

# An innovative solution for the treatment of poultry industry wastewater with advanced hybrid technology sono-photocatalysis

#### Dogdu G.\* and Sen N.E.

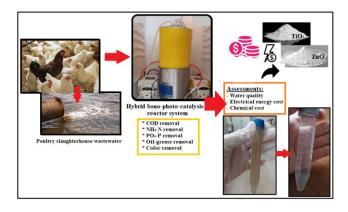
Department of Environmental Engineering, Bolu Abant Izzet Baysal University, Bolu, Turkey

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\*to whom all correspondence should be addressed: e-mail: gamzedogdu@ibu.edu.tr

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## **Graphical abstract**



## **Abstract**

Poultry slaughterhouse wastewater (PSWW) is a crucial concern fundamentally due to extensive generation and related substantial amounts of recalcitrant organic content due to high COD and oil and grease (O&G) levels. Hybrid advanced oxidation processes are promising, green, and innovative options to treat various industrial wastewater; to date, this potential has not been implemented to highstrength wastewater from the poultry industry. This work aims to investigate the combination of ultrasound and photocatalysis processes in the treatment of poultry slaughterhouse wastewater for the removal of COD and O&G under different operating conditions such as catalyst type (TiO<sub>2</sub> and ZnO), irradiation type (UVA<sub>365</sub> and UVC<sub>254</sub>), catalyst dosage (0.5-2.5 g/L), pH (2-10), hydrogen peroxide concentration (0-10 mM) and operation time (30-180 min). Taguchi's experimental design based on the L<sub>36</sub> orthogonal array was adequately applied to optimize the process. The finding results presented that ZnO concentration of 2.5 g/L, pH 2, and operation time of 180 min under UVC<sub>254</sub> were optimum parameters to achieve maximum COD removal while, ZnO concentration of 1.5 g/L, pH 6, H<sub>2</sub>O<sub>2</sub> concentration of 5 mmol/L and operation time of 30 min under UVC<sub>254</sub> were optimum parameters to achieve maximum oil and grease removal. With these optimum conditions, the best attained COD and O&G removal yields were 54% and 99%, respectively.

**Keywords:** Optimization, poultry slaughterhouse wastewater, Sonophotocatalysis, synergetic effect, Taguchi method

#### 1. Introduction

The poultry meat sector is accepted as one of the largest and rapidly growing agro-based industries in many parts of the world due to being an affordable and nutritious protein source (Ngobeni et al., 2022; Hilares et al., 2021). Despite high economic gains, poultry processes such as slaughtering, de-feathering, evisceration trimming, and washing are extremely water-intensive processes, consuming an averagely of 26.5 L per bird (Fatima et al., 2021). PSWW contains different complex organic and inorganic substances such as blood, fat oil and grease (FOG), protein, heavy metals, and detergents for cleaning, as well as high concentrations of nutrients such as nitrogen (N) and phosphorus (P), chemical oxygen demand (COD), biochemical oxygen demand (BOD), suspended and dissolved solids and pathogens (Bustillo-Lecompte et al., 2016). FOG can be a crucial problem for wastewater treatment because of their slow decomposition (Gaur et al., 2010). Especially for biological treatment systems, biodegradation activity can be hindered due to clogging and blockage of the piping system and encapsulation of the granules in the presence of FOG (Bingo et al., 2021). Besides, the discharge of untreated poultry wastewater into the receiving environment creates eutrophication and over-fertilization of soils. Moreover, it causes infection-related health risks for humans and animals using contaminated water, as well as problems such as congestion and foam formation in the drainage systems of wastewater treatment plants, due to excess nitrogen and phosphorus in their content (Cao et al., 2021; Garcia et al., 2020). Therefore, the urgency to develop an efficient, environmentally friendly and innovative advanced treatment technology to reach specific discharge limits and to ensure sustainable economic growth is inevitable (Dyosile et al., 2021).

Recently, several advanced oxidation processes (AOPs) have been explored for the treatment of PSWW, i.e., electrocoagulation (Potrich *et al.*, 2020; Emerick *et al.*, 2020), Fenton (Cui *et al.*, 2021), electro-Fenton (Zhang *et* 

al., 2020), ultrasound (Abdelhay et al., 2020) and photocatalysis (Samsudin et al., 2019; Asha and Kumar, 2015); however, to the best of our knowledge, there has yet been no published researches have been established on the synergistic effect of combined photocatalysis and sonolysis processes called "sono-photocatalysis" (SPC) for the PSWW treatment. The hybrid SPC technique has gained much popularity to perfectively degrade all kinds of pollutants in extending the photogenerated electron-hole transfer pathway owing to easy operational, cheapness, and rapid pollution-free technology (Khan et al., 2022; Karim and Shriwastav, 2020; Fan et al., 2022). Besides, critical limitations, including the required long reaction time for the single process and their low capability to achieve complete mineralization of organic molecules for their individual use can be eliminated by the combination of photon, ultrasonic waves, and a semiconductor catalyst (Abdurahman et al., 2021; Khitab et al., 2022).

Although several kinds of research have been reported so for concerning the photocatalytic activity of TiO<sub>2</sub> (~3.2 eV) and ZnO (~3.37 eV), which are widely used functional semiconductors that are chosen for their low cost, chemical stability, non-toxicity, and higher photosensitive properties (Stando et al., 2021; Boutra et al., 2021), as the first time, the comparison performances of these two catalysts on the treatment of PSWW were widely explored in this work. Also, treatment efficiencies of SPC for PSWW under UVA<sub>365</sub> and UVC<sub>254</sub> irradiations have not been studied by any researchers, which was investigated in this study. Moreover, hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is a strong oxidant, which enhances the generation of HO. and pollutant degradation is rarely reported in the SPC process (Wei et al., 2021; Silva et al., 2007). In this work, the effect of H<sub>2</sub>O<sub>2</sub> concentration and was evaluated in detail. Taguchi's experimental design based orthogonal (OA) on array analysis was used to determine the contribution of each factor, optimal condition and the response with number of tests, time, and experiment cost (Fard et al., 2020) in various treatment processes industrial wastewater. Nonetheless, based on an extensive literature survey, research on the application of Taguchi's method for PSWW treatment using the SPC technique has not been available. Therefore, to address these knowledge gaps, the primary aim of this study was to optimize the individual and interactive effect of process variables such as catalyst type and dosage of catalysts (TiO2 and ZnO), pH of solution, H<sub>2</sub>O<sub>2</sub> concentration, ultraviolet (UV) irradiation type (UVA<sub>365</sub> and UVC<sub>254</sub>) and operation time on the COD and oil and grease from PSWW using Taguchi experimental design with L<sub>36</sub> (2<sup>2</sup> x 3<sup>4</sup>) orthogonal array.

### 2. Materials and methods

## 2.1. Poultry slaughterhouse wastewater

The poultry slaughterhouse wastewater (PSWW) used in the present study was collected from a local integrated poultry processing plant, which has a capacity of 180.000/chicken and produces an averagely of 1300-1500 m³ wastewater per day located at Bolu, Turkey. The sample was taken from the effluent pipe of the slaughterhouse

unit of the plant. After collection, the samples were stored at 4 °C in dark conditions. Raw poultry wastewater samples were filtered to remove feather, bone, and meat particles before each experiment. The chemical analysis of PSWW was shown in Table 1.

#### 2.2. Chemicals

The nano-sized photocatalysts, TiO<sub>2</sub> (AEROXIDE® P25  $\geq$  %99,5, anatase form, 21 nm, 35–65 m²/g BET surface area) and ZnO (<5  $\mu$ m particle size, %99,9; <10-25 m²/g) were supplied from Sigma-Aldrich (Germany). H<sub>2</sub>O<sub>2</sub> (30%), used as an oxidizing agent, was also obtained from Sigma-Aldrich (Germany). Sodium hydroxide (NaOH, 99% purity) and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, 97% purity) were obtained from Merck (Germany) to ensure the desired pH conditions in the experiments. All chemicals were used without additional purification. During the experiments, deionized water was used to prepare the necessary solutions (Merck Milli-Q, Germany, spec. resistivity: 18.2 M $\Omega$ ).

## 2.3. Experimental set-up and procedure

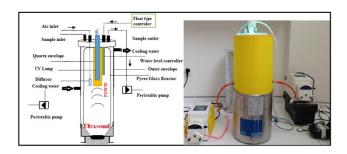
A schematic of SPC reactor used in this work is shown in Figure 1. All ultrasound, photocatalytic, and sonophotocatalytic experiments were performed using the same cylindrical Pyrex glass immersion well reactor filled with 200 mL solution with a certain amount of catalyst. The batch-mode reactor was equipped with a water jacket and a PL-L UVA<sub>365</sub> lamp (Philips, Dutch) (360 W; 315 to 380 nm; 110  $\mu$ W/cm<sup>2</sup>), and a UVC<sub>254</sub> lamp (Philips, Dutch) (9 W; 254 nm) were used to study the SPC method. Air at 3.5 L/min was supplied to the reactor system using a diffuser. For the US experiments, the whole photoreactor system was immersed in an ultrasonic bath (Bandelin DT 106, Germany) with a capacity of 5.6 L (operating volume, 200 mL), a tank size of D = 240 mm, and L = 125 mm, and an operating frequency of 35 ± 3 kHz (120 W, 220 V) that was equipped with the in-built piezoelectric transducer placed at the bottom and an external generator was used. The reactor temperature was kept constant at 25 ± 3 °C with a continuous water bath (NUVE ST-30, Turkey) and a cold water pump (Filtec PH-15X3S-FPP-1, Germany).

# 2.4. Analytical methods

PSWW samples were taken at the beginning and end of each experiment run, centrifuged at 5000 rpm for 15 min, and filtered using 0.45 µm filters (Minisart RC25, Sartorious) to remove catalysts. O&G was measured according to standard method 1164, EPA with hexane extraction. COD values before and after treatment were measured by (Merck Spectroquant Pharo100) spectrophotometer that was adapted for the Standard Method for Water and Wastewater (APHA, AWWA & WEF, 2012). COD was determined in the influent and effluent of the reactor samples using the Merck spectroquant COD cell test kits, respectively. When the sample containing peroxide (H<sub>2</sub>O<sub>2</sub>), interference in COD determination was reduced by increasing the pH to above 10 to decompose the hydrogen peroxide to oxygen and water (Talinli and Anderson, 1992). The pH of the wastewater was adjusted either using 0.1 M concentrated H<sub>2</sub>SO<sub>4</sub> or 0.1M NaOH. Electrical conductivity (EC), redox potential, and pH of the wastewater were determined by direct measurement using Termo Orion 5 Star multimeter device. The reactor was operated in a batch mode for 180 min when was needed to reach steady-state COD readings according to preliminary studies. Each experiment was conducted twice, the pollutant concentrations were measured from influent and effluent from the reactor system each time and the average results were given in this study. The pollutant removal efficiency was calculated as the following formula:

$$R(\%) = 100\% \times (C_i - C_f)/C_i$$
 (1.1)

where,  $C_{\rm i}$  and  $C_{\rm f}$  represent the mean values of initial concentration and final concentration after treatment, respectively.



**Figure 1.** Experimental setup for the hybrid sonophotocatalysis (SPC) process.

Table 1 The characterization of raw poultry slaughterhouse wastewater (PSWW)

Parameter	Unit	Value
рН	-	7.37 ± 0.03
Temperature	ōC	23.0 ± 0.06
Redox potential	mV	25.7 ± 0.38
Electrical conductivity	μS/cm	1356 ± 2.65
Turbidity	NTU	722 ± 2.52
Color	Pt/Co	6127 ± 46.0
Oil & grease	mg/L	5647 ± 19.3
Suspended solids	mg/L	493 ± 2.52
COD	mg/L	3280 ± 39.1
NH <sub>4</sub> -N	mg/L	95 ± 5.00
NO <sub>2</sub> -N	mg/L	6.67 ± 0.15
NO <sub>3</sub> -N	mg/L	30.0 ± 1.00
PO <sub>4</sub> -P	mg/L	105 ± 2.00
Physical appearance	-	Reddish brown
T-N	mg/L	305 ± 5.00
T-P	mg/L	47.7 ± 1.53

Table 2. Experimental factors and design levels of orthogonal array

Code	Symbol	Factors	Unit	Level 1	Level 2	Level 3
Α	$C_{T}$	Catalyst type	=	TiO <sub>2</sub>	ZnO	=
В	$UV_T$	UV irradiation type	-	UVA <sub>365</sub>	UVC <sub>254</sub>	-
С	Cc	Catalyst concentration	g/L	0.5	1.5	2.5
D	рН	рН	-	2	6	10
Е	C <sub>H2O2</sub>	H <sub>2</sub> O <sub>2</sub> concentration	mmol/L	0	5	10
F	t	Time	min	30	105	180

## 2.5. Taguchi experimental design

To identify and optimize the process parameters on the selected response though the use of analysis of variance (ANOVA), Taguchi's design of experiments (DOE) approach was conducted by employing a minimum number of the test via orthogonal arrays (OA) (Aydıner  $et\ al.$ , 2019). In this study, Taguchi's approach with six controllable factors (A: catalyst type (C<sub>T</sub>), B: UV irradiation type (UV<sub>T</sub>), C: catalyst concentration (Cc), D: pH, E: H<sub>2</sub>O<sub>2</sub> concentration (CH<sub>2</sub>O<sub>2</sub>), F: operation time (t) were chosen for investigating SPC process. The levels of the factors used in the process were selected according to the literature review (Ayare and Gogate, 2020; Karim and Shriwastav, 2020; Steven  $et\ al.$ , 2021; Khan  $et\ al.$ , 2022). Table 2 shows the Taguchi L<sub>36</sub> (2<sup>2</sup> x 3<sup>4</sup>) orthogonal array that defines a total of 36 experimental sets, two independents with two levels and

four independents with three levels used in the study. Minitab 17.0 Statistical Software was applied for optimization and statistical analysis. COD and O&G removal yields were chosen as two responses for investigating the hybrid SPC method. The signal/noise ratio (S/N) defines the measurement of the deviation of the response from the desired value (Fdez-Sanromán *et al.*, 2021). While the mean value shows "signal" that represents the desirable effect, the standard deviation indicates "noise" represents the undesirable effect of the data set measured by the S/N ratio (Sohrabi *et al.*, 2017).

To achieve maximum pollutant removal performance with lower variability in the process, the "largest best" (eqn. 2.1) S/N ratio was chosen (Abbas and Abbas, 2021):

$$\frac{S}{N}[dB] = -10log \left[ \frac{1}{n} \sum_{i=1}^{n} \frac{1}{Y_i^2} \right]$$
 (2.1)

where n is the number of experimental replicates and  $Y_i$  is defined as the performance value of the  $i^{th}$  experiment.

#### 3. Results and discussion

**Table 3.** COD removal efficiencies resulting from using a variety of individual and combined processes (35 kHz US frequency; 120 W US power; pH 6; 1.5 g/L catalyst concentration; 10 mmol/L  $H_2O_2$  concentration; 36 W UV power; 25 ± 3 °C temperature; 180 min)

Advanced oxidation treatment method	COD removal (%)
Ultraviolet A (UVA <sub>365</sub> )	5 ± 0.08
Ultraviolet A (UVC <sub>254</sub> )	7 ± 0.01
Ultrasound (US)	5 ± 0.16
TiO <sub>2</sub>	6 ± 0.01
ZnO	9 ± 0.1
$H_2O_2$	6 ± 0.24
UVA <sub>365</sub> +TiO <sub>2</sub>	16 ± 0.01
UVA <sub>365</sub> +ZnO	19 ± 0.22
UVC <sub>254</sub> +TiO <sub>2</sub>	22 ± 0.07
UVC <sub>254</sub> +ZnO	28 ± 0.02
US+UVA <sub>365</sub> +TiO <sub>2</sub>	22 ± 0.74
US+UVC <sub>254</sub> +TiO <sub>2</sub>	30 ± 0.85
US+UVA <sub>365</sub> +ZnO	33 ± 0.34
US+UVC <sub>254</sub> +ZnO	47 ± 0.30

The preliminary experiments suggest that the COD removal efficiencies were 5%, 5%, 7%, 6%, 9%, and 6% using sonolysis (US), photolysis (P) (UVA<sub>365</sub> and UVC<sub>254</sub>), catalysis ZnO) and oxidation, respectively. It was observed that none of the individual processes were capable of satisfactory COD removal from PSWW. Similar to our findings, Al-Bsoul et al., (2020) stated that only 3% sono-photocatalytic oxidation of olive mill wastewater (OMW) was achieved after UV treatment for 180 min versus 5% obtained with ultrasound. Especially at low frequencies, the formation of highly hydrophilic intermediates via HO. the attack leads to a long sonochemical degradation time to achieve complete mineralization (Ahmedchekkat et al., 2011). Wei et al., (2021) indicated that sono-photocatalytic degradation efficiencies of acid orange 7 (AO7) dye were 3.61%, 8.14%, 1.15%, and 1.97% using sonolysis, photolysis, catalysis, and oxidation, respectively. Kakavandi and Ahmadi (2019) declared that the minimum efficiencies for single sonolysis or photolysis could allude to by the lower generation rate of free radicals in the whole system. Coupling the UV irradiations with TiO2 and ZnO catalysts called "photocatalysis (PC)" significantly improved the removal rate of COD (between 16% and 28%) due to the excellent activation capability of TiO2 and ZnO under UV irradiations (Yang et al., 2021). Higher oxidation resulted with the absorbance of incident light by semiconductors. Consequently, higher energy photo-induced electrons (e-) and holes (h<sup>+</sup>) were generated which migrated to the catalyst surface and produced reactive oxygen radicals (ROS) that participated in oxidation reaction with pollutants (Patidar and Srivastava, 2021).

For the hybrid system, the introduction of US with UVA $_{365}$ /TiO $_{2}$ , UVC $_{254}$ /TiO $_{2}$ , UVA $_{365}$ /ZnO, and UVC $_{254}$ /ZnO significantly improved the COD removal efficiencies by 22%, 30%, 33%, and 47%, respectively. The synergy index is the ratio of the hybrid SPC removal percentage value to the sum of the individual processes' removal percentage values with regard to COD removal. The synergy was calculated to determine the system's synergy according to eqn. 3.1 (Poblete *et al.*, 2020):

3.1. Performance of individual AOPs and synergistic effect

Several energy-based AOPs, namely UVA $_{365}$ , UVC $_{254}$ , US, TiO $_2$ , ZnO, and H $_2$ O $_2$  oxidation were applied individually to

attain the oxidation potential in terms of COD removal

from PSWW for 180 min under the same operation

conditions as shown in Table 3.

Synergy index

$$= \frac{\text{Removal of pollutants with US and PC}}{\text{Removal of pollutants with PC} + \text{Removal of pollutants with US}}$$

For the oxidation of PSWW, the observed synergy index values for the combined US/UVA $_{365}$ /TiO $_2$ , US/UVC $_{254}$ /TiO $_2$ , US/UVA<sub>365</sub>/ZnO, and US/UVC<sub>254</sub>/ZnO were obtained as 1.05, 1.11, 1.38 and 1.42, respectively. Kakavandi and Ahmedi (2019) proposed that synergy index (R) equals to 1 and R > 1.0 implies the integrated effect was greater than the sum of all individual effects and the combination of photocatalysis (PC) and ultrasound processes (US) exhibited higher oxidation performance of PSWW when compared with individual processes. The combination of two systems into one system enhances the amount of HO. radicals formed as a result of the increment in the surface area of the catalyst that accelerates the degradation of organic pollutants as indicated by Al-Bsoul et al., (2022). The cavitation energy followed by its transformation into microbubbles is generated by ultrasonic waves which produce effectively higher localized temperature and pressure that destroy water molecules and produce additional hydroxyl radical HO., respectively. The additional hydroxyl free radical HO. formation along with

superoxide radical (O2\*) helps in the improvement of the photodegradation ability of SPC (Khan *et al.*, 2022). Secondly, US conditions help to improve the dispersal of nanoparticles by preventing agglomeration, thus generation of radicals could be increased with higher catalyst active sections exposed (Babu *et al.*, 2019). So, the US can enable to increase the photocatalytic reaction rate by the catalytic activity of the catalyst. Also, acoustic cavitation cleans the catalyst surface and mass transfer induced by the US is accelerated between the solution and catalyst, which might lead to an increase in the photocatalytic degradation rate (Van de Moortel *et al.*, 2020).

## 3.2. Taguchi analysis to determine optimum parameters

Taguchi L<sub>36</sub> design with the mean S/N ratios for each parameter level and removal efficiencies of COD and O&G are shown in Table 4. Since "larger-the-better" criteria were chosen for S/N ratios to achieve maximum removal yield, the highest values were optimum values. While COD removal efficiencies were between 15% and 54%, O&G removal efficiencies changed between 68% and 99%. Taguchi response table was used to determine the most effective process parameter between C<sub>T</sub>, UV<sub>T</sub>, C<sub>C</sub>, pH, C<sub>H2O2</sub>, and t for optimum COD removal efficiency level. S/N ratios were calculated using Eq. 2 are given in Table 5.

Operating conditions for the highest COD removal were obtained as A<sub>2</sub>, B<sub>2</sub>, C<sub>3</sub>, D<sub>1</sub>, E<sub>1</sub>, and F<sub>3</sub> as presented in Table 5. The optimum parameters for COD removal efficiency were A (catalyst type) at level 2 (ZnO), B (UV irradiation type) at level 2 (UVC<sub>254</sub>), C (catalyst concentration) at level 3 (2.5 g/L), D (pH) at level 1 (2), E (H<sub>2</sub>O<sub>2</sub> concentration) at level 1 (0 mmol/L) and F (time) at level 3 (180 min), respectively. According to optimum conditions, COD removal efficiency was obtained as 54%.

In the present study, the importance of operating parameters on O&G removal efficiency was analyzed and S/N ratios were obtained are given in Table 5. Since 'the largest is the best' criteria were chosen, the highest values were optimum values. O&G removal efficiencies were between 68% and 99%. The optimum conditions were obtained as A<sub>2</sub>, B<sub>2</sub>, C<sub>2</sub>, D<sub>2</sub>, E<sub>2</sub>, and F<sub>1</sub> for O&G removal. The optimum parameters for O&G removal efficiency were A (catalyst type) at level 2 (ZnO), B (UV irradiation type) at level 2 (UVC<sub>254</sub>), C (catalyst concentration) at level 2 (1.5 g/L), D (pH) at level 2 (6), E (H<sub>2</sub>O<sub>2</sub> concentration) at level 2 (5 mmol/L) and F (time) at level 1 (30 min), respectively. Under optimum conditions, O&G removal was obtained as 99%. These results showed that the optimum operating parameters' values changed according to the pollutant parameter, even if the same type of wastewater and reactor system were used as expressed by Adar and Acar (2021). It was observed that complete COD mineralization was more difficult and required a long time than O&G removal. Hence, the system should be optimized in terms of COD removal.

# 3.3. Statistical analysis

Table 6 gives the analysis of variance (ANOVA) results revealing the effects on COD and O&G removal efficiencies

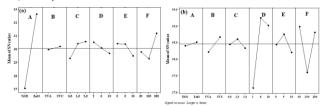
of the control factors of C<sub>T</sub>, UV<sub>T</sub>, C<sub>C</sub>, pH, C<sub>H2O2</sub>, and t at the confidence level of 95% and a significance level of  $\alpha$  = 0.05. The analysis was evaluated for  $\alpha$ =0.05 significance level. As given in Table 6, the errors were 8.6% and 22.37% (<50%) for COD and O&G removal, respectively which were well below the limit value. This implied that the errors of the experiments were insignificant. Further, the quantitative assessment can be obtained using the percentage contribution rate (Cr %) (Yildiz, 2008). According to these results, the most important parameters for COD removal efficiency were presented as catalyst type (F: 230.2, Cr: 79%) > operation time (F:8.75  $C_r$ : 6.02%) > UV lamp type (F: 5.46, C<sub>r</sub>: 1.88%), respectively as shown in Table 6. However, the contributions of catalyst concentration (F: 2.4, Cr: 1.65%), pH (F: 2.61, C<sub>r</sub>: 1.8%) and H<sub>2</sub>O<sub>2</sub> concentration (F: 3.33,  $C_r$ :0.92%) were statistically insignificant (p >  $\alpha$ ) in COD removal by SPC process. Besides, pH (F: 29.93, C<sub>r</sub>: 54%) and operation time (F: 9.22, C<sub>r</sub>: 17%) were the most significant factors (p< $\alpha$ ) in the removal efficiency of O&G in the SPC process.

3.4. Effects of operating parameters on COD and O&G removal efficiencies

## 3.4.1. Catalyst type effect

The main effect graph of the process parameters on the removal efficiencies of COD and O&G in real poultry slaughterhouse effluent is clearly shown in Figure 2. As shown in Figure 2(a-b), the sono-photocatalytic performance of the ZnO catalyst was found to be strongly higher than the TiO2 catalyst in removing COD and O&G from raw PSWW, especially for the degradation of organic oil molecules due to its excellent catalytic activity, low-cost, non-toxicity, super hydrophilicity properties (Zioui et al., 2019). For the elimination of O&G from wastewater, TiO<sub>2</sub> and ZnO catalysts play an important role in two removal mechanisms: (1) the interaction of oil on the adsorbent surface by adsorption that depends on the physical trapping of oil molecule on the adsorbent surface (Cui et al., 2014); (2) photocatalysis for the separation of oil/water emulsion. TiO2 or ZnO catalysts can be adsorbed on the interfacial surface between the oil and water when the oil in the water emulsion is blended with a photocatalyst. The surface of an organic emulsifier can be oxidized by the hydroxyl radicals between water and oil that are resulted in the destruction of the interfacial film structure (Liang and Esmaeili, 2021). Although ZnO has a similar band gap energy (3.37 eV) to TiO<sub>2</sub>, the stability and electron mobility of ZnO (~100 cm<sup>2</sup>/Vs) is twice that of TiO<sub>2</sub> (>1cm<sup>2</sup>/Vs) (Gole et al., 2017). Patidar and Srivastava (2021) also reported similar results during the sono-photocatalytic degradation of ofloxacin (OFLX) with 0.4 g/L ZnO catalyst under 40 kHz frequency, 125 W UV power at pH 6.3 within 120 min. Under UVA<sub>365</sub>/US/TiO<sub>2</sub> process, OFLX mineralization efficiency was 24.1% while, 36.2% was obtained under UVA<sub>365</sub>/US/ZnO system. The reason for the higher performance of the ZnO catalyst in comparison to the TiO<sub>2</sub> is due to the absorption of more light quanta as the quantum efficiency of the ZnO photocatalyst is higher than TiO<sub>2</sub> (Patidar and Srivastava, 2021). Besides, the ZnO catalyst is thought to have a lower light-scattering effect

due to its lower refractive index (ZnO: 2.0, TiO<sub>2</sub>: 2.5-2.7) which also favors better photocatalytic efficiency (Jyothi *et al.*, 2015).



**Figure 2.** Main effects plot for S/N ratios with the larger-the better criteria of pollutant (a: COD, b: O&G) removal efficiencies (A: catalyst type, B: UV irradiation type, C: catalyst concentration, D: pH, E:  $H_2O_2$  concentration, F: operation time).

Table 4. System performances obtained for all response parameters in SPC process

							COD remo	oval	O&G removal		
No.	C <sub>T</sub>	UV <sub>T</sub>	рН	C <sub>C</sub> (g/L)	C <sub>H2O2</sub> (mmol/L)	t (min)	Percentage (%)	S/N (db)	Percentage (%)	S/N (db)	
1	TiO <sub>2</sub>	UVA	2	0.5	0	30	17.15	25.87	77.66	37.80	
2	TiO <sub>2</sub>	UVA	6	1.5	5	105	20.83	26.80	88.49	38.90	
3	TiO <sub>2</sub>	UVA	10	2.5	10	180	22.87	26.84	83.20	38.42	
4	TiO <sub>2</sub>	UVA	2	0.5	0	30	23.69	25.87	77.65	37.80	
5	TiO <sub>2</sub>	UVA	6	1.5	5	105	23.12	26.80	87.66	38.90	
6	TiO <sub>2</sub>	UVA	10	2.5	10	180	21.19	26.84	83.48	38.42	
7	TiO <sub>2</sub>	UVA	2	0.5	5	180	19.32	25.72	78.14	37.86	
8	TiO <sub>2</sub>	UVA	6	1.5	10	30	21.62	26.70	90.29	39.11	
9	TiO <sub>2</sub>	UVA	10	2.5	0	105	21.54	26.66	77.90	37.83	
10	TiO <sub>2</sub>	UVC	2	0.5	10	105	18.86	25.51	55.25	34.85	
11	TiO <sub>2</sub>	UVC	6	1.5	0	180	29.28	29.33	90.25	39.11	
12	TiO <sub>2</sub>	UVC	10	2.5	5	30	24.46	27.77	95.14	39.57	
13	TiO <sub>2</sub>	UVC	6	0.5	10	30	15.06	23.56	86.10	38.70	
14	TiO <sub>2</sub>	UVC	10	1.5	0	105	17.59	24.90	89.46	39.03	
15	TiO <sub>2</sub>	UVC	2	2.5	5	180	32.86	30.33	80.22	38.09	
16	TiO <sub>2</sub>	UVC	6	0.5	10	105	20.78	26.35	91.64	39.24	
17	TiO <sub>2</sub>	UVC	10	1.5	0	180	30.98	29.82	98.51	39.87	
18	TiO <sub>2</sub>	UVC	2	2.5	5	30	30.26	29.62	75.45	37.55	
19	ZnO	UVA	6	0.5	0	180	47.29	33.49	94.45	39.50	
20	ZnO	UVA	10	1.5	5	30	39.79	32.00	92.18	39.30	
21	ZnO	UVA	2	2.5	10	105	36.96	31.35	71.45	37.08	
22	ZnO	UVA	6	0.5	5	180	50.53	34.07	94.65	39.52	
23	ZnO	UVA	10	1.5	10	30	34.67	30.80	97.11	39.74	
24	ZnO	UVA	2	2.5	0	105	45.13	33.09	59.68	35.52	
25	ZnO	UVA	10	0.5	5	30	36.12	31.15	93.05	39.37	
26	ZnO	UVA	2	1.5	10	105	40.32	32.11	55.48	34.88	
27	ZnO	UVA	6	2.5	0	180	43.51	32.77	82.16	38.29	
28	ZnO	UVC	10	0.5	5	105	37.39	31.45	84.13	38.50	
29	ZnO	UVC	2	1.5	10	180	52.00	34.32	86.90	38.78	
30	ZnO	UVC	6	2.5	0	30	40.41	32.13	97.50	39.78	
31	ZnO	UVC	10	0.5	10	180	46.11	33.28	96.85	39.72	
32	ZnO	UVC	2	1.5	0	30	53.63	34.59	81.36	38.21	
33	ZnO	UVC	6	2.5	5	105	40.44	32.14	98.50	39.87	
34	ZnO	UVC	10	0.5	0	105	38.75	31.76	77.46	37.78	
35	ZnO	UVC	2	1.5	5	180	45.42	33.14	76.50	37.67	
36	ZnO	UVC	6	2.5	10	30	47.54	33.54	95.80	39.63	

## 3.4.2. Irradiation type effect

As shown in Figure 2(a-b), the removal of COD from PSWW by using UVA (320-400 nm) and UVC (253.7 nm) radiation was investigated and UVC radiation showed statistically significant performance in comparison with UVA (p=0.028, p< $\alpha$ ). According to photon theory and Planck's eqn. 3.2:

$$E = hC/\lambda \tag{3.2}$$

h is Planck's constant (6.626 x  $10^{-34}$  Js), c is the speed of light (3 x  $10^8$  m/s) and  $\lambda$  defines the cut-off wavelength, respectively. The wavelength is inversely proportional to the energy, thus shorter wavelength has a higher energy that results in increased activity and a higher probability of

photocatalytic reaction (Termtanun, 2013). Bushnaq (2006) stated that under UVC irradiation, atrazine was degraded by direct photolysis by about 95% within 60 min compared with 10% UVA irradiation. Besides, in the presence of 15 mg/L, atrazine removal was obtained as 84% by UVC irradiation compared with UVA in the presence of 25 mg/L TiO<sub>2</sub> catalysts after 60 min of irradiation. TiO<sub>2</sub> could absorb light at a lower wavelengths better than at a higher wavelength, hence UVC with a shorter wavelength corresponds to higher photon energy (Termtanun, 2013). Similarly, Yatmaz and Sen (2018) explained that homogeneous and heterogeneous photo-Fenton with the use of mineral catalysts achieved higher dye removal under UVC irradiation in comparison to those under UVA and mineral catalysts gave shorter removal times under UVC than under UVA.

## 3.4.3. Catalyst concentration effect

The effect of three catalyst dosages of 0.5 g/L, 1.5 g/L, and 2.5 g/L on the COD and O&G degradation were investigated for  $TiO_2$  and ZnO catalysts, respectively as given in Figure 2(a-b). As expected, with an increase in catalyst dosage,

percentage removal efficiencies of COD and O&G increase up to 1.5 g/L which is due to the maximum number of active sites available with more photo-generated electron holes for the generation of maximum active species during the degradation of organic pollutants (Abdelhay et al., 2020; Khan et al., 2022). Also, increasing catalyst concentration causes an increase in the number of cavitation bubbles due to providing additional nuclei, and consequently, more HO. radicals are generated in the bulk solution (Fan et al., 2022; Karim and Shriwastav, 2020). Nevertheless, for doses greater than 1.5 g/L, more catalyst aggregation will occur and the number of active sites decreases that resulting in inhibition of light penetration, and the loss of active sites depends on shielding effects, generating HO. in the solution (Boutra et al., 2021). Furthermore, Hapeshi et al., (2013) declared that the ultrasound waves are scattered due to the use of a sonocatalyst in the system resulting in a lower amount of energy. Less amount of ultrasonic irradiation could be passed into the system because of the dominancy of the scattering of sound waves, in the presence of a higher dosage of catalyst.

**Table 5.** S/N response table for response parameters in SPC system (%)

COD Removal (%)									O&G Removal (%)						
	Ст	UV <sub>T</sub>	C <sub>c</sub>	pН	C <sub>H2O2</sub> (mmol/L)	t (min)		Ст	UV <sub>T</sub>	Cc	рН	C <sub>H2O2</sub> (mmol/L)	t (min)		
Level	Α	В	С	D	E	F	Level	Α	В	С	D	E	F		
1	27.05	29.96	29.29	30.51	30.4	29.79	1	38.39	38.21	38.44	37.12	38.43	38.98		
2	32.62	30.2	30.41	30.08	30.38	29.29	2	38.51	38.66	38.60	39.24	38.74	37.59		
3			30.57	29.68	29.49	31.19	3			38.33	39.01	38.2	38.8		
Delta	5.57	0.24	1.28	0.84	0.92	1.91	Delta	0.11	0.45	0.27	2.12	0.55	1.39		
Rank	1	6	3	5	4	2	Rank	6	4	5	1	3	2		

Table 6. ANOVA results for performance parameters in SPC system

•	Sum of squares	Degree of	Mean	F	p value	Contribution		Sum of	Degree of	Mean	F	р	Contribution
Source		freedom	square	value		(%)	Source	squares	freedom	square	value	value	(%)
COD remova	ıl (%)						O&G ren	noval (%)					
A	3691.21	1	3691.21	230.2	0.000	79.14	Α	22.92	1	22.92	0.57	0.459	0.51
В	87.63	1	87.63	5.46	0.028	1.88	В	145.24	1	145.24	3.59	0.070	3.22
С	76.97	2	38.49	2.4	0.111	1.65	С	53.22	2	26.61	0.66	0.527	1.18
D	83.87	2	41.93	2.61	0.093	1.8	D	2420.67	2	1210.34	29.93	0.000	53.59
E	42.74	2	21.37	1.33	0.282	0.92	E	118.66	2	59.33	1.47	0.250	2.63
F	280.65	2	140.32	8.75	0.001	6.02	F	745.32	2	372.66	9.22	0.001	16.5
Error	400.92	25	16.04			8.6	Error	1011	25	40.44			22.38
Lack-of-Fit	375.52	22	17.07	2.02	0.311	8.05		1010.62	22	45.94	357	0.000	22.37
Pure Error	25.39	3	8.46			0.54		0.39	3	0.13			0.01
Total	4663.98	35				100	Total	4517.04	35				100
R <sup>2</sup> =0.9140		R <sup>2</sup> <sub>(pred)</sub> =0.879	97	R <sup>2</sup> (adj)=0	.8218		R <sup>2</sup> =0.535	<b>i</b> 9	R <sup>2</sup> <sub>(pred)</sub> =0.686	57	R <sup>2</sup> (adj)=0	.5359	

# 3.4.4. pH effect

As given in Figure 2(a), lower pH improved better removal of COD than a higher pH level; the highest S/N ratio was obtained at pH 2 under acidic conditions. The pH of the solution influenced the electrostatic attraction and repulsion of sono-photocatalyst which resulted with decreasing in the COD removal rate at higher pH values (Wei *et al.*, 2021). Previous research stated that the pH value of point of zero charges ( $pH_{zpc}$ ) of the TiO<sub>2</sub> and ZnO

were approximately  $\approx 6.50$  and 9.0, respectively indicating that the surface of the TiO<sub>2</sub> and ZnO will be positively charged in an acidic medium (pH<6.50 and pH<9.0) while the catalyst's surface will carry negative charges in alkaline medium (pH>6.5). Therefore, at the initial pH of 2, while the photocatalyst surface is positively charged by absorbing H<sup>+</sup> ions, the pollutant molecules in the poultry slaughterhouse wastewater will be negatively charged and decomposed rapidly (Soltani *et al.*, 2016). Further, in acidic

media (2.59 V at pH 0), the oxidizing potential of HO. is higher than in alkaline media (1.64 V at pH 14) (Mullapudi et al., 2020). Hence, the main reason for the marginal COD decreases at higher pH values could be result of lower oxidation potential and generation rate of hydroxyl radicals (Ayare and Gogate, 2020). On the other hand, the maximum O&G removal was obtained at pH 6 (close to neutral pH) given in Figure 2(b) (F=29.93, p=0.000, p< $\alpha$ ). Potrich et al., (2022) stated in their study that O&G removal from raw poultry slaughterhouse wastewater was 99% for the iron electrode and 97% for the aluminum electrode under a 6.2 pH and a time of 20 min.

## 3.4.5. H<sub>2</sub>O<sub>2</sub> effect

 $H_2O_2$  is a strong oxidizing agent, although it increases the rate of photo-oxidation, as seen in Figure 2(a), it could not have a statistically significant effect on the COD removal efficiency (F=1.33, p=0.282, p> $\alpha$ ). Due to the scavenging effect that is given in the following equation, the excess  $H_2O_2$  would react with HO. and COD removal is inhibited (Patidar and Srivastava, 2021):

$$H_2O_2 + HO. \rightarrow H_2O + HO_2^{\bullet}$$
 (3.3)

$$HO_2^{\bullet} + HO. \rightarrow H_2O + O_2$$
 (3.4)

However, combining of SPC process with  $H_2O_2$  enhanced the degradation of O&G at 5 mM as shown in Figure 2(b). An ultrasound would enhance O&G degradation by dissociation of hydrogen peroxide into (HO.) radicals (Hinge *et al.*, 2016). The rate of degradation falls from 5 mM to 10 mM because after 5 mM due to the scavenging effect rather than supplying additional radicals (Mishra and Gogate, 2011).

# 3.4.6. Operation time effect

Operation time is one of the most effective factor in the COD and O&G removals by SPC method as illustrated in Figure 2(a-b) and Table 6 (p=0.001, p< $\alpha$ ). When the operation time of SPC process increased from 30 to 105 minutes due to higher generation of hydroxyl radicals, sharply decreased trends in COD and O&G removals were achieved, while the COD removal performance reached a maximum of 180 minutes. Also, the S/N of the O&G removal increased from 105 min to 180 min due to possible degradation of intermediate products and oil molecules. Ultrasound could be considered an alternative method that enables and accelerates to degrade of high organic strength wastewater for the first time combined with photocatalyst and UV irradiation to treat PSWW and this is consistent with work done by Abdelhay et al., (2020) reported that the removal efficiency of COD from PSWW continued to increase during the sonication time (180 min). Çancı and Kılıç (2020) declared that the COD removal rate from rose processing wastewater decreased with increased reaction time due to the formation of colored intermediates as a result of oxidation. In addition, the reaction rate was reduced because of competition between these colored intermediates and main molecules. According to obtained results, the recalcitrant target compounds could be decomposed in the first 30 min by combining ultrasound and photocatalysis processes. However, the maximum COD removal efficiency was obtained at 180 min which proved the necessity of a long time for the degradation of generated high concentration of intermediate products that cannot be further oxidized by HO. radicals, thus, accumulated in the system (Al-Bsoul et al., 2020). Furthermore, as given in Table 3(b), ultrasound enabled to break of O&G besides oxidation and destruction of aromatic compounds in PSWW for 30 minutes in the presence of photocatalyst and UV irradiation. It has been observed that the ultrasound process combined with the photocatalysis process shortens the oil grease removal time from PSWW. Coha et al., (2021) explained in their study that radical-radical recombination and dissipation of ultrasonic energy are improved by the elevated concentrations of reactive radicals such as HO., H<sup>o</sup>, O<sub>2</sub>o, etc. Nevertheless, such recombination may be inhibited and secondary radicals such as CO<sub>3</sub>•-, Cl<sub>2</sub>•-and •NO<sub>2</sub> may be produced that degrade pollutants and may form harmful byproducts by the increased concentration of anions. In this study, as shown in Figure 2(b), the performance of the SPC to remove O&G from PSWW could be thought to reduce the production of secondary pollutants and by-products as a result of a breakdown of oil molecules. Emerick et al., (2020) indicated in their study that 90% of O&G in swine slaughterhouse wastewater was removed electrocoagulation-flotation (ECF) using an Al electrode in 60 minutes, although 97.5% of O&G was removed by ultrasound and electrocoagulation-flotation (ECF/US) treatment in 25 min. The ECF treatment performance and O&G removal rate were increased by ultrasound irradiation because free radicals (HO., H<sup>o</sup>, O<sub>2</sub><sup>o</sup>) that are formed by ultrasound power helped to degrade organic pollutants (Emerick et al., 2020).

## 4. Conclusion

In this work, for the first time, the removal efficiencies of COD and oil and grease from PSWW were conducted by a hybrid technology sono-photocatalysis. Taguchi's experimental design based on an  $L_{36}$  orthogonal array (OA) was adequately applied to optimize the process. The key results of this study could be summarized as:

- The synergy index value for the combined US/UVC<sub>254</sub>/ZnO was attributed to 1.42 which performed higher oxidation performance of PSWW when compared with individual advanced oxidation processes.
- The optimum conditions obtained from Taguchi experiments for the SPC process under the following conditions: 2.5 g/L ZnO under UVC<sub>254</sub> at pH 2, 180 min for COD removal; for O&G removal, they were 1.5 g/L ZnO at pH 6 under UVC<sub>254</sub>, with 5 mmol/L of H<sub>2</sub>O<sub>2</sub>, and 30 min. With these optimum conditions, the best attained COD and O&G removal yields were 54% and 99%, respectively. Since the allowable COD and O&G concentrations for slaughterhouses according to Turkish legislation are 250 mg/L and 30 mg/L, respectively for a composite sample of 2h, hence, this step of hybrid treatment should be implemented in the primary treatment of recalcitrant

- organic substances or tertiary treatment process for the complete organic pollutants.
- According to the ANOVA results, while catalyst type was of great importance in COD removal yield, pH had the most significance in O&G removal, for the SPC process.
- ZnO showed an excellent COD and O&G removal performance in comparison to the TiO<sub>2</sub> due to absorption of more light quanta as the quantum efficiency by adsorption and photocatalysis mechanisms. Further, UVC radiation showed statistically significant degradation performance in comparison with UVA owing to shorter wavelengths with higher energy. The presence of H<sub>2</sub>O<sub>2</sub> performed a slight effect on the enhancement of O&G removal from PSWW, although COD removal efficiency decreased due to the scavenging effect in the presence of H<sub>2</sub>O<sub>2</sub>.
- The maximum COD removal efficiency was obtained at 180 min which proved the necessity of a long time for the degradation of generated high concentration of intermediate products that cannot be further oxidized by HO. radicals, thus, accumulated in the system. Ultrasound enabled to break of O&G and shortened the removal times besides oxidation and destruction of aromatic compounds in PSWW for 30 minutes in the presence of photocatalyst and UV irradiation.

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