Photocatalytic Activity of Polyaniline/TiO$_2$/ZnO Composite for Degradation of Aromatic Compounds in Abattoir Wastewater

Thabo J. Brooms, Maurice S. Onyango and Aoyi Ochieng

Abstract—The present study was undertaken to evaluate the degradation of phenol and p-cresol as recalcitrant aromatic compounds in abattoir wastewater using TiO$_2$/ZnO composite modified by polyaniline (PANI). Cleaning detergents and residual blood were the main sources of organic matter in wastewater. The modified method used to prepare the composite was “in situ” chemical oxidative polymerization of aniline and was tested on annular reactor fitted with 25W UV-C lamp. High resolution transmission electron microscopy (TEMEDX), Fourier-transform infrared spectra (FT-IR) and UV-visible spectroscopy were carried out to characterize the photocatalysts modified with PANI. It was found out that the modification did not alter the crystalline structure of the TiO$_2$ and ZnO particles according to the XRD patterns. Ratio of about 0.5:1:1 (PANI/TiO$_2$/ZnO) mass loading and flow rate of 45 ml/min were the optimum conditions for phenol and p-cresol degradation with about 99.2% and 97% in 10 h of irradiation time. Chemical oxygen demand (COD) reduction efficiency of about 38% and 88% in 10 h of irradiation time was recorded for TiO$_2$/ZnO and PANI/TiO$_2$/ZnO composites, respectively. The study showed that PANI-modified catalyst has a potential for degradation of recalcitrant aromatic compounds in abattoir wastewater.

Keywords—Abattoir wastewater, photocatalytic degradation, polyaniline, TiO$_2$/ZnO.

I. INTRODUCTION

The slaughterhouse (abattoir) wastewater is characterized by high organic compounds and nutrients concentrations, either in a particulate or dissolved matter [1]. This industry produces wastewater with high pollutant loading consisting of pauch, manure, animal fat, uncollected blood, oils and feces [1,2]. The sources of these contaminants are mostly from slaughteryard, stalls, cleaning detergents and disinfection solutions used for cleaning activities and disinfecting the slaughterhouse facilities [2,3]. Some of the disinfecting chemical solutions contain substances such as aldehydes, phenols, cresols of which are for destruction of microorganisms, and peroxide compounds (microorganism cell penetration) [4]. Typical concentrations of chemical oxygen demand (COD) and suspended solids (TSS) found in abattoir wastewater ranged from 2333-8627 mg/l and from 736 to 2099 mg/l, respectively [4]. Furthermore, the residual blood and feces are regarded as the principal sources of organic matter found in abattoir wastewater [5]. In order to meet and achieve compliances with state and central environment regulations, an effective treatment of this wastewater with such pollutants is essential prior to discharge from the plant. The treatment of these wastewater will necessary eliminate the critical effects on the environment and human health.

Anaerobic and aerobic technologies have been regarded as the effective treatment methods for abattoir wastewater in removing COD above 90% [2,6,7]. Kobya et al. [8] reported that, even though aerobic process is reliable in the treatment of slaughterhouse wastewater, however, it lacks potential for treatment of high COD, BOD, total nitrogen (TN) and pathogens concentration in the treated effluent. As for anaerobic, the process biodegrade organics with high concentration, reduces odour while generating high amount of biogas. However, technologies such as advanced oxidation processes (AOP) especially photocatalysis using UV-Vis light and solar systems, has not been given much attention for treatment of abattoir wastewater as far as the literature is concern. Cao and Mehrav [9] used UV/H$_2$O$_2$ integrated with anaerobic digestion for treatment of synthetic slaughterhouse wastewater and about 98% COD and 95% TOC was removed, respectively. While Louvet et al. [5] reported the use of flourescene and UV-Visible absorbance in monitoring the biodegradation of slaughterhouse wastewater using sequential bioreactor (SBR).

In addition, AOP provides an efficient and economical method that is able to decompose the organic pollutants using TiO$_2$, WO$_3$, ZnO and SnO$_2$ into harmless matter [10, 11]. Lately, an intense conducting polymer supporting material such as polyaniline (PANI) or polypyrrole (Ppy)), has been a preferred supporting material to the catalysts (TiO$_2$ and ZnO ) due to the decolorized conjugated structure in electron-transfer processes and a photosensitizer of catalysts [12,13]. However, PANI has been a preferred polymer due to the fact that it is cheaper than other conducting polymers, high...
environmental stability, and good electrical properties and it is also able to form a synergy or complementary behaviour with inorganic matrices [14]. According to previous reports, the support of PANI on catalysts remarkably improves photocatalytic activity of the catalyst under visible light. In the present study, PANI-modified TiO$_2$/ZnO was used for the first time to degrade phenol and p-cresol as recalcitrant aromatic compounds in abattoir wastewater under UV-light illumination. The aim of the work was to apply newly produced PANI/TiO$_2$/ZnO photocatalyst to degrade the aromatic compounds into harmless product(s), reduce colour of the residual blood and COD content of the wastewater.

II. EXPERIMENTAL

A. Materials

TiO$_2$ (Degussa P-25, Aerioxide) with BET surface area (50 m$^2$/g) was purchased from Acros Organics (South Africa). Aniline monomer (Reagent Plus, 99%) and ammonium peroxodisulfate (APS, (NH$_4$)$_2$S$_2$O$_8$) were purchased from Sigma-Aldrich (South Africa). ZnCl$_2$ (min. 98%), NaOH (pellet min. 98%), HCl (35%) was received from Merck (Germany). A 25W UV-C Germicidal Lamp from Philips (Ltd), was supplied by Technilamp, South Africa. All chemicals were used as received without undergoing any pre-treatment or purification.

B. Characterization of Abattoir Industrial Water

Abattoir wastewater was sampled from Boekenhout abattoir (Sheep) plant, North of Pretoria, South Africa. An influent (inlet) volume of about 5L pre-treated abattoir wastewater of residual blood was collected after undergoing screening (fats, nails, hair and bones etc.) process from the abattoir meat processing plant before discharged into the wastewater reservoir. In the laboratory, wastewater was screened again to remove solids that are larger than 0.5mm using Whatman Filter Paper with pore size (2.5 µm). After that, the wastewater was stored at 4°C until required for use. Table 1 shows a detailed characterization of abattoir industrial wastewater.

<table>
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<th>TABLE I</th>
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<td>CHARACTERIZATION OF ABATTOIR WASTEWATER FROM AN INFLUENT (INLET) EMPLOYED</td>
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<td>Phenol (mg/l)</td>
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<td>BOD$_5$ (mg/l)</td>
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<td>Phosphates (mg/l)</td>
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Symbols: (-) mean not analysed/detected, $^a$ Bustillo et al.[17], $^b$ Caixeta et al. [18].

C. Preparation of ZnO

Metal oxide (ZnO) was prepared by dissolving ZnCl$_2$ (20g) in 200 mL water at 80 ºC together with 5 ml of HCl, at a stirring speed of 250 rpm for 2 h. An aqueous solution of 0.1M NaOH was added dropwise until stable precipitate of Zn(OH)$_2$ was formed. The supernatant dispersion solution was separated from particles by sedimentation for 2 h. Precipitate from supernatant solution was washed 3-4 times with deionised water to remove chloride. The precipitate was filtered (Whatman filter paper, pore size; 2.5 µm) and washed with deionised water until free from chloride and dried at 70°C, calcined at 250 ºC and white powder of ZnO was obtained. The method of preparation of ZnO was adopted from Maroni et al. [15]. A simple TiO$_2$/ZnO (1:1) blend mixed oxide was prepared through mechanical mixing. One gram of each TiO$_2$ and ZnO was accurately weighed and well mixed using pestle and mortar with few drops of deionised water (solvent) for preparation of a paste. The mixture was dried at 70°C for 1h. Thermal treatment of TiO$_2$/ZnO was performed using pre-heated muffle furnace at calcination temperature of 450 ºC for 5 h.

D. Preparation of Polyaniline/TiO$_2$/ZnO

PANI preparation method using ammonium persulphate (APS) as oxidant was adopted as described by Wang et al. [1]. Polyaniline modified TiO$_2$, ZnO and TiO$_2$/ZnO composites were prepared in-situ by a chemical oxidative polymerization method. Various known amounts of aniline to APS were mixed and dissolved in 0.5M HCl (10 ml) followed by 2g (TiO$_2$/ZnO; 1:1 mass ratio) under stirring condition (250 rpm) into 200 mL deionised water. The polymerization process was allowed to proceed for 24 h at 25 ºC and the dark blue-green precipitated polyaniline salt was recovered from the polymerization vessel, filtered, and the precipitate formed were washed with small amount of acetone. The resulting polyaniline salt was finally dried for 24 h at 55°C. The bare catalysts were TiO$_2$/ZnO composite photocatalysts with different molar ratios of PANI/TiO$_2$/ZnO (0.1:1:1 to 5:1:1) were used and deposited by PANI. In this way, a series of different composites was prepared and labelled as PANI/TiO$_2$/ZnO (0.1:1:1), (0.5:1:1), (1:1:1), (2:1:1) and (5:1:1). In order to confirm effect of PANI in the composites materials of the bare catalysts and that of the mixed-blend catalysts, the bare metal oxides were treated in the same procedure as that of composites except in the presence of the aniline.

E. Analytical Methods

A Shimadzu UV-Visible 1800 Spectrophotometer (Japan) was employed to quantify phenol and p-cresol at wavelength 270 nm 277 nm, respectively. A calibrated pH meter (Hanna 675, Japan) was used to adjust the pH condition of the wastewater. The parameters measured throughout photocatalytic degradation experiments included Chemical
Oxygen Demand (COD), Biochemical Oxygen Demand (BOD₅) and Total Suspended Solid (TSS) were determined on adaptation of the US Environmental Protection Agency (USEPA) 410.4 approved method [16]. Moreover, nitrates and phosphates were determined through the use of Ion Chromatography (IC) instrument (Swiss Compact 882, Metrohm, USA).

F. Measurement of Photocatalytic Activity test

The total volume capacity of the photoreactor (annular) was about 450 ml in the reactor as shown in Fig. 1. The total height was 330 mm, and the internal diameter was 26 mm, thickness of the photoreactor wall was 2 mm and the capacity of the UV lamp was 25 W UV-C. The inner (length, 451.6 mm, diameter, 28 mm) shield is made of quartz glass (quartz) to allow full penetration of the UV-rays, while the entire outer fabrication was of Perspex. At the bottom of the reactor (the inlet section) had distribution compartment fitted with a distribution plate which has 34 evenly distributed holes each of 1 mm in diameter. The radial length of the annular space was 8 mm. The horizontal irradiation distance of the photoreactor was 1.2 mm. Total recirculation volume of the feed solution was 1 L. Before irradiation, suspension was stirred magnetically for 1h in the dark conditions until adsorption-desorption equilibrium was established. The recirculation flow of the solution through reactor was regulated by a peristaltic pump (Masterflex, Head 77916-20, Cole-Parmer instruments, Canada) and operated under ambient temperature. The pH of the solution was adjusted using 0.1M NaOH or HCl aqueous solution. Suspension of the wastewater with modified catalyst was stirred at 650 rpm using magnetic stirrer (Heidolph MR 1000) to keep it homogeneous. Sample solution withdrawals for all experimental during photocatalytic process were filtered using 0.45µm Nylon Membrane Filters. The concentrations of aromatics detected (phenol and p-cresol) during irradiation process were analyzed through UV-Visible Spectroscopy method. Percentage degradation efficiency of aromatics was estimated by:

\[
\text{Degradation (\%) } = \left( \frac{C_0 - C}{C_0} \right) \times 100
\]

where \(C_0\) represents the concentration of the phenol and p-cresol before illumination, \(C\) denotes the concentration of the phenol and p-cresol after a certain irradiation time, respectively.

III. RESULTS AND DISCUSSION

A. Fourier Transformer Infra-Red (FTIR)

Figure 2 showed the comparison of the FTIR transmission spectra of photocatalysts and PANI-modified composites. The main characteristic peaks of PANI (a) were assigned from the bands at 1497 cm\(^{-1}\) (attributed to N–H stretching mode, C=C and C=N for quinonoid rings) and 1630 cm\(^{-1}\) (attributed to N–H stretching mode, C=C and C=N stretching mode for benzenoid rings). The broad sharp peaks between 3419 cm\(^{-1}\) and 3508 cm\(^{-1}\) for (b) are attributed to NH stretching vibration (due to high hydrogen bonded OH stretching mode). The new weak peak appearing at 663 cm\(^{-1}\) is probably due to C-H out of plane bending vibration of which is based on the benzene ring deformation on PANI chain. The small peak of PANI at 3419 cm\(^{-1}\) was assigned to N–H stretching vibration.

Zhang [19] reported that there is a strong interaction of H-bonding between PANI and (b) particles. Meanwhile, stretching bands at 1328 cm\(^{-1}\) is assigned to C–N for benzenoid unit, while the band at 1178 cm\(^{-1}\) is assigned to C–N for quinonoid unit of PANI. The shift to lower wavenumbers was observed at 1497 cm\(^{-1}\) and 1360 cm\(^{-1}\) corresponding to the stretching C=C and C=N, to about 1492 cm\(^{-1}\) and 1630 cm\(^{-1}\), respectively. The shift or the differences in the spectra was due to the constrained growth of the restricted modes of vibrations in PANI when in-cooperated with (b) [20].
Fig. 2. FTIR spectra of photocatalysts and PANI-modified composites

B. Transition Electron Microscopy (TEM)

Figure 3 (Ι) clearly shows the HRTEM images of catalysts and their PANI-modified composites. The images indicated that PANI-modified on TiO$_2$/ZnO in sample (D) did not change the size of blend material as depicted in (C) and (D), respectively. Moreover, ZnO marked as sample B, showed a clear rodlike hexagonal structure with a particle size of about 200 nm and diameter of about 50 nm, similar observations were reported by Dhingra et al. [13]. The sizes of neat TiO$_2$ (A) and TiO$_2$/ZnO (C), were monodispersed at particle size ranging from 20 to 30 nm. The aggregations of all PANI composite particles were caused by high surface energy; however, agglomeration of the modified PANI composite was elevated as compared with that of the neat catalyst. As for (D), the presence of ZnO particle could not be observed even at higher magnification due to PANI which shielded almost its entire surface. Moreover, Fig. 4 labelled as (FF), showed the elemental compositions (EDX) analysis spectrum of constituents present in the PANI-modified TiO$_2$/ZnO after 10 h of irradiation. An EDX spectrum showed that in addition to H element, the composite composed of N, C, O, Ti and Zn elements. The contents of titanium (Ti), zinc (Zn) and oxygen (O) elements confirm that TiO$_2$/ZnO particles are coated by PANI partly. As for chlorine (Cl), the atom came from HCl during preparation of PANI composite. Traces of sulfur (S) and phosphorus (P) atoms resulted from APS reagent, while calcium (Ca), silicon (Si) and iron (Fe) found in trace amounts, resulted from composition of abattoir wastewater as nutrients. Cu atoms depicted were due to copper grids found during performance of TEM during analysis.

C. Photocatalytic degradation of aromatic compounds under UV-visible illumination

The abattoir wastewater was characterized by COD content of 3603 mg/l and BOD$_5$ 1950 mg/l and the values are shown in Table 1. The absorbance spectra for abattoir wastewater indicating haemoglobin, were similar to those reported by Sutherland-Stacey et al. [21] and [5] except for aromatics (phenol and p-cresol). The presence of aromatics could have possibly been due to cleaning detergents and disinfectant that were used for cleaning residual blood after slaughtering [4]. The presence of these aromatics were concentrated on the inside walls of the pipe leading to effluent. Photocatalytic treatment of abattoir wastewater using PANI/TiO$_2$/ZnO (0.5:1:1) showed degradation of about 99.2% and 97% for phenol and p-cresol, respectively during 10 h of UV-light degradation at pH 7.21. Lower molar ratios of aniline (PANI) to TiO$_2$/ZnO lead to high degradation of these pollutants due to thin PANI coating which allowed enough penetration of light into the catalyst surface [18] and the ratio activities of aniline (PANI) to TiO$_2$/ZnO are shown in Fig. 6. The difference between the two pollutants with regard to degradation efficiency has to do with differences in molecular weight since p-cresol (108 g/mol) is a derivative of phenol (94 g/mol). The presence of haemoglobin resulting from residual blood waste at peak (410-460 nm) nm, was completely degraded as seen in both Fig. 4 (phenol and p-
resol), even though it was never studied. Furthermore, the concentration of phenol and p-cresol was measured by UV-vis spectroscopy at wavelength of 270 nm and 277 nm, respectively, and the intermediate exhibiting absorbance at the same wavelength was considered. According to the studies by Bitenc et al. [22], the possible reaction intermediates affects the determined concentration of organics and are also adsorbed onto the catalyst surface until simple molecules, CO₂ and H₂O are formed. However, the intermediate could be adsorbed within the examined UV-visible spectra especially since the PANI-modified had an efficient adsorbing capacity of the pollutants as indicated during blank experiment on both phenol and p-cresol degradation spectra. Therefore, we used the UV-spectra as the simplest indication for determining/verifying the mineralization of aromatic compounds in abattoir wastewater after photocatalytic treatment under UV-visible region.

![Absorbance](image1)

**Fig. 4.** Ultraviolet-spectra for phenol and p-cresol on photocatalytic treatment of abattoir wastewater using PANI/TiO₂/ZnO composite.

### D. Photocatalytic Activity of PANI Composites for Aromatics Degradation in Abattoir Wastewater

The effect of aniline (PANI) molar ratio on TiO₂/ZnO composite for degradation of aromatic compounds present in abattoir wastewater at pH 7.21, was studied (Fig. 5). The degradation of aromatic compounds in the absence of catalyst indicated a very low degradation percentage of about 18% and 12% in 10 h of irradiation time on phenol (A) and p-cresol (B), respectively. As for TiO₂/ZnO blend, photodegradation of about 76% 54% for phenol and p-cresol, respectively, was achieved. This low photodegradation of phenol and p-cresol can be assumed that there was low charge separation and high electron recombination of TiO₂/ZnO during photocatalytic treatment of aromatics in abattoir wastewater. In addition to photocatalytic activity of TiO₂/ZnO on aromatic compounds, the support of TiO₂/ZnO with PANI seemed to have improved the photodegradation of the aromatic compounds due to its polymeric conductive characteristics. The result showed that introduction of PANI ratio (0.5 to 2) to TiO₂/ZnO blend decreases the photocatalytic activity of PANI/TiO₂/ZnO for degradation of both phenol and p-cresol as aromatic compounds. However, further increase in PANI ratio (2 to 5) lead to a slight low photocatalytic activity of the PANI/TiO₂/ZnO composite. This decrease in photocatalytic activity can be explained in terms of different transferring rates of the photoinduced carriers and electron recombination and electron-holes pair when different amounts of PANI are coated [23]. Furthermore, photocatalytic degradation of phenol and p-cresol were highly enhanced with the least aniline (PANI) molar ratio of about 0.5/TiO₂/ZnO giving 99.2% and 97%, respectively, while the highest aniline ratio of about 2/TiO₂/ZnO and 5/TiO₂/ZnO gave about 86% and 77%, respectively. Min and Wang [24] reported photocatalytic mechanism coated with PANI and confirmed that the delocalized conjugated structures of PANI enhanced the charge separation efficiency by transferring photoinduced carrier. In accord with the mechanism,

![Absorbance](image2)

**Fig. 5.** Photocatalytic degradation of (A) phenol at 101ppm concentration and (B) p-cresol at 106ppm concentration in abattoir wastewater on different PANI ratios for PANI/TiO₂/ZnO composite under UV-Vis illumination.
The excited electrons generated by $\pi-\pi^*$ transition of PANI can be delivered into conduction band of TiO$_2$/ZnO to the adsorbed electron acceptor, thus yielding powerful oxidant (hydroxyl radicals) to degrade the aromatics in abattoir wastewater. According to our study findings, the synergy effect between the PANI and TiO$_2$ or ZnO composite caused a rapid charge separation and predicted much slow charge recombination. Therefore, it can be concluded that low aniline (PANI) ratio to TiO$_2$/ZnO blend, has high degradation impact for treatment of abattoir wastewater even in the presence of matrices which can act as inhibitors or radical scavengers during photodegradation processes.

E. Removal of Organic Matter during Photocatalytic Degradation in Abattoir Wastewater

The reduction of COD content (3603 ppm) using PANI/TiO$_2$/ZnO composite in abattoir wastewater is shown in Fig. 6. The COD content was reduced from 3603 mg/l to almost 550 mg/l, and this corresponds to removal of about 88% in 10 h of irradiation time at pH 7.23. In addition, the BOD$_5$ (Table 1) was analysed in five consecutive days and the removal efficiency was about 85% in total irradiation time of about 10 h of irradiation time. It was observed that more of the COD and BOD$_5$ were removed in prolonged irradiation time at a high flow rate measurement [25]. According to the World Health Organization (WHO) wastewater discharge standard level [26], the discharge limits for nitrates and phosphates are 0.5-1.0 mg/L. However, both nitrates (8.60 mg/l) and phosphate (0.46 mg/l) were completely removed from 3 h to 10 h of degradation process. The removal in both nitrates and phosphates could have been influenced by high flow rate facilitating the adsorption of anions faster into the surface of the catalyst.

![Fig. 6. COD measurements for photocatalytic treatment of abattoir wastewater.](image)

However, the high COD reductions indicated that PANI/TiO$_2$/ZnO composite did not attribute any hydroxyl radical scavenger, but, only hydroxyl radicals resulted from reaction mechanism with water molecules from wastewater treated. Cao and Mehvar [25] reported the use of UV/H$_2$O$_2$ in abattoir wastewater for removal of COD and found that H$_2$O$_2$ acted as both oxidant and hydroxyl radical scavenger when the concentration of H$_2$O$_2$ was increasing and decreasing during photocatalytic process. This study indicated that PANI/TiO$_2$/ZnO composite photogenerated abattoir wastewater quite higher during degradation of organics in the absence of oxidants which could have acted as hydroxyl radical scavengers.

F. Decolourization of Abattoir Wastewater during Photocatalytic Degradation.

The decolourization of abattoir wastewater during photodegradation at varying irradiation time assumed to have reached complete mineralization as shown in Fig. 14. Abattoir wastewater displayed a partially dense red colour with COD content of 3603 ppm and the wastewater began decolorizing after 1 h of adsorption equilibrium (blank) test was done. However, the COD was reduced lower as compared to colour reduction which leads to almost mineralization (clear colour). A clear decolourization was observed from 6 h to 10 h when using PANI/TiO$_2$/ZnO composite.

![Fig. 7. Decolourization image of abattoir wastewater after photodegradation using PANI/TiO$_2$/ZnO composite.](image)

G. Reusability of PANI modified Catalyst for Photodegradation of Abattoir Wastewater

Figure10 showed the reusability graph of PANI/TiO$_2$/ZnO on a number of recycling operations during treatment of abattoir wastewater. During cycle 1, it was found that degradation of abattoir wastewater was 93% and 53% for cycle 2, respectively, at 10 h of irradiation time. However, further reusability of PANI/TiO$_2$/ZnO lead to a decrease in photocatalytic activity of PANI/TiO$_2$/ZnO for degradation of wastewater as seen for cycle 3 (39%) and cycle 4 (29%), respectively. Li et al [18] reported that a decrease in photocatalytic activity after each cycle was due to slight aggregation of particle and matrices on the surface of the catalyst, and this prevents excitation of photos during photocatalytic degradation process. This interpretation can be linked according to the composition of constituents found during HRTEM-EDX elemental analysis as seen in Fig. 4 (E). Therefore, it can be concluded that, the reusability of PANI composite in abattoir wastewater is not recommended due to its poor photocatalytic activities for removal of organic compounds.
This study related the photocatalytic degradation of organic matter in abattoir wastewater using PANI-modified TiO$_2$/ZnO composite. The performance of the composite gave removal efficiencies of about 85% and 88% for BOD$_5$ and COD at maximum irradiation time of about 10 h at flow rate of 45 ml/min. The low ratio of aniline (PANI) to TiO$_2$/ZnO blend gave a high degradation of phenol and p-cresol as compared to high aniline (PANI) ratio. The structure of the PANI-modified composite was not changed during photodegradation process in abattoir wastewater. The presence of ZnO served as an indirect oxidant enhancing the degradation efficiencies when supported by PANI/TiO$_2$ composite. Therefore, results show that an optimum photocatalyst of about PANI/TiO$_2$/ZnO (0.5:1:1) can be assumed to degrade organic pollutants in abattoir wastewater in a prolonged irradiation time.

ACKNOWLEDGMENT

The authors wish to acknowledge The Water Research Commission (WRC) of South Africa for funding the project.

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