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Research Article

Evaluation the Impact of NFC-dopped Titania Photocatalyst on Initial Inert COD Fraction of Çorlu Town Domestic Wastewater

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Abstract: In this paper, experimental studies were carried out to determine the impact of NFC dopped Titania, which is proven success in emerging pollutants such as antibiotic and color removal as a photocatalyst, on influent non-biodegradable COD fraction of Çorlu town domestic wastewater treatment plant. The determination of S_I and X_I components of wastewaters which are exposed (raw + photocatalyst) and unexposed (raw) to the NFC-dopped titania, are based on the experimental method carried out in both raw and filtred reactors which are run parallel to the glucose reactor prepared as same concentration with filtered wastewater. All reactors had been run thorugh 30 days in batch reactor mode. According to the results; total and soluble COD were measured as 535 mg/L and 315 mg/L, repectively and S_{T0}/C_{T0} ratio was calculated as 59%. Furthermore, at the end of the experimental study, COD removal ratio for both reactors were calculated as 67 % and 60% for total COD and 77% and %75 for soluble COD. Inert COD fractions of both wastewaters were found for Soluble Inert COD (S_{I0}) as 23 mg /L and 9 mg/L, particulate inert COD (X_{I0}) as 56 mg /L and 60 mg/L and C_{I0}/C_{T0} ratio was calculated as 15% and 13%. These all results shows that, after photocatalyst exposition, at the end of the 30 days, it was not observed any important variation in terms of inert total COD fraction between two reactors. Furthermore, it is understood that at the end of the 30 days, while soluble inert fraction reduces 2%, particulate inert fraction also increases 2%. The reason of this situation can be thought as the adsorbtion effect of NFC-dopped Titania.

Keywords: *Domestic waste water, Inert COD fraction, NFC-dopped titania, Photocatalyst.*

NFC Doplu Titania Fotokatalizörünün Çorlu Evsel Atıksu Giriş İnert KOİ fraksiyonu Üzerindeki Etkisinin Değerlendirilmesi

Özet: Bu makalede; antibiyotik, renk gibi gelişmekte olan kirleticilerin gideriminde bir fotokatalist olarak etkinliği kanıtlanmış NFC-doplu Titanyumun, Çorlu ilçesi evsel atıksu arıtma tesisi giriş suyunda biyolojik olarak parçalanamayan KOİ fraksiyonu üzerindeki etkisini belirlemeye yönelik deneysel çalışmalar yürütülmüştür. NFC-doplu titanyuma maruz kalan (ham + fotokatalist) ve maruz kalmayan (ham) atık suların S_I ve X_I bileşenlerinin belirlenmesi, süzülen atık su ile aynı konsantrasyonda hazırlanan glikoz reaktörüne paralel olarak çalışan hem ham hem de filtrelenmiş atıksu reaktörlerinde gerçekleştirilen deney yöntemine dayandırılmıştır. Bütün reaktörler kesikli modda 30 gün boyunca çalıştırılmıştır. Konvansiyonel karakterizasyon sonuçlarına göre, incelenen evsel ham atıksuyun toplam ve çözünmüş KOİ değerleri sırasıyla 535 mg/l ve 315 mg/l olarak ölçülmüş ve S_{T0}/C_{T0} oranı %59 olarak hesaplanmıştır. Ayrıca, deneysel çalışma sonunda fotokatalist maruziyeti öncesi ve sonrası COD giderim verimi toplam KOİ için sırasıyla %67 ve %60, çözünmüş KOİ için %77 ve %75 olarak hesaplanmıştır. Her iki reaktördeki atıksuların inert KOİ fraksiyonları ise sırasıyla Çözünmüş İnert KOİ (S_{I0}) için 23 mg /l ve 9 mg/l, Partiküler inert KOİ (X_{I0}) için 56 mg /l ve 60 mg/l ve C_{I0}/C_{T0} oranı da %15 ve %13 olarak hesaplanmıştır. Tüm bu sonuçlar göstermektedir ki; giriş ham atıksuyunun fotokataliste maruziyetinden 30 gün sonra, her iki reaktörde inert toplam giriş KOİ fraksiyonlarında önemli bir değişim gözlemlenmemesine ragmen, ham + fotokatalist atıksu reaktöründe çözünmüş inert kısmın %2 azaldığı, partiküler inert kısmın da %2 arttığı görülmüştür. Bunun nedeni NFC-doplu Titanyumun adsorbsiyon etkisi olarak düşünülebilir.

Anahtar kelimeler: *Evsel atıksu, İnert KOİ fraksiyonu, NFC-doplu titanyum, Fotokatalist.*

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1. Introduction

Domestic wastewater contains suspended, colloidal and dissolved organic and inorganic substances.The main organic compounds in domestic wastewater are proteins, carbohydrates, fats, petroleum residues and urea. Various synthetic organic substances such as detergents (pesticides), phenols and pesticides are also included in the wastewater. Both climatic conditions and living standards and cultures of people significantly affect wastewater characteristics.

Urban domestic wastewater can contain pre-treated or raw industrial wastewater and rainwater as well as domestic wastewater. Therefore, this type of wastewater includes conventional pollutants as well as a number of new generation micropollutants such as antibiotics, endocrine disruptors which are toxic and persistent pollutans that cannot be removed in conventional treatment plants. So, advanced oxidation processes and especially photocatalytic processes have received much attention in recent years due to their high efficiency to improve biodegradation of these pollutants. But still, there are not enough study in the literature about how these inorganic based photocatalyst such as titania used extremely successfully in photocatalytic processes effect the wastewater organic fraction, Because, in a domestic wastewater having modarate character, it is known that about 75% of suspended solids and about 40% of filterable solids are of organic character. Beside, although the COD parameter is the most commonly used collective parameter in the definition of wastewater organic characteristics, the inert part that is not biodegradable or permanently in the environment cannot be initially distinguished. Therefore, it is useful to determine the inert parts of COD (soluble (S_I) and particulate (X_I)) both in the evaluation of biological treatment plants efficiency and also to see the effect of photocatalytic process on the biological system as a pretreatment stage.

Organic matter in domestic can be classified into the four fractions of readily biodegradable COD (RBCOD), slowly biodegradable COD (SBCOD), non-biodegradable soluble COD (NBDSCOD), and non-biodegradable particulate COD (NBDPCOD) (Figure 1) [1;2].

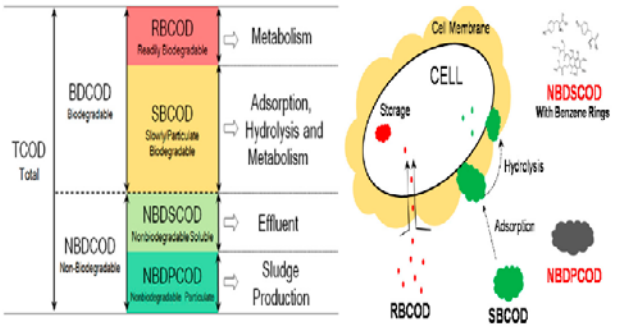


Figure 1. Schematic diagram of the chemical oxygen demand (COD) fractions and their fates in a biological wastewater treatment plant [6,7].

Although organic matter such as volatile fatty acids, as readily degraded by microbial metabolism, is called RBCOD, particulate organic matter, degraded slowly by a series of microbial reactions, composed of hydrolysis, metabolisis, adsorption exct. is called SBCOD. On the other hand, NBDSCOD, which is persistent in biodegradation, is mostly contained aromatic compounds which are used in various activities in our life. Forexample, depending on the type of industry, some industrial discharges are abundant in NBDSCOD, and thus, more refractory in a biological treatment process. It has been reported that dyeing factories discharge a large amount of NBDSCOD [3;4;5]. Because many chemical dyes, cleaning agent exct. synthetic based products are based on aromatic or heterocyclic ring structures, which are considered non-biodegradable. Especially textile dyeing mills likely contains substantial amounts of NBDSCOD. In addition, domesti industrial discharge from paper mill factories was reported to contain a large amount of lignin and lignin derivatives, which are also known to be non-biodegradable, and hence, increase the amount of NBDSCOD[6;7]. Besides, nowadays, domestic wastewaters has a moderate strong structure and present recractory structure in terms of emerging pollutants as well as industrial discharge.[8]

The COD parameter includes different forms of organic carbon that require a more detailed classification based on their biological decomposition properties. Therefore, total COD (C_{T0}) of raw wastewater is considered in two different classes as total degradable COD (C_{S0}) and total inert COD (C_{I0}) (Fig.2)[9]

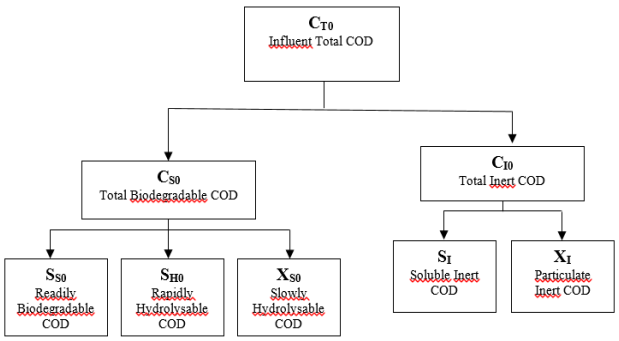


Figure 2. General distribution of COD components in influent wastewater [9].

Effluent of biological treatment system has a different COD structure than wastewater. The total dissolved COD (ST1) in the output stream consists of the non-biodegradable portion (S_{I1}) originating from the wastewater and exiting as it enters the system, a small portion (S_{S1} + S_{H1}) remaining after biological oxidation, and dissolved inert microbial products (S_{P1}).

As a result, the effluent generally contains more dissolved inert COD than the influent wastewater. The total dissolved inert COD at the effluent (SR) contains dissolved inert microbial products as well as dissolved inert COD in the wastewater that leaves the system unchanged ($S_R = S_I + S_P$) [9; 10).

Particulate COD has 4 components in the effluent wastewater. The major component is the active biomass XH1, which is retained in the reactor and uses biodegradable COD as the carbon and energy source. The other component is XS1, a small fraction of particulate degradable organics from hydrolysis and subsequent use. The effluent is also contained in particulate inert COD (XI1) present in the influent held by the sludge and deposited in the reactor. The fourth component is X_P, a particulate inert product resulting from microbial metabolic activity [11].

An advanced oxidation process might be required to degrade NBDSCOD containing aromatic compounds from dyeing mill, paper mill and municipal wastewaters [5;8;13].

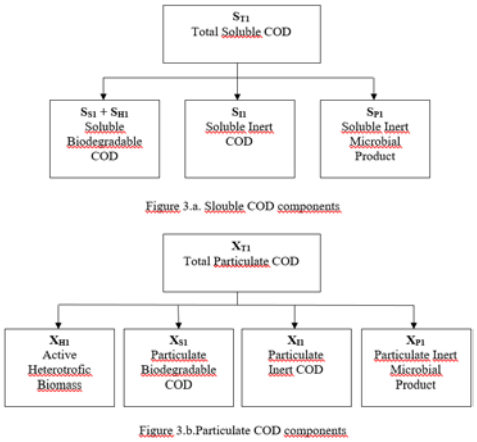


Figure 3. General distribution of COD components in effluent wastewater [11;12].

Particularly titanium dioxide (TiO_2), as an heterogeneous photocatalyst, is an effective water and wastewater treatment process for the removal of recalcitrant and photo-stable organic contaminants such as pharmaceuticals and antibiotics in wastewater treatment [14]. Although having an excellent photocatalytic activity, non-toxicity, long-term stability and low cost, TiO_2 has limited applications with a 3.2 eV band gap energy in anatase form absorbing only ultraviolet (UV) light in an effective way. In order to extend the photo absorbtion capability from the UV to the visible light region, recently doping methods with N, C, F, B, P, S elements have been widely used. [15;16;17;18] . Ata et.al. was reported that 2.25 mg/l NFC-dopped Titania is very succesfull to remove emerging pollutant such as antibiotic and color from both municipal and industrial wastewater [8].

So, in this paper, experimental studies were carried out to determine the impact of NFC dopped Titania on influent non-biodegradable COD fraction of Çorlu town domestic wastewater treatment plant. Determination of particulate and dissolved inert fractions of wastewaters is of great importance in terms of determining discharge standards and operating conditions. The dissolved inert COD is not affected by the biochemical reactions in the activated sludge reactor. Since this fraction has a direct effect on the effluent COD level and thus the performance of the system; In wastewater characterization, accurate determination of the SI value at the outlet is of great importance in terms of discharge standards. In addition, particulate inert COD is incorporated into the activated sludge and accumulates in proportion to the sludge age and leaves the system with the output stream. Therefore, the operating parameters of the treatment plants should be selected very well and the amount of sludge held in the system and the efficiency of the system must be calculated accurately. Within the scope of this study, untreated raw wastewater from the entrance of Çorlu domestic wastewater treatment plant was used, Then, Experimental studies carried out for the raw and raw + photocatalyst wastewaters reactors based on total, filtered and glucose comparasion method proposed by Orhon et.al [11].

2. Materials and Methods

2.1. Sampling and Characterization

Wastewater samples were collected from influent points of Çorlu domestic wastewater treatment plant including advanced biological treatment units in July. according to the standard of "TS ISO 5667-10 Sampling from Domestic and Industrial Wastewaters", kept in to the glass containers, labeled and transported under +1ºC temperature. Conductivity, pH, DO (Dissolved Oxygen) measurements were performed simultaneously with sampling at the same time via a Hach HQ40D Multimeter device. All measurement were carried out according to the international standard methods carefully; COD (Chemical Oxygen Demand) measurements to ISO 6060 (ISO 6060, 1986), and all other analyzes to APHA, 1998 Standart Methods [19].

2.2. Method

This study was carried out on the basis of experimental method for total and filtered (0,45 µ) wastewater and glucose reactors proposed by Orhon et al. [11;20]. According to this method; as a result of consuming the total degradable substrate and mineralizing the whole biomass in all reactors with a small amount of biomass (10-50 mg UAKM/1) previously considered, the values obtained in COD experiments reach a constant threshold. Inert COD profiles for this method are given in Figure 4 and Figure 5 [10].

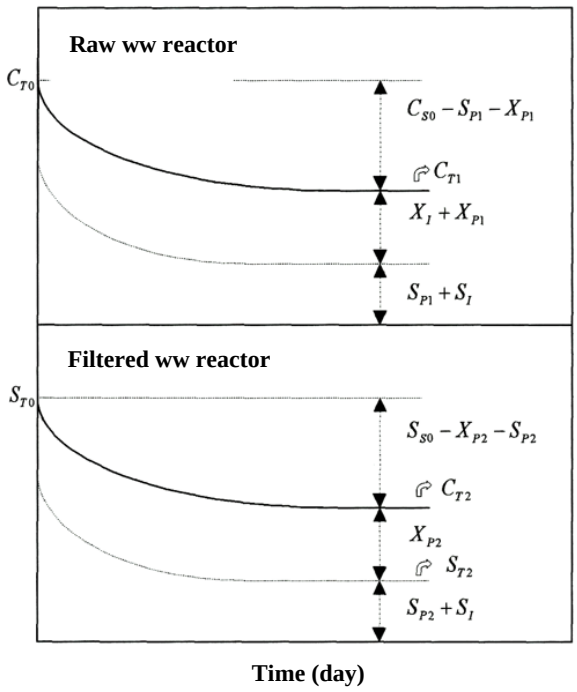


Figure 4. Inert COD profiles for raw and filtered wastewater reactors [20].

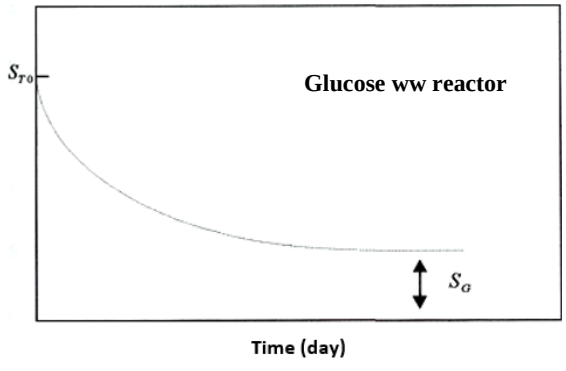


Figure 5. Inert COD profiles for glucose reactors [20].

The following correlations can be obtained by means of COD values of the second reactor fed with the filtered wastewater sample and the third reactor fed with glucose.

$$Y_{SP} = f_{ES} \cdot Y_H = S_G/S_{T0} \quad (2.1)$$
$$f_{ES} = (1/Y_H) \cdot (S_G/S_{T0}) \quad (2.2)$$

Where f_{ES} is soluble inert biomass fraction, S_G is Soluble COD at end of the experiment in glucose reactor, Y_H is heterotrofic yield coefficient. S_{T0} is the initial soluble COD in the glucose reactor as shown in Fig.5.

In the filtered wastewater reactor, S_I and f_{EX} are calculated according to the following equations:

$$S_{P2} = S_{T2(f)} - S_I \quad (2.3)$$
$$S_{P2} = f_{ES}*Y_H*(S_{T2(i)} - S_I) \quad (2.4)$$
$$S_I = [\ S_{T2(f)} - (f_{ES}*Y_H*S_{T2(i)})\ ] / (1 - f_{ES}*Y_H) \quad (2.5)$$
$$X_{P2} = C_{T2(f)} - S_{T2(f)} \quad (2.6)$$
$$S_{S0} = S_{T2(i)} - S_I \quad (2.7)$$
$$Y_{XP} = f_{EX}*Y_H = X_{P2} / S_{S0} \quad (2.8)$$
$$f_{EX} = (1 / Y_H)*[\ (C_{T2(f)} - S_{T2(f)}) / (S_{T2(i)} - SI)] \quad (2.9)$$

Where f_{EX} is particulate inert biomass fraction, S_{P2} is soluble inert microbial product concentration, S_I is soluble inert COD, $ST_{2(i)}$ is initial soluble COD, $S_{T2(f)}$ is residual final soluble COD, X_{P2} is particulate inert microbial product concentration, $C_{T2(f)}$ residual final total COD, S_{S0} is readiliy biodegradable soluble COD, Y_{XP} is the ratio of particulate microbial product to the total soluble COD in the filtered wastewater reactor.

The determination of XI from the raw wastewater reactor was made according to the following equations;

$$X_{T1} = C_{T1} - S_{T1} = X_I + X_{P1} \quad (2.10)$$
$$C_{S0} = C_{T0} - X_I - S_I \quad (2.11)$$
$$X_{P1} = f_{EX}*Y_H*(C_{T0} - X_I - S_I) \quad (2.12)$$
$$X_I = [\ (X_{T1} – f_{EX}*Y_H*(C_{T0} – S_I)\ ] / (\ 1- f_{EX} \cdot Y_H) \quad (2.13)$$

2.3. Activated Sludge Aclimation and Experimental Set-Up

In order to be used in inert COD experiments, the activated sludge taken from the aeration tank of investigated treatment plant was acclimated by feeding 50% -50% with 24-hour composite raw domestic wastewater and glucose in two seperate reactors. While one reactor was feeded only raw wastewater, the other was feeded raw + 2,25 mg/l NFC-dopped photocatalyst every day at sludge age 10 days, F/M ratio 0,5 mg COD/mg VSS-d. Since the nutrients in the raw wastewater were sufficient, no additional nutrients were added. The details of the experimental set up were given Table 1.

Table 1. Experimental set-up and reactor operation conditions.

EXPERIMENTAL SET UP						
REACTOR CONDITIONS	Raw ww	Filtered ww	Glucose	Raw ww + Photocatalyst (2,25 mg/L)	Filtered ww + Photocatalyst (2,25 mg/L)	Glucose ww + Photocatalyst (2,25 mg/L)
WW volume (mL)	1000	1000	1000	1000	1000	1000
COD(mg/L)	535	315	315	535	315	315
Biomass (mg VSS/L)	50	50	50	50	50	50

The reactors established under these conditions were fed and the sludge was added and the pH values of all reactors were adjusted to 7 using 6 N NaOH and 6 N H_2SO_4 solutions. After this stage, all reactors were connected to the air compressor and the experimental work started at the same time. Simultaneous samples were taken periodically from the all reactors through 30 days and total and soluble COD were measured. Before sampling, distilled water was added to the previous level up to the evaporating portion and the marking was made after each sampling. Experiments were continued until the total degradable substrate in all reactors was depleted and all biomass mineralized and COD concentrations reached a constant

All these reactors were establihed for both raw and and 2,25 mg/l NFC-dopped photocatalyst added wastewaters. NFC-dopped photocatalyst, prepared according to the sol-gel method without using thermal processing.and then characterized, was provided from another Project task group (NKUBAP.06.GA.18.183) in NKU Env. Eng. Department.

3. Results and Discussion

3.1. Wastewater Characterization

In this study, raw input water characterization of Çorlu domestic wastewater treatment plant is given in Table 2.

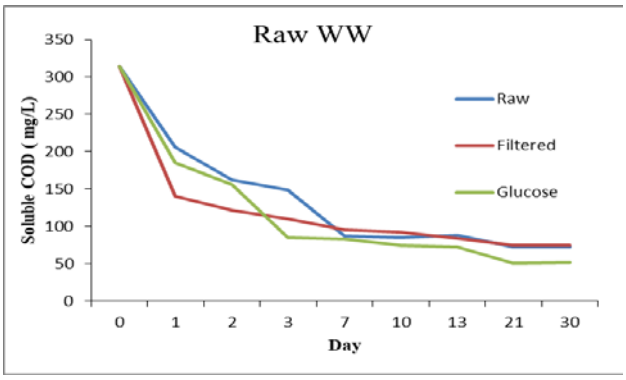
Table 2. Influent wastewater caharacterization of Çorlu Domestic WWTP.

Parameter	Unit	Range	Average
Total COD	mg/L	500-700	535
Soluble COD	mg/L	285-450	315
SS	mg/L	250-320	285
TKN	mg/L	50	50
NH_3-N	mg/L	17-23	20
T-P	mg/L	5,5-8,5	7
pH		6,5	6,5

According to the characterization results given Table 2, investigated domestic wastewater has modarate domestic character in terms of pollution pollutant concentrations. Furthermore, nütrient contents such as nitrogen and phosphorus, it is understood that it appears to have a balanced structure. These results also found compatible with the values reported in the national and international literatures.[21;22]

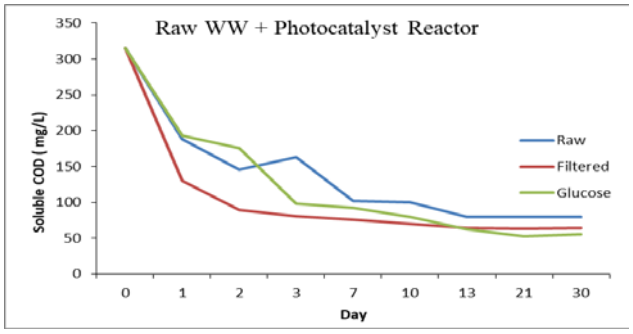
3.2. Experimental Results for Inert COD Determination

Soluble COD change during 30 days of Raw, filtered and Glucose reactors for both Raw WW and Raw ww + Photocatalyst (2,25 mg/L) are given at Garph 1 and Graph 2.



Graph 1. Soluble COD change during 30 days of Raw WW.

Initial and final residual total and soluble COD measurment results in all reactors were summarized on Table 3.



Graph 2. Soluble COD change during 30 days of Raw WW + Photocatalyst (2,25 mg/L).

Table 3. Initial and final residual total and soluble COD measurment results.

Paramete	Unit	Raw ww	Raw ww + Photocatalyst (2,25 mg/L)
C_{T0}	mg/L	535	535
C_{T1}	mg/L	177	215
S_{T0}	mg/L	315	315
S_{T1}	mg/L	73	80
$C_{T2(i)}$	mg/L	315	315
$C_{T2(f)}$	mg/L	106	113
$S_{T2(i)}$	mg/L	315	315
$S_{T2(f)}$	mg/L	75	64
S_{G0}	mg/L	315	315
S_G	mg/L	52	55

From the data obtained as a result of experimental studies, inert COD components were calculated according to the the method prposed by Orhon et.al.[11;20] and were given in Table 4 and Table 5.

Table 4. Inert COD components of Raw WW and Raw ww + Photocatalyst (2,25 mg/L).

Reactors	Filtered					Raw				
	Y_{SP}	F_{ES}	S_I (mg/L)	S_{P2} (mg/L)	X_{P2} (mg/L)	Y_{XP}	F_{EX}	X_{T1} (mg/L)	X_I (mg/L)	X_{PI} (mg/L)
Raw ww	0,17	0,26	23	48	31	0,106	0,17	104	56	48
Raw ww + Photocatalyst (2,25 mg/L)	0,17	0,28	9	53	49	0,160	0,25	135	60	75

Table 5. Compare of the study results to reported literature values.

	C_{T1}	S_{T1}	S_{II}	X_{II}	S_{II}/C_{T1} (%)	X_{II}/C_{T1} (%)
This study (Raw ww)	535	315	71	135	13	25
This study (Raw ww + Photocatalyst)	535	315	62	184	12	34
Domestic WW [20]	650	230	37	39	6	6
Domestic WW [21]	325-1283	200-760	19-60	31-210	1,5-8,5	9,5-10,5
Domestic WW [22]	540	110	36	86	7	16

According to the Table 3, removal ratios were calculated for total COD as 67% and 60% in raw ww reactors and as 66% and 64% and in filtered ww reactors, for soluble COD 77% and %75 in raw ww reactors and as 76% and 80 % in filtered ww reactors and as 83% in glucose reactors for both ww type. These results show that after photocatalyst exposition, it was not observed any important variation in terms of COD removal ratios for all reactors. So that, Table 5 shows that although there was observed a little bit increase in the particulate inert COD ratio calculated as 1%, reduction in S_I/C_{T0} and C_I/C_{T0} were found as 2% in Raw WW + Photocatalyst (2,25 mg/L) reactor. So, it is thought that the reason of this situation may be explained as the adsorbtion effect of NFC-dopped Titania. On the other hand, in terms of both S_I and X_I and C_I contents, this study resuls were found compatible with the values reported in the literature given Table 5 for domestic wastewaters.

4. Conclusion

Studies reported in the literature were shown that to determine inert COD fractions of wastewaters treated in biological treatment system are very important to understand the decomposition mechanism of subtrate. Especially although domestic wastewaters were known as more biodegradable organic fraction compared to the industrial wastewaters, recently determined emerging refractory pollutants such as antibiotics, endocrine disruptors, drugs and cleaning chemicals result in to increase in the nonbiodegradable fraction of domestic wastewater. So, to enhance this problem, most of domestic wastewater treatment systems were updated to remove these refractory pollutants with photocatalytic based advanced oxidation processes. Since this technology uses new generation photocatalysts, it has been inevitable to investigate the impacts of them on biological sytem. So, in this study, experimental studies were carried out to determine the impact of NFC dopped Titania photocatalyst on influent non-biodegradable COD fraction of Çorlu town domestic wastewater treatment plant.

As a result of the studies, after photocatalyst exposition, at the end of the 30 days, although it was not observed any important variation in terms of inert total COD fraction between two reactors, it was calculated 13% reduction for raw + photocatalyst wastewater reactor. By the way, it is understood that at the end of the 30 days, while soluble inert fraction reduces 2%, particulate inert fraction also increases 2%. The reason of this situation can be thought as the adsorbtion effect of NFC-dopped Titania.So, consequently, next study may be carried on the biodegradation kinetics of activated sludge exposed to NFC-dopped Titania with respirometric experiments and then this kinetic results can be attributed with the inert fraction results found in this study.

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