, respectively. It is concluded that HTS wastewater has great toxicity to nitrifying bacteria which accumulates in sludge and hinders nitrification reaction. But the effect on effluent's COD is very small with the effluent's COD concentration remaining lower than 60 mg/L. In view of this situation, it is considering to reduce the amount of HTS wastewater to 0.50% for the next domestication process.

3.3. Effects of Mixing 0.50% HTS Wastewater to FCC Wastewater

In the early stage of adding 1.0% HTS wastewater, the domestication effect was not good; the main consideration is how to make adapt microorganisms to the toxicity of HTS wastewater. Therefore, Since the experimental work, the amount of HTS wastewater into FCC wastewater has been reduced to 0.50%, thus the parameters of the removal contaminants from activated sludge such as ammonia-N (experimental conditions are the same as Table 1) are tested. Situation of the domestication period from serials 1 to 10 is shown in Table 2.

Table 2. Effect of adding 0.50% HTS wastewater to FCC wastewater on effluent parameters.

Serial No.	1	2	3	4	5	6	7	8	9	10
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	83	80	81	78	73	64	55	45	35	25
Ammonia-N removal rate (%)	75	76	75	76	78	81	83	86	89	92
Influent TN (mg/L)	330	330	330	330	330	330	330	330	330	330
Effluent TN (mg/L)	180	180	178	178	181	183	183	184	180	180
TN removal rate (%)	45	45	46	46	45	45	45	44	45	45
Influent COD (mg/L)	1320	1320	1320	1320	1320	1320	1320	1320	1320	1320
Effluent COD (mg/L)	58	60	59	57	52	58	53	53	58	53
COD removal rate (%)	96	96	96	96	96	96	96		96	96
SV_{30} (%)	90		91		90		90		90	
MLSS (mg/L)	7042		7089		7044		7037		7032	
SVI (mL/g)	128		128		128		128		128	
Serial No.	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330	330	330	
Effluent ammonia-N (mg/L)	20	22	23	23	21	15	8	3	3	
Ammonia-N removal rate (%)	94	93	93	93	93	95	98	99	99	

Continued

Influent TN (mg/L)	330	330	330	330	330	330	330	330	330
Effluent TN (mg/L)	178	175	170	170	168	170	167	165	160
TN removal rate (%)	46	47	48	48	49	48	49	50	52
Influent COD (mg/L)	1320	1320	1320	1320	1320	1320	1320	1320	1320
Effluent COD (mg/L)	52	59	59	57	59	59	59	59	59
COD removal rate (%)	96	96	96	96	96	96	96	96	96
SV_{30} (%)	90		90		90		90		90
MLSS (mg/L)	7049		7079		7049		7011		7029
SVI (mL/g)	128		127		128		128		128

As it can be seen from **Table 2**, in the initial three serials (No. 1, No. 2, and No. 3), the concentration of ammonia-N of the inlet water from 330 mg/L, decreased in the effluent to 83 mg/L, 81 mg/L and 80 mg/L respectively, and ammonia-N removal rate is basically about 75%.

From serials 4 to 7, it was found that the concentration of ammonia-N in the effluent was decreasing every day, from the concentration of ammonia-N in the effluent to 78 mg/L, 73 mg/L, 64 mg/L and 55 mg/L, respectively. The removal rate of ammonia-N also increased from 76% to 83%, showing that the sludge domestication played a role.

From serials 8 to 10, under continued domestication, the concentration of ammonia-N in the effluent has been declining. By serial 8, the concentration of ammonia-N in the effluent was 45 mg/L, and over the next three (3) serials (9, 10, 11), the concentration of ammonia-N in the effluent decreased gradually to 35 mg/L, 25 mg/L and 20 mg/L, respectively. The removal rate of ammonia-N also increased from 86% to 94%. It is shown that, although the domestication time of 0.50% HTS wastewater to FCC wastewater is no longer than in A/O system, the concentration of ammonia-N in the effluent is decreasing every day, which indicates that the microorganisms are suitable for added 0.50% of HTS wastewater.

From serial 16, microorganisms began to adapt, and the concentration of ammonia-N in the effluent has been declining. By serials 17, 18 and 19, the concentrations of ammonia-nitrogen were 8 mg/L, 3 mg/L and 3 mg/L, respectively. This indicates that addition of 0.50% HTS wastewater to FCC wastewater into A/O system, after domestication, the resulting ammonia-N concentration in the effluent can be discharged.

From **Table 2**, it can also be found that by adding 0.50% HTS wastewater in FCC wastewater, after treatment by A/O system, the removal rate of TN reached about 50%, indicating that the removal of TN is also in a favorable direction. At this stage, the concentration of COD in the effluent was basically stable below 60 mg/L.

3.4. Effects of Mixing 0.65% HTS Wastewater to FCC Wastewater

In the case of 0.50% HTS wastewater, ammonia-N removal from the effluent can meet the standard; After a while, the amount of HTS wastewater into FCC wastewater was increased to 0.65% in order to observe the activated sludge removal efficiency for ammonia-N and other pollutants parameters.

Experimental data are shown in below Table 3. Between serial No. 1 and No. 6, the ammonia-N concentration of the system effluent can reach standards; this indicates that by using 0.65% HTS wastewater into FCC wastewater within the A/O system, through domestication, the concentration of ammonia-N in the effluent (99%) meets the discharge standards. From Table 3, it can also be found that by adding 0.65% HTS wastewater into FCC wastewater, after the treatment by A/O system, the removal rate of TN was increasing slightly, and by serial 6, it reached about 58%, indicating that the TN removal is also in a favorable direction. At this stage, the concentration of COD in the effluent is basically stable below 60 mg/L.

Table 3. Effect of the addition of 0.65% HTS wastewater to FCC wastewater.

Serials	1	2	3	4	5	6
Influent ammonia-N (mg/L)	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	4	4	3	3	3	3
Ammonia-N removal rate (%)	99	99	99	99	99	99
Influent TN (mg/L)	330	330	330	330	330	330
Effluent TN (mg/L)	155	150	148	145	142	138
TN removal rate (%)	53	55	55	56	57	58
Influent COD (mg/L)	1320	1320	1320	1320	1320	1320
Effluent COD (mg/L)	57	59	59	57	59	59
COD removal rate (%)	96	96	96	96	96	96
SV_{30} (%)	90		90		90	
MLSS (mg/L)	7032		7019		7024	
SVI (mL/g)	128		128		128	

3.5. Effects of Mixing 0.75% HTS Wastewater to FCC Wastewater

In the case of adding 0.65% HTS wastewater, ammonia-N from the effluent can meet the standards; afterwards, the amount of HTS wastewater into FCC wastewater was increased to 0.75%, thus activated sludge's effect on the removal of ammonia-N and other pollutants parameters are observed.

Experimental data as shown in Table 4, during the period from serial 1 to serial 8, the system of ammonia-N concentration was reached standards, indicating that after the addition of 0.75% HTS wastewater into FCC wastewater in the A/O system, the domestication and the concentration of ammonia-N in the effluent have met the standards of discharge.

Table 4. Effects of adding 0.75% HTS wastewater to FCC wastewater on effluent parameters.

Dates	1	2	3	4	5	6	7	8
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	3	3	4	4	4	5	5	5
Ammonia-N removal rate (%)	99	99	99	99	99	99	99	99
Influent TN (mg/L)	330	330	330	330	330	330	330	330
Effluent TN (mg/L)	138	136	136	135	133	134	132	133
TN removal rate (%)	58	59	59	59	60	59	60	60
Influent COD (mg/L)	1320	1320	1320	1320	1320	1320	1320	1320
Effluent COD (mg/L)	59	58	59	57	57	58	57	59
COD removal rate (%)	96	96	96	96	96	96	96	96
SV_{30} (%)	90		90		90		90	
MLSS (mg/L)	7043		7039		7034		7054	
SVI (mL/g)	128		128		128		128	

From **Table 4**, it can also be found that by addition of 0.75% HTS wastewater into FCC wastewater, after treatment by A/O system, the removal rate of TN reached about 60% by serial 8, indicating that the removal rate of TN is not obvious. At this stage, the concentration of COD in the effluent is basically stable below 60 mg/L. In the next experiment, the amount of HTS will be further increased, and the concentration of TN into the effluent can be further reduced by adjusting various parameters.

3.6. Effects of Mixing 0.80% HTS Wastewater

From serial No. 1, the amount of HTS wastewater added into FCC wastewater was increased to 0.80%, and the removal of pollutants parameters such as ammonia-N from activated sludge is observed, and the experimental data are shown in **Table 5**.

Table 5. Effect of adding 0.80% HTS wastewater to FCC wastewater on effluent parameters.

Date	1	2	3	4
Influent ammonia-N (mg/L)	330	330	330	330
Effluent ammonia-N (mg/L)	5	6	5	3
Ammonia-N removal rate (%)	98	98	98	99
Influent TN (mg/L)	330	330	330	330
Effluent TN (mg/L)	133	136	140	150
TN removal rate (%)	60	59	58	55

Continued

Influent COD (mg/L)	1320	1320	1320	1320
Effluent COD (mg/L)	52	56	59	57
COD removal rate (%)	96	96	96	96
SV_{30} (%)	80		80	
MLSS (mg/L)	6979		6969	
SVI (mL/g)	115		115	

As it can be found from Table 5, on serials No. 2, No. 3 and No. 4, the concentrations of ammonia-N into the effluent were 6 mg/L, 5 mg/L and 3 mg/L, respectively. This is indicating that, when mixing 0.80% HTS wastewater into FCC wastewater in A/O system, the concentration of ammonia-N in the effluent can be discharged. At this point, the concentration of ammonia-N in the effluent dropped to 5 mg/L, even to 3 mg/L. The concentration of COD in the effluent is basically lower than that of 60 mg/L. The TN concentration decreased from 330 mg/L to 150 mg/L, and its removal rate was 55%.

3.7. Effects of Mixing 0.90% HTS Wastewater into FCC Wastewater on Effluent Parameters

After adding 0.80% HTS wastewater, under the situation of effective discharge of effluent's ammonia-N concentration, from serial 1, the amount of HTS wastewater into FCC wastewater increased to 0.90%, and the status of the removal of ammonia-N and other contaminants' parameters by activated sludge was observed and experimental results are shown in Table 6.

Table 6. Effect of adding 0.90% HTS wastewater to FCC wastewater on effluent parameters.

Serial	1	2	3	4
Influent ammonia-N (mg/L)	330	330	330	330
Effluent ammonia-N (mg/L)	5	4	3	3
Ammonia-N removal rate (%)	99	99	99	99
Influent TN (mg/L)	330	330	330	330
Effluent TN (mg/L)	145	150	148	145
TN removal rate (%)	56	55	55	56
Influent COD (mg/L)	1320	1320	1320	1320
Effluent COD (mg/L)	57	59	59	57
COD removal rate (%)	96	96	96	96
SV_{30} (%)	80		80	
MLSS (mg/L)	6932		6919	
SVI (mL/g)	115		116	

As it can be found from Table 6, during with the four (4) serials from No. 1 to No. 4, the concentration of ammonia-N of the system decreased from 5 mg/L to 3 mg/L, and meet the discharge standard conditions. It is indicated that the domestication of 0.90% HTS wastewater into FCC wastewater in A/O system does not affect the degradation of ammonia-N concentration. The concentration of COD in the effluent is basically lower than 60 mg/L. The removal rate of TN from the effluent is high around 55%. The removal rate of TN is basically similar to that of 0.80% HTS wastewater.

3.8. Effects of Mixing 1.0% HTS Wastewater into FCC Wastewater on Effluent Parameters

After adding 0.90% HTS wastewater into FCC wastewater, the concentration of ammonia-N in the effluent was met emission standards; from serial 1, the amount of HTS wastewater into FCC wastewater was increased to 1.0%, and the removal efficiency of ammonia-N and other pollutants parameters by activated sludge as well as the experimental results are shown in Table 7.

Table 7. Effect of adding 1.0% HTS wastewater into FCC wastewater on effluent parameters.

Serial	1	2	3	4	5	6	7	8
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	30	25	24	20	14	8	5	5
Ammonia-N removal rate (%)	91	92	93	94	96	98	98	98
Influent TN (mg/L)	330	330	330	330	330	330	330	330
Effluent TN (mg/L)	140	136	136	136	143	136	136	135
TN removal rate (%)	58	59	59	59	58	59	59	59
Influent COD (mg/L)	1320	1320	1320	1320	1320	1320	1320	1320
Effluent COD (mg/L)	58	58	58	57	57	59	58	59
COD removal rate (%)	96	96	96	96	96	96	96	96
SV_{30} (%)	80		80		79		79	
MLSS (mg/L)	6943		6939		6934		6954	
SVI (mL/g)	115		115		114		114	

As it can be found from Table 7, at the beginning of serial No. 1, the concentration of ammonia-N in the system rose to 30 mg/L, indicating that this concentration of bacteria could not be adapted immediately. After five days (serials No. 1, No. 2, No. 3, No. 4, and No. 5) of domestication experiments, the concentration of ammonia-N in the effluent gradually decreased from the 30 mg/L, 25 mg/L, 24 mg/L, 20 mg/L to 14 mg/L, respectively, indicating that the concentration of ammonia-N in the effluent tends to decrease, meaning that this domestication is effective. Domestication to the sixth day (serial 6), the concentration of ammonia-

N in the effluent continued to decline to 8 mg/L. By the seventh and eighth days (serials 7 and 8), the concentration of ammonia-N in the effluent was reduced to 5 mg/L. It is indicated that by adding 1.0% HTS wastewater into FCC wastewater in A/O system, after domestication, the concentration of ammonia-N in effluent can be discharged.

From Table 7, at the same time, the concentration of COD in the effluent is basically lower than 60 mg/L. The removal rate of TN from effluent was 59%, and the TN concentration from 330 mg/L decreased to 135 mg/L.

3.9. Effects of Mixed 1.1% HTS Wastewater to FCC Wastewater on Effluent Parameters

By the addition of 1.0% HTS wastewater, effluent's ammonia-N can meet the standards; from serial No. 1, the amount of HTS wastewater into FCC wastewater was increased to 1.1% in order to cope with the actual treatment device in the operation of the inlet water load fluctuation changes. At the same time, the C/N ratio of intake water was increased from 4.0:1.0 to 5.0:1.0, mainly considering the deficiency of carbon source when removing TN. The experimental data are shown in Table 8.

Table 8. Effect of adding 1.1% HTS wastewater to FCC wastewater on effluent parameters.

Date	10.25	10.26	10.27	10.28	10.29	10.30	10.31
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	20	19	15	10	8	5	3
Ammonia-N removal rate (%)	94	94	95	97	97	98	99
Influent TN (mg/L)	330	330	330	330	330	330	330
Effluent TN (mg/L)	136	136	136	127	125	120	117
TN removal rate (%)	59	59	59	62	62	64	65
Influent COD (mg/L)	1650	1650	1650	1650	1650	1650	1650
Effluent COD (mg/L)	60	58	58	57	57	59	58
COD removal rate (%)	96	96	96	96	96	96	96
SV_{30} (%)	80		80		79		79
MLSS (mg/L)	6973		6979		6974		6974
SVI (mL/g)	115		115		113		113

As it can be found from Table 8, from serial No. 1, in the effluent concentration of ammonia-N in the system was 20 mg/L. By continuing domestication, the concentration of ammonia-N in the effluent decreased gradually. Between serials 5, 6 and 7, the concentration of ammonia-N in the effluent eventually dropped to 3 mg/L. It is indicated that 1.1% HTS wastewater into FCC wastewater in A/O system, after domestication, the concentration of ammonia-N in the effluent can be discharged.

From Table 8, it can also be found that 1.1% of HTS wastewater into FCC wastewater, the C/N ratio increased to 5.0:1.0, after A/O system treatment, the concentration of COD in the effluent was basically lower than 60 mg/L. In addition, it was found that the removal rate of TN also improved, and by serial 7, the removal rate of TN reached 65%, indeed the TN concentration of inlet water from 330 mg/L fell to 117 mg/L in the effluent.

3.10. Effects of Mixing 1.2% HTS Wastewater

After adding 1.1% HTS wastewater, and when the effluent ammonia-N met the standard, the amount of HTS wastewater added to FCC wastewater was increased to 1.2% from serial 1, in order to cope with the fluctuation of the inlet water load during the operation of the actual treatment unit, experimental data are shown in Table 9.

Table 9. Effect of adding 1.2% HTS wastewater to FCC wastewater on effluent parameters.

Serial No.	1	2	3	4	5	6	7
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	15	10	8	5	5	3	3
Ammonia-N removal rate (%)	95	97	97	98	98	99	99
Influent TN (mg/L)	330	330	330	330	330	330	330
Effluent TN (mg/L)	100	90	80	70	65	63	64
TN removal rate (%)	70	73	76	79	80	81	81
Influent COD (mg/L)	1650	1650	1650	1650	1650	1650	1650
Effluent COD (mg/L)	60	60	59	57	58	59	59
COD removal rate (%)	96	96	96	96	96	96	96
SV_{30} (%)	79		80		80		79
MLSS (mg/L)	7012		6989		7012		6984
SVI (mL/g)	113		114		114		113

From Table 9, it can be seen that in the first day (Serial No. 1) of 1.2% HTS wastewater addition into FCC wastewater, the concentration of ammonia-N in the effluent was 15 mg/L. Since then, after three days of domestication, the effluent ammonia-N concentration decreased gradually to reach effluent concentration of 5 mg/L up to 3 mg/L (Serials No. 5, No. 6, and No. 7). This is indicating that the A/O system experimental device's sludge has been adapted to the increasing amount of HTS wastewater.

From Table 9, it can also be found that addition of 1.2% of HTS wastewater in FCC wastewater, by increasing the C/N ratio to 5.0:1.0, after the A/O system treatment, the concentration of COD in the effluent is basically lower than 60 mg/L. The removal rate of TN has been significantly improved, the TN removal rate has reached 81% by Serials No. 7; meanwhile from TN concentration in the inlet water of 330 mg/L, the effluent's TN concentration has dropped to 64 mg/L. This shows that the removal rate of TN can be further improved.

4. Effects of Inlet Water's C/N Ratio on TN Removal Rate

After adding 1.2% HTS wastewater, the effluent's ammonia-N can meet the standard, but when the TN cannot meet the standard. The consideration may be due to the insufficiency of carbon source which can impact the TN to not be reduced. From the first day (serial No. 1), the C/N ratio of inlet water was first increased to 5.5:1.0, and then was added the amount of 1.0% HTS wastewater into FCC wastewater, the experimental data are shown in **Table 10**.

Table 10. Effect of adding to FCC + 1.0% HTS wastewater on effluent parameters by C/N ratio 5.5:1.0.

Serial No.	1	2	3	4	5	6	7	8
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	3	3	3	5	3	3	3	3
Ammonia-N removal rate (%)	99	99	99	98	99	99	99	99
Influent TN (mg/L)	330	330	330	330	330	330	330	330
Effluent TN (mg/L)	60	57	60	55	53	51	39	37
TN removal rate (%)	82	83	82	83	84	85	88	89
Influent COD (mg/L)	1815	1815	1815	1815	1815	1815	1815	1815
Effluent COD (mg/L)	60	58	59	57	60	58	60	58
COD removal rate (%)	96	96	96	96	97	97	97	97
SV_{30} (%)	69		70		69		69	
MLSS (mg/L)	6855		6989		6855		6865	
SVI (mL/g)	101		100		101		101	

From **Table 10**, it can be found that from serial No. 6, the lowest concentration of TN in the effluent was 51 mg/L, so it cannot be rejected. Considering that the ambient temperature was now low (winter started), the water's temperature can only reach about 16˚C, so this can significantly reduce the activity of microorganisms; however, when the temperature of the wastewater was increased to 25˚C, by serial No. 7 and serial No. 8, the TN concentration of the effluent dropped to 39 mg/L and 37 mg/L, respectively, therefore satisfying the national discharge standards.

The effect of different C/N ratios on TN removal is shown in **Figure 2**. By operating with the concentration of inlet water of 330 mg/L ammonia-N and the C/N ratio of 4.0:1.0, the TN concentration of outlet water was about 136 mg/L. Then the C/N ratio was increased to 5.0:1.0, and after one week of activated sludge's domestication, the concentration of TN in the outlet water decreased from 100 mg/L to about 65 mg/L. Thereafter, the C/N ratio was increased to 5.5:1.0, and after one week of activated sludge's domestication, the concentration of TN from the outlet water decreased from 65 mg/L to about 50 mg/L; then after increasing the temperature from 16˚C to 25˚C, the TN concentration of the effluent fell to 37 mg/L, so that the effluent meets the discharge standards. In summary, the C/N ratio has a great influence on the removal of TN. Finally, the C/N ratio of 5.5:1.0 was selected in this small test experiment. However, in actual industrial operation, the amount of activated sludge is large, and the resistance to fluctuation and impact are strong, so it can be adjusted according to the specific situation, which can further reduce C/N and the cost of chemicals.

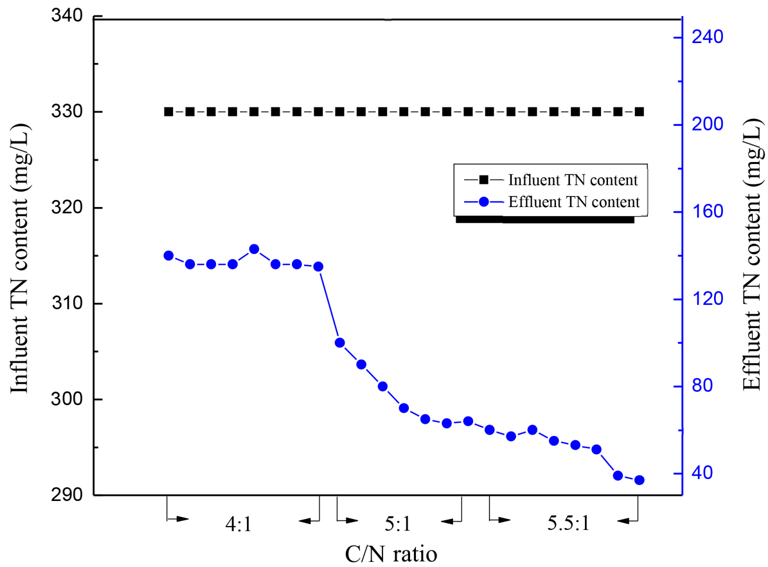

Figure 2. TN concentration variation under different C/N ratios.

5. Effect of Temperature on Mixed Wastewater in A/O System Treatment

5.1. Effect of Temperature from 30˚C to 35˚C on Wastewater Treatment

Given that the inlet temperature of the wastewater from the catalyst company is approximately 40˚C, then the wastewater temperature is further increased to 30˚C and 35˚C, and the amount of HTS wastewater added to the FCC wastewater is 1.0%, the obtained experimental data are presented in Table 11.

Table 11. Effects of increasing the temperature of wastewater of 30˚C and 35˚C on effluent parameters.

Serial No.	1 (30˚C)	2 (30˚C)	3 (35˚C)	4 (35˚C)
Influent ammonia-N (mg/L)	330	330	330	330
Effluent ammonia-N (mg/L)	3	3	3	3
Ammonia-N removal rate (%)	99	99	99	99

Continued

Influent TN (mg/L)	330	330	330	330
Effluent TN (mg/L)	39	37	37	37
TN removal rate (%)	88	89	89	89
Influent COD (mg/L)	1815	1815	1815	1815
Effluent COD (mg/L)	60	58	58	58
COD removal rate (%)	97	97	97	97
SV_{30} (%)	69		68	
MLSS (mg/L)	6865		6878	
SVI (mL/g)	101		99	

From Table 11, it can be found that after raising the temperature of wastewater to 30˚C and 35˚C, the TN in the effluent decreases to 37 mg/L, while the ammonia-N concentration drops to 3 mg/L and the concentration of COD reduces to 58 mg/L.

5.2. Effects of Wastewater's Temperatures of 37˚C and 40˚C on Mixed Wastewater Treatment

On the basis of the above experiment, considering that the temperature of the actual catalyst company wastewater can be higher than 40˚C, the effluent's COD, ammonia-N and TN concentrations can be discharged, therefore, in the next experiment, the temperature of wastewater can be gradually raised, and the effects of raising temperatures on the treatment of wastewater should be investigated. The specific inlet parameters, the treatment efficiency and performance of sludge are shown in Table 12.

Table 12. Effects of increasing the temperature of wastewater to 37˚C and 40˚C on effluent parameters.

Serial No. Temperatures	1 (37˚C)	2 (37˚C)	3 (38˚C)	4 (38˚C)	5 (39˚C)	6 (39˚C)	7 (40˚C)	8 (40˚C)
Influent ammonia-N (mg/L)	330	330	330	330	330	330	330	330
Effluent ammonia-N (mg/L)	4	4	8	10	20	35	50	60
Ammonia-N removal rate (%)	99	99	98	97	94	89	85	82
Influent TN (mg/L)	330	330	330	330	330	330	330	330
Effluent TN (mg/L)	39	40	45	60	88	102	150	154
TN removal rate (%)	88	88	86	82	73	69	55	53
Influent COD (mg/L)	1815	1815	1815	1815	1815	1815	1815	1815
Effluent COD (mg/L)	60	58	65	70	90	97	103	104
COD removal rate (%)	97	97	96	96	95	95	94	94
SV_{30} (%)	54		50		50		49	
MLSS (mg/L)	6565		6378		6157		5823	
SVI (mL/g)	82		78		81		93	

As it can be seen from Table 12, when the wastewater's temperature is increased to 37˚C, the concentration of ammonia-N in the effluent is 4 mg/L, the concentration of TN at 39 - 40 mg/L, COD concentration 60 - 58 mg/L, are within the discharge range. But when the temperature was increased to 38˚C, the TN from the effluent raised to 45 mg/L, and the effluent COD reached 65 mg/L. Then, as the temperature was gradually increased to 39˚C and 40˚C, the concentration of ammonia-N in the effluent increased from 20 mg/L to 60 mg/L, and the TN concentration increased from 88 mg/L to 154 mg/L while COD concentration raised from 90 mg/L to 104 mg/L. The concentration of pollutants parameters was obviously increased, and none of them can meet the standard emissions. In addition, it can be seen from the performance of sludge index SVI that the concentration of activated sludge in wastewater decreases gradually, and the disintegration of microorganisms and the turbidity of effluent can also be observed obviously. The amount of sludge is continuously decreasing. In order to see more clearly the effect of temperature on the performance of activated sludge treatment, the effluent's ammonia-N, TN and COD concentrations at different temperatures are plotted as shown in Figures 3-5. Under 37˚C, ammonia-N, TN and COD both can reach the standards. Therefore, when operating in the actual plant, the temperature of the wastewater should be cooled to 37˚C to ensure the activity of microorganisms in the activated sludge.

Therefore, when operating in the actual plant, the temperature of the wastewater should be cooled to at least 37˚C to ensure the activity of microorganisms in the activated sludge.

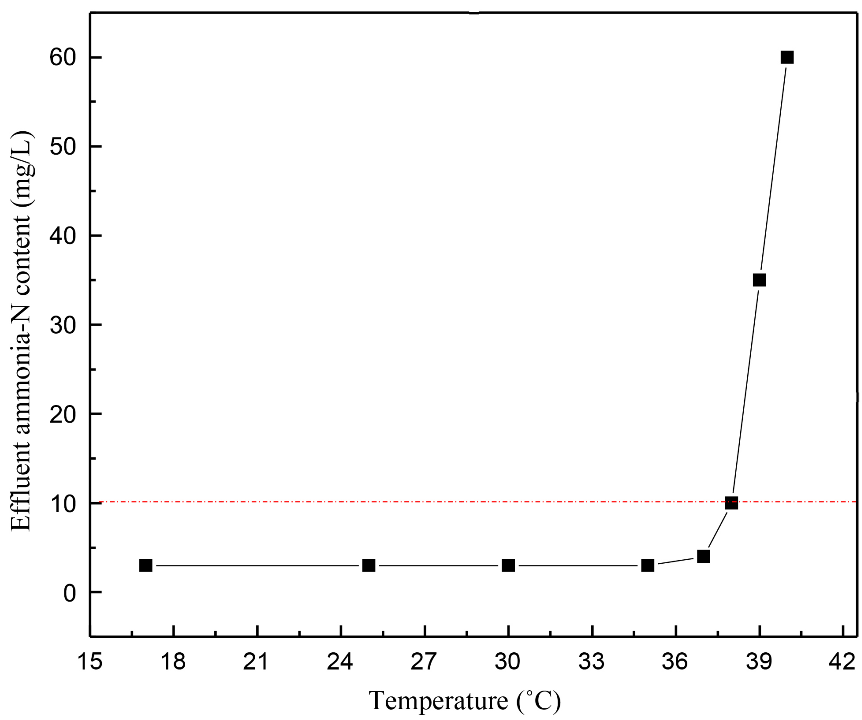

Figure 3. Ammonia-N concentration variation in effluent at different temperatures.

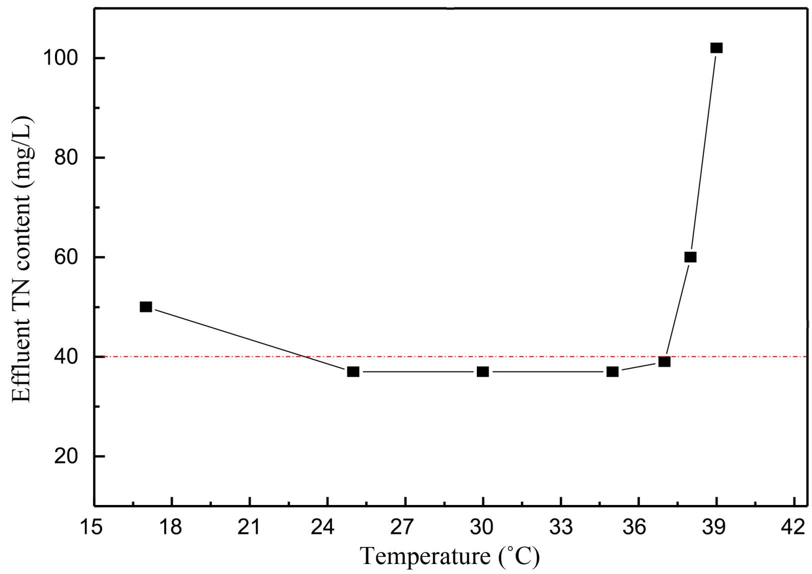

Figure 4. Curve of TN concentration variation in effluent at different temperatures.

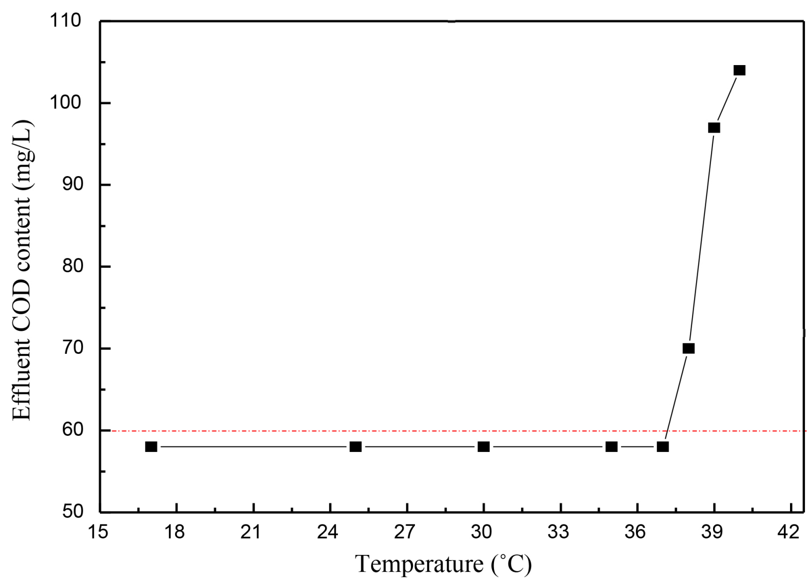

Figure 5. Curve of COD concentration variation in effluent at different temperatures.

6. GC-MS Component Analysis of Inlet and Outlet Wastewater of A/O System

In order to analyze the treatment process of wastewater theoretically, GC-MS was used to determine and to analyze the water quality of incoming and outgoing water as shown in Figure 6 and Figure 7.

From the GC-MS Map, it can be found that the organic matter in the water is mainly long chain alkanes, acids and 3,5-di-tert-butylphenol, as well as an undetected organic matter. Long chain alkanes, acids and 3,5-di-tert-butyl phenol are toxic to microorganisms, will be adsorbed on the microbial surface, are very difficult to degrade, but will cause the death of microorganisms.

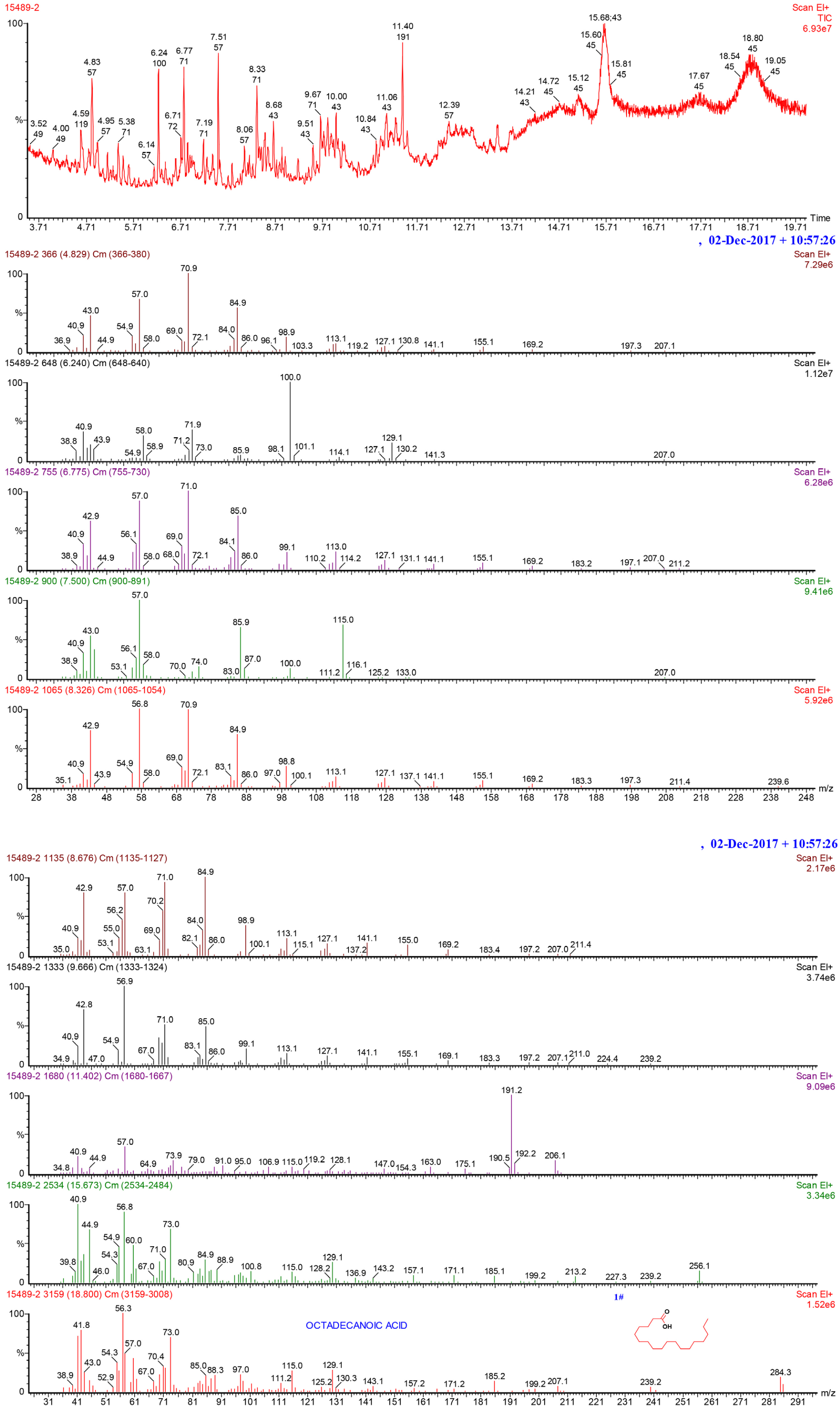

Figure 6. GC-MS spectrum analysis of inlet FCC + HTS.

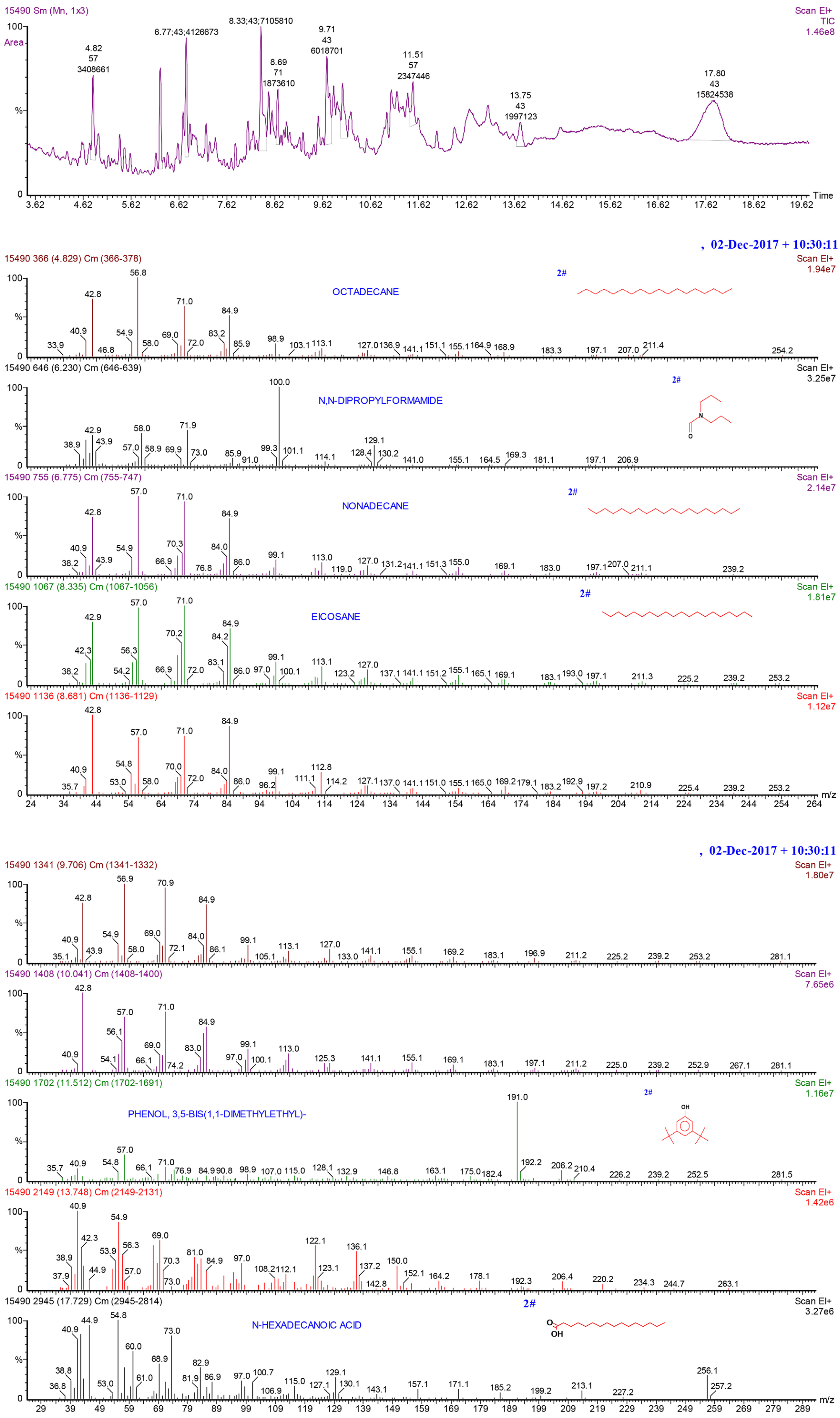

Figure 7. GC-MS spectrum analysis of effluent FCC + HTS.

From the GC-MS Map of the effluent, it can also be seen that some substances are partially decomposed or degraded, reducing the concentration of pollutants in the wastewater and therefore the difficulty of biochemical treatment. From **Figure 7**, it can also be found that certain organic matter not detected in the inlet water are not checked out in the effluent because they are likely biodegraded. These refractory organic compounds are basically derived from HTS catalyst wastewater, so the microbial method cannot be used alone to treat HTS wastewater.

7. Discussion

At the beginning of operation, when the HTS wastewater is mixed by 1.0% into FCC wastewater, the added HTS wastewater into FCC wastewater has total COD concentration of ~3300 mg/L and ammonia-N concentration of ~80 mg/L result to final mixed wastewater's ammonia-N concentration of 330 mg/L; and after 9 days of treatment, the concentration of ammonia-N in the effluent was 87 mg/L. It is considered that by adding 1.0% HTS wastewater to FCC wastewater, microorganisms do not adapt to the sudden addition of excessive HTS wastewater so that the activated sludge is affected and the nitrification bacteria undergoes toxic side effects conducting to TN removal rate of about 45%. This result is not satisfied the discharge standards. It is concluded that in the process of implementing the treatment of HTS wastewater mixed to FCC wastewater, it is necessary to gradually increase the amount of HTS wastewater so that microorganisms slowly adapt to the changing of environment's pollutants concentration [13]-[17]. This situation has been reported when the level of contents of wastewater concentration was high and toxic to sludge microorganisms as reported by Xiang, L. *et al.* [13]. Moreover, Ren *et al.* [14] has also observed that benzyl quaternary ammonia compounds (BAC) have significant inhibitory effects on activated sludge yield and its ability in substrate removal. In fact, their research observed that BAC can be degraded by activated sludge however the degradation needs a relative long time.

On the other side, the above AO operation's effect on COD removal rate was very small with COD concentration in the effluent lower than 60mg/L. The presence of refractory organic compounds found by GC-MS analysis are also toxic to microorganisms contributing to the poor removal efficiency as observed in the above operation and also reported by Ali *et al.,* [16]. Cheng *et al.* [18] has also reported that the activated sludge with zeolite can improve biodegradability of organic contaminates, and the COD removal rate reached about 90%, TOC removal rate of colature by 0.22 μm filter membrane was nearly 97% and TOC removal rate of colature by 0.45 μm filter membrane reached 92%. The sludge with zeolite can depredate further organic contaminants, which was difficult to degrade using organisms. This is a beneficial method to degrade some organic matter since it was found some recalcitrant organic compounds in such wastewaters in our previous studies [16] [17] which are very difficult to eliminate. It was also corroborated by Pan *et al.* [19], when treating High Strength Quaternary Ammonium Salt-Organic Wastewater. It needs to treat special organic wastewater with influent COD of 2500 - 3000 mg/L by A/O/O to achieve the total COD removal rate of 95%, and by oxidation and flocculation in different operating conditions the effluent COD can be reduced to below 80 mg/L. The total COD removal rate is up to 97%, meeting the national discharge standard in which COD limit value is determined between 100 to 500 mg/L depending to Class 1, 2, and 3 standards of GB8978-1996, GB 13458-2001 [9].

In order to adapt the activated sludge to the process toxicity, the amount of HTS wastewater into FCC wastewater was reduced to 0.5%. After 10 days of acclimatization, the concentration of ammonia-N in the inlet water decreased gradually to 10 mg/L; the effect is obvious with ammonia-N concentration of inlet water of 330 mg/L. The removal rate of TN was about 45% and the effluent's COD concentration was below 60 mg/L. On the basis of the above research, the amount of HTS wastewater mixed to FCC wastewater was changed from 0.80%, 0.90%, 1.0%, 1.1% to 1.2%, and through the acclimatization treatment of the A/O system, the inlet water's ammonia-N concentration of 330 mg/L was degraded to 5 mg/L, 3 mg/L, 5 mg/L, 3 mg/L and 3 mg/L, respectively. The effluent COD concentration was lower than 60 mg/L. This means that the amount of HTS wastewater into FCC wastewater must be controlled to about 1% meaning that the HTS proportion has a real toxicity on microorganisms. It's therefore a limiting factor which must be taken into account. On the other side, it was observed that the mixed wastewater C/N ratio of 4.0 was relatively small for denitrification. In order to increase the microbial growth energy, the C/N ratio was gradually increased to 5.0 and 5.5. When the mixed wastewater's C/N ratio was increased to 5.5:1.0 with HTS/FCC inlet water mixing ratio of 1.0% and the temperature at 16˚C as winter started (**Table 10**, serial No. 6), the effluent's ammonia-N concentration reached 3 mg/L while COD concentration was 58 mg/L and the TN concentration decreased to 51 mg/L. Thus, the effluent's ammonia-N and COD concentrations met rejection standards, but the TN concentration was consistently substandard. Thus, all conform to national discharge standards [9] except regards to TN for which China does not have a single nationwide limit discharge, as it varies by location and water quality standards. However, as temperature of 16˚C may affect microorganisms' activities therefore under the above conditions, by increasing the temperature to 25˚C, it was observed that the ammonia-N concentration was 3 mg/L - 4 mg/L, COD concentration was 58 mg/L and a decrease of the TN concentration to 37 mg/L (**Table 10**, serial No. 8). According to these experiments, it can be seen that the C/N is an important factor for nitrogen removal in this system which is impacted by the sensibility of the microorganisms toward temperature's variation. In fact, this was confirmed when the mixed wastewater was treated under same conditions of C/N ratio of 5.5:1.0, HTS wastewater mixed with FCC wastewater ratio of 1.0%, and water temperature's changes from 25˚C to 37˚C, with the inlet water COD concentration of 1815 mg/L (here including the COD concentration of glucose), and ammonia-N concentration of 330 mg/L. After treatment, the effluent's ammonia-N concentration decreased to 3 mg/L, COD concentration was less than 60 mg/L and the TN concentration was lower than 40 mg/L therefore respecting the discharge standards. However, when the temperature of A/O system wastewater was increased to 40˚C in this experimental device, the sludge was lost and the whole system was destroyed meaning the temperature is another important factor for nitrogen removal as it can affect the microorganisms' activities.

From the parameters obtained for operations of the A/O system device in the laboratory for the treatment of these types of wastewater, it is possible to adapt it to an external pilot system that can use some automatic engineering devices such as water cooling systems (which may be possible with water already pre-treated at the plant) to control the temperature between 25˚C and 37˚C, also control the external carbon dosages (with another, less expensive carbon source, instead of glucose) to ensure the regularity of the C/N ratio, etc.). In fact, when the A/O system is opened to free air, the additional operating parameters to respect are pool A DO concentration maintaining within 0.5 mg/L by stirring the water body slowly, and pool O's DO concentration between 2 - 4 mg/L by mean of aeration device. In pool's O NaOH or another salt can be used to maintain pH value between 7.5 - 8.5. The system's wastewater reflux ratios are to be controlled to R = 500% and the sludge reflux ratio r = 100%. Depending on the climate (hot or cold) of the pilot plant installation area, the cost of cooling or heating the system can be reduced.

8. Conclusions

Sludge acclimatization failed when the blending ratio of HTS wastewater to FCC wastewater was introduced suddenly by 1.0% ratio with C/N ratio of 4.0:1.0 into the A/O system for treatment. The sludge's microorganisms did not adapt to the toxicity of the HTS wastewater resulting to a progressive increase of effluent's ammonia-N and TN concentrations and justifying a probable death of certain microorganisms. It can be related also to the presence of refractory organic compounds such as 3,5-di-tert-butylphenol that are toxic to microorganisms.

Then the microorganisms' acclimatization was slowly performed by reducing the HTS/FCC ratio to 0.50%.

After acclimating the microorganisms, the efficiency of the A/O system was assessed by varying the HTS/FCC ratios from 0.65% to 1.2% under operating conditions with a C/N ratio of 5.0/1. The result showed that the best HTS/FCC ratio giving a high nitrogen removal rate is 1%.

During these A/O system operations, it was found that the C/N ratio is a limiting factor for nitrogen removal and a C/N ratio of 5.5:1 is the most appropriate.

The temperature has also a great impact on the bioactivity of the sludge; it was found that when using the A/O system treatment with the HTS/FCC wastewater mixed ratio of 1.0% by using the temperature range of 25˚C - 37˚C, the resulting effluent's ammonia-N concentration was lower than 5 mg/L, the COD concentration less than 60 mg/L, while the TN concentration was lower than 40 mg/L therefore satisfying the discharge standards.

However, when the temperature of wastewater reached 40˚C, the performance of microorganisms decreased obviously, the sludge fell and floated, and the A/O system was unable to operate, resulting in the effluent's COD, ammonia-N and TN concentrations which could not meet the standard requirements.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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