

# **SIMULTANEOUS REMOVAL OF CU, FE, ZN AND EDDS FROM A SOIL WASHING STREAM THROUGH PHOTOCATALYTIC PROCESSES**

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**ABSTRACT** – The soil contamination is a global problem connected with illegal waste disposal due to the uncontrolled industrial development. These disposals are normally done with waste enriched by metals. One of the ways to solve the metal soil contamination problem is the soil washing. It uses a chelating agent (EDDS, EDTA i.e.) that complexes with metallic ions and carries them out. But in the end remains an aqueous solution with metals and organics at high concentration. Some reports indicate that the treatment of this solution employing traditional techniques, such as precipitation, is generally unsuitable due to the complexes stability. Therefore, the possibility to destroy the organic species (and the complexes) by means of TiO<sub>2</sub> photocatalysis has been proposed in the past for the case of EDTA. Hence, the present investigation aims to evaluate the possibility to remove Cu, Fe and Zn from synthetic and real soil washing solutions through photocatalytic processes.

## **1. INTRODUCTION**

One of the most used methods on the soil decontamination is an "ex situ" technique called soil washing or flushing. It uses organic chelating agents that are capable to extract metals from the soil (Tsang *et al.*, 2012). This technique generally causes less surface damages and removes the metallic contaminants from the soil. Therefore, after the washing it remains a waste solution containing the extracted metals and it is needed to be treated (Voglar and Lestan, 2012).

The ethylenediamine tetraacetate di-sodium salt (EDTA) is commonly used on the soil washing as the complexing chelate, thanks to its availability and relatively low cost (Park *et al.*, 2006). Unfortunately, EDTA is quite persistent in the environment due to its low biodegradability (Pitter and Sýkora, 2001; Hinck *et al.*, 1997), therefore the easily biodegradable chelating agent ethylenediaminedisuccinic acid (EDDS) has been proposed to replace the EDTA since it is safer

and environmental friendly (Zhang *et al.*, 2008). As a metal-chelate coordinated compound is quite stable, it prevents the metal precipitation and sorption. Then, this kind of waste cannot be treated using conventional physical methods, such as filtration, flocculation and precipitation. One of the alternative treatments could be the use of Advanced Oxidation Processes (AOP) since they have high removal efficiency (Rosas *et al.*, 2013), but still, little has been done about the decontamination by AOP processes of soil washing waters containing metal-EDDS complexes. It is known that some metal-aminopolycarboxylic complexes are photosensitive under sunlight irradiation and their photodecomposition may lead to the metal reduction and ligand radical formation. That is the case of Fe(III)-EDDS that, under irradiation, works like a “homogeneous photo-Fenton systems” but at neutral pH’s, enhancing the oxidative degradation of the organic ligands and promoting the precipitation of some metal ions present in the solution as metal hydroxide or as metal zero-valent state (Huang *et al.*, 2013). In some cases, an alternative route may be pursued using heterogeneous TiO<sub>2</sub>-photocatalysis, since it has been proved that, at low pH, Cu(II) ions chelated with EDDS are photoreduced to zero-valent copper which is efficiently removed from the solution (Satyro *et al.*, 2014).

In the present investigation, the possibility to simultaneously remove EDDS and metals (Cu, Fe and Zn) from synthetic and real soil washing solutions, for pH conditions close to neutrality, is explored by artificial solar homogeneous and heterogeneous photocatalytic processes.

## **2. MATERIALS AND METHODS**

### **2.1. Soil sampling and soil washing procedure**

The soil used in the study was sampled in Giugliano in Campania, in the province of Naples (South of Italy), in the core of the “Land of Fires”. The superficial samples were collected manually, covering an area of about 1m<sup>2</sup>, and stored in hermetic containers. After collection, the samples were dried (40°C), sieved (2 mm), and preserved at room temperature. Only the fraction of soil smaller than 2 mm (Gallego *et al.*, 2002) was used in the experiments and analytical determinations.

The soil washing (SW) experiments were performed in 50 mL polyethylene bottles. EDDS was used as chelating agent. The extractions were done at fixed liquid-solid ratio (L/S) (10:1) and EDDS concentration (0.36mM). The samples were stirred in a mechanical shaker (190 rpm) at ambient temperature. After 96h the samples were centrifuged at 4800 rpm for 15 min using an IEC Centra GP8R centrifuge and filtered at 1.5 µm. It was also done an experiment of soil washing without EDDS (SWB), but with the same conditions.

### **2.3. Photocatalytic treatment**

Photocatalytic runs were performed in an annular batch glass reactor equipped with a 125 W lamp ( $\lambda = 300\text{--}400$  nm, Helios Italquartz) (Andreozzi *et al.*, 2000). Before turning on the lamp, the chosen solution was purged with the chosen gas (N<sub>2</sub> or O<sub>2</sub>) for 30 min at dark. The pH of the soil washing solution was not changed, being around 7-8, and on the synthetic solutions (SS) it was

modified to this value. The metals (Cu, Fe and Zn) and EDDS concentrations in the synthetic solutions are the same found on the real soil washing solution. The samples collected at different reaction times were filtered on regenerated cellulose filters ( $\Phi_{\text{pore}}=0.20 \mu\text{m}$ ).

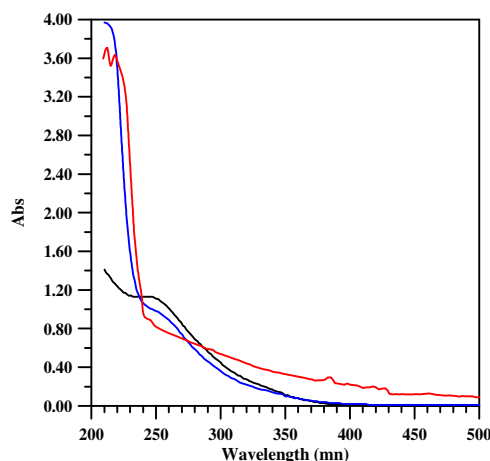
## 2.4. Analytical methods

The EDDS analyses were performed through a colorimetric method previously reported by Vandevivere *et al.* (2001) (ATI-Unicam UV/Vis spectrophotometer). The metals analysis (Cu, Fe, and Zn) were done with an Atomic Absorption Spectrometry (AAS), using Varian Model 55B SpectraAA equipped with flame (FAAS) and a GBC Avanta AAS equipped with a graphite furnace (GFAAS). Before the metal analysis the samples were digested following the EPA method 3051 [USEPA, 2001]. The UV spectra between 200 – 800nm were done in a UV ATI-Unicam UV/Vis spectrophotometer.

## 3. RESULTS AND DISCUSSION

### 3.1. UV-Vis light absorption spectra of synthetic and real soil washing solutions

UV/Vis absorption spectra of a synthetic (SS), a real soil washing with (SW) and without (SWB) EDDS solutions were recorded and compared in the Fig. 1.



**Figure 1.** Absorption spectra of synthetic solution (—), real soil washing with EDDS (—) and real soil washing without EDDS (—). SS:  $[\text{EDDS}]_0$   $3.6 \cdot 10^{-1}$  mM,  $[\text{Cu(II)}]_0$   $8.0 \cdot 10^{-2}$  mM,  $[\text{Fe(III)}]_0$  0.10 mM,  $[\text{Zn(II)}]_0$   $8.0 \cdot 10^{-2}$  mM, pH 7.5. SWB:  $[\text{EDDS}]_0$  0 mM  $[\text{Cu(II)}]_0 \sim 1.0 \cdot 10^{-3}$  mM,  $[\text{Fe(III)}]_0 \sim 4.5 \cdot 10^{-2}$  mM,  $[\text{Zn(II)}]_0 < 7.6 \cdot 10^{-4}$  mM, pH 7.8. SW:  $[\text{EDDS}]_0$   $3.6 \cdot 10^{-1}$  mM,  $[\text{Cu(II)}]_0 \sim 8.0 \cdot 10^{-2}$  mM,  $[\text{Fe(III)}]_0 \sim 7.0 \cdot 10^{-2}$  mM,  $[\text{Zn(II)}]_0 \sim 8.0 \cdot 10^{-2}$  mM, pH 7.8.

The spectra of the three samples showed a decrease of the total absorbance as the wavelength increased. However, the real solutions, including the one not containing EDDS and

with a low metal content (SWB), showed a total absorption in the wavelength of 210 to 230 nm quite higher than the SS. These results suggest that both SWB and SW contain a relatively high fraction of hydrophilic terrestrial Dissolved Natural Organic Matter (DNOM) released from the soil during the washing process (Dilling *et al.*, 2002). DNOM fraction is characterized by very high molecular weights and generally possesses phenolic, ketonic and carboxylic groups able to complex soluble metal ions (Chorover *et al.*, 2001). In particular, depending on the experimental conditions, some metal/DNOM complexes, such as Fe(III)/DNOM, undergo rapid photochemical reaction under sunlight irradiation generating reactive oxidant species (ROS) (Faust *et al.*, 1993). In the end we can say that the real solutions are much more complex than the synthetic ones.

### 3.2. Photocatalytic treatments

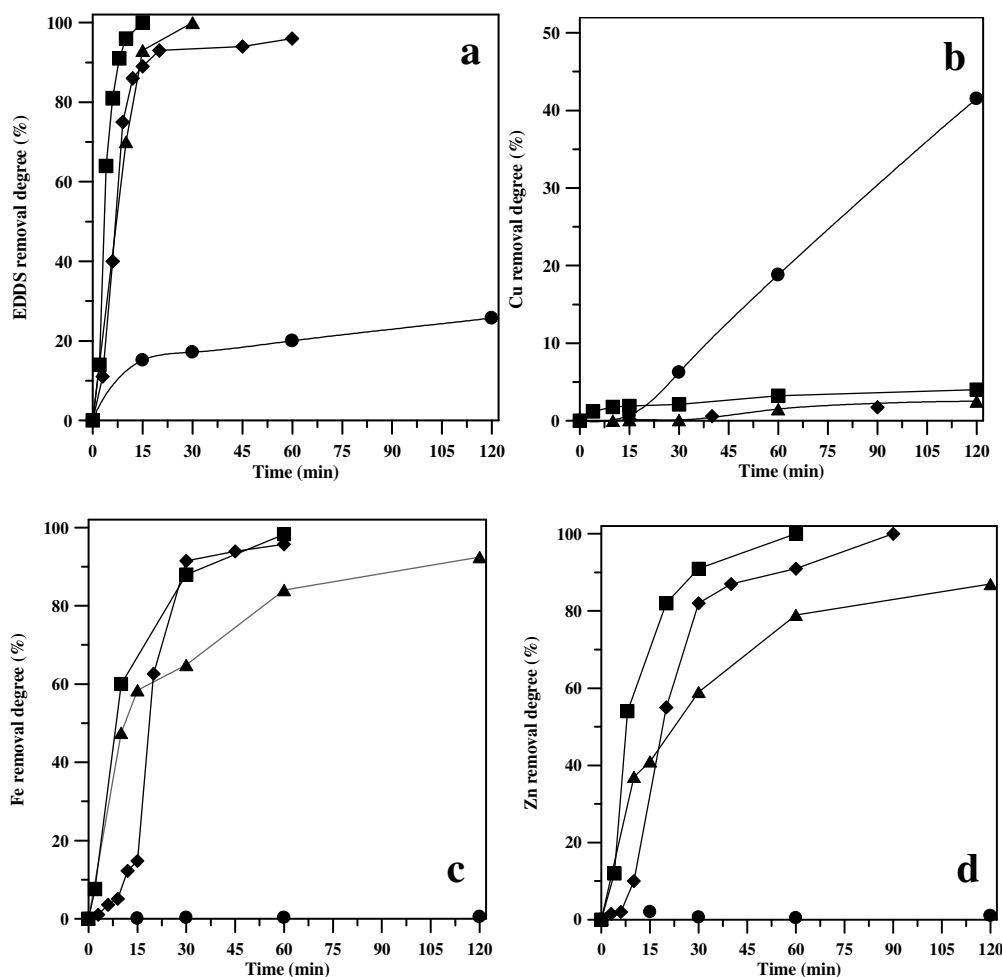
The simultaneous removal of EDDS and Cu(II), Fe(III) and Zn(II) species has been attempted by four different photocatalytic systems:  $\text{TiO}_2/\text{h}\nu/\text{N}_2$ ,  $\text{TiO}_2/\text{h}\nu/\text{O}_2$ , Fe(III)-EDDS/ $\text{h}\nu/\text{O}_2$  and Fe(III)-EDDS/ $\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$ .

#### Synthetic aqueous solution (SS) photocatalytic treatments

In the Fig. 2a-d are reported the removal degrees for EDDS and soluble Cu, Fe and Zn species from synthetic aqueous solutions by heterogeneous or homogeneous photocatalytic treatments. Preliminary runs, carried out at dark, in presence of 100 mg/l of  $\text{TiO}_2$ , evidenced no adsorption of EDDS and of the metallic investigated species on the solid surface (data not shown).

Regarding to EDDS species (Fig. 2a), it is observed a marked difference between the removal degrees achieved by  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  (25% at 120 min) system and the others processes (>90% in 30 min). The high efficiency of EDDS abatement in the  $\text{O}_2$  presence is ascribed to the generation of hydroxyl radicals, which are powerful oxidizing species, capable to rapidly destroy a very large number of organic molecules. On the contrary, adopting  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  system, EDDS is mainly removed, as previously reported (Satyro *et al.*, 2014) through the reaction with the photogenerated holes, but this process is the only one capable of removing Cu(II) species, where it is reduced through the reaction with the photoelectrons (Fig. 2b).

In the case of the Fe, it is almost complete removed on the aerated photocatalytic processes able to efficiently destroy the Fe(III)/EDDS or Fe(III)/EDDS-like complexes. This high removal level is ascribed to the precipitation of  $\text{Fe}(\text{OH})_3$  ( $K_{\text{ps}} = 1.6 \cdot 10^{-39} \text{ M}^4$  at 25 °C (Martell and Smith, 1976)). On the Zn case, it is not possible to remove it by sacrificial photocatalysis (Fig. 2d), since it is thermodynamically unlikely (Chenthamarakshan *et al.*, 2000). On the other hand, Zn removal degrees progressively increase moving from  $\text{TiO}_2/\text{h}\nu/\text{O}_2$  system to Fe(III)-EDDS/ $\text{h}\nu/\text{O}_2$  process and finally to Fe(III)-EDDS/ $\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$ . The  $K_{\text{ps}}$  value for  $\text{Zn}(\text{OH})_2$  ( $3.0 \cdot 10^{-16} \text{ M}^3$  at 25 °C (Martell and Smith, 1976)) is too high, if compared with that above reported for  $\text{Fe}(\text{OH})_2$ , to support its precipitation under the same adopted experimental conditions. However, there are indications that suggest the Zn ions reduction through the formation of organic radical intermediates which are generated by the  $\text{HO}^\cdot$  species attack to EDDS and its oxidation by-products (Somasundaram *et al.*, 2004; Kabra *et al.*, 2008).



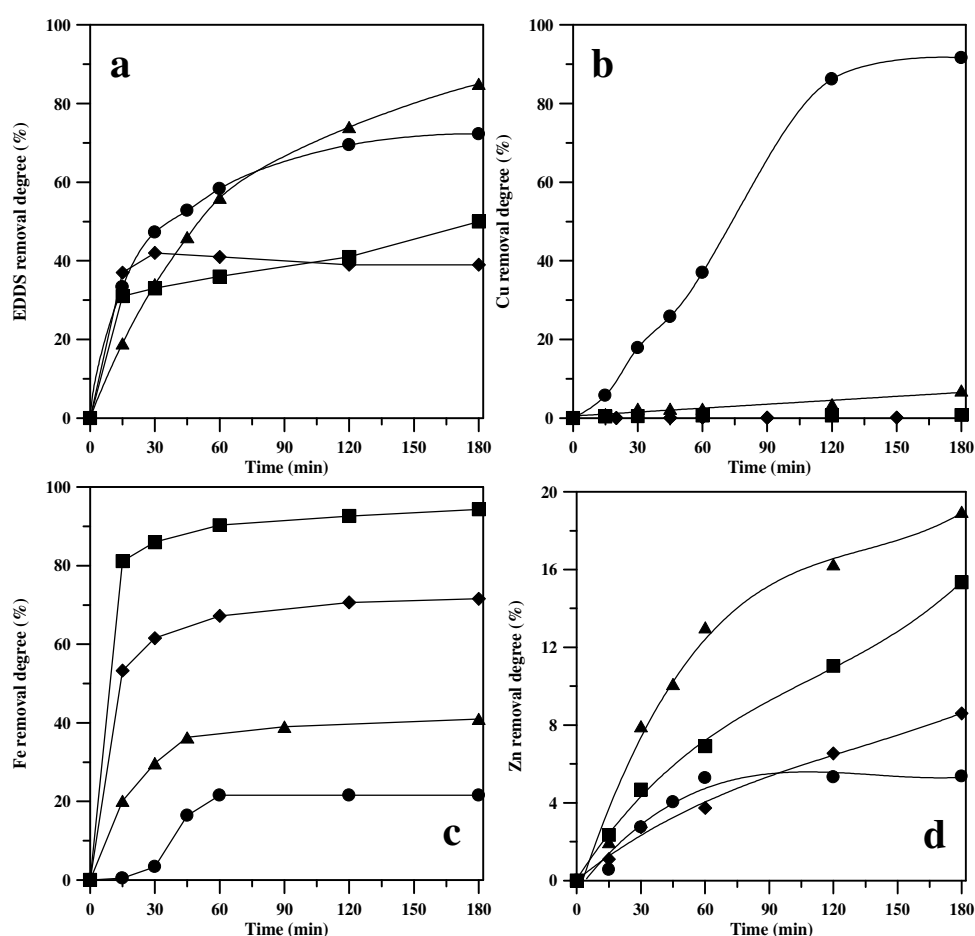
**Figura 2.** Removal of EDDS (2a), Cu (2b), Fe (2c) and Zn (2d) from synthetic aqueous solutions by  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  (●),  $\text{TiO}_2/\text{h}\nu/\text{O}_2$  (▲),  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{O}_2$  (◆) and  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$  (■).  $[\text{EDDS}]_0$   $3.6 \cdot 10^{-1}$  mM,  $[\text{Cu(II)}]_0$   $8.0 \cdot 10^{-2}$  mM,  $[\text{Fe(III)}]_0$   $1.0 \cdot 10^{-1}$  mM,  $[\text{Zn(II)}]_0$   $8.0 \cdot 10^{-2}$  mM.  $\text{TiO}_2$  initial load: 100 mg/l,  $[\text{H}_2\text{O}_2]_0$  5.0 mM, pH 7.5, T 25 °C.

### Real soil washing solution (SW) photocatalytic treatments

The same experiments done with SS were carried out on SW containing naturally EDDS, Cu, Fe and Zn. The collected results are reported in Fig. 3a-d.

The most efficient processes for the EDDS removal were  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  and  $\text{TiO}_2/\text{h}\nu/\text{O}_2$  (Fig. 3a). It is noteworthy to observe that, for  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  system, the EDDS removal profile is strictly dependent on the concentration of Cu(II) species in the solution. For very high degrees of cupric ions removal, the electron-hole recombination is promoted, thus disadvantaging the oxidation of EDDS with the photogenerated positive holes. Lower removal degrees were achieved with  $\text{h}\nu/\text{O}_2$  and  $\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$  techniques, probably due to the presence of less photoactive or no-photoactive of

more stable Fe(III)-L complexes which could be generated, during the  $\text{HO}^\bullet$  mediated oxidation of both EDDS and DNOM, between the ferric ions and the organic natural species present in the soil washing waters. For example, it is reported that some carboxylic acids, such as oxalic, malonic and formic acids, normally formed during the photoassisted Fenton and Fenton-like processes of organic substrates, are capable of forming stable Fe(III) complexes, which, under particular experimental conditions, can exhibit poor efficiency of the organic matter degradation whereas the Fe(III)-acetate complexes are even no-photoactive (Abrahamson *et al.*, 1994; Xiao *et al.*, 2014).



**Figure 3.** Removal of EDDS (3a) Cu (3b), Fe (3c) and Zn (3d) from real soil washing solutions by  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  ( $\bullet$ ),  $\text{TiO}_2/\text{h}\nu/\text{O}_2$  ( $\blacktriangle$ ),  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{O}_2$  ( $\blacklozenge$ ) and  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$  ( $\blacksquare$ ) techniques.  $[\text{EDDS}]_0$   $3.6 \cdot 10^{-1}$  mM,  $[\text{Cu(II)}]_0 \sim 8.0 \cdot 10^{-2}$  mM,  $[\text{Fe(III)}]_0 \sim 7.0 \cdot 10^{-2}$  mM,  $[\text{Zn(II)}]_0 \sim 8.0 \cdot 10^{-2}$  mM,  $\text{TiO}_2$  initial load: 100 mg/l,  $[\text{H}_2\text{O}_2]_0$  5.0 mM, pH 7.8, T 25 °C.

Also in SW the  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  system was the only effective for the Cu(II) removal (Fig. 3b). Regarding to Fe species, the removal degrees increases on the following order:  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  <

$\text{TiO}_2/\text{h}\nu/\text{O}_2$ ,  $< \text{Fe(III)-EDDS}/\text{h}\nu/\text{O}_2 < \text{Fe(III)-EDDS}/\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$  (Fig. 3c). As said before, ferric ions are removed by precipitation as hydroxide. For the Zn species, low removal degrees were achieved in any case (Fig. 3d), the best results were obtained using  $\text{Fe(III)-EDDS}/\text{TiO}_2/\text{h}\nu/\text{O}_2$  (19,5% in 180 min) and  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$  (15,5 % in 180 min) which are able of generating organic radical intermediates that can reduce the metal ion.

#### Synthetic solution (SS) vs real soil washing (SW) mixtures

A comparison between the sets of data reported in the Figs. 2a-c and 3a-c indicates that the conversion degrees achieved for the SS were higher than on the SW, with one exception represented by the  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  process. The  $\text{TiO}_2/\text{h}\nu/\text{O}_2$ ,  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{O}_2$  and  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$  processes are mainly based on the production of hydroxyl radicals, whose activity was reduced by the presence of DNOM acting as a radical scavenger (Staehelin and Hoigne, 1985) on the SW. A different explanation can be given for the sacrificial photocatalysis ( $\text{TiO}_2/\text{h}\nu/\text{N}_2$ ) where EDDS is mainly removed for reaction with positive holes, while there is no  $\text{O}_2$  in the system. In this case, DNOM could enhance the removal degree of EDDS acting as “photosensitizer” for the generation of reactive radical organic species, capable to attack the metal-EDDS complexes (Kamiya and Kameyama, 2001).

## 4. CONCLUSIONS

The results demonstrated that for the EDDS and metals removal, from synthetic solutions and real soil washing mixtures, it is necessary different photocatalytic treatments. The EDDS removal degrees, by using  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  or  $\text{TiO}_2/\text{h}\nu/\text{O}_2$  processes on the real solutions, are close to 70 – 80 % after 3 hours of treatment, while shorter reaction times (10 minutes) are requested to reach the same percentage conversion degrees for synthetic contaminated waters through  $\text{TiO}_2/\text{h}\nu/\text{O}_2$ ,  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{O}_2$  and  $\text{Fe(III)-EDDS}/\text{h}\nu/\text{H}_2\text{O}_2/\text{O}_2$  processes.

Among the investigated metal species, only cupric ions can be removed by photoreduction to zero-valent copper through  $\text{TiO}_2/\text{h}\nu/\text{N}_2$  process for both synthetic and real aqueous solutions.

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