

# Degradation of sodium dodecyl sulphate in water using solar driven Fenton-like advanced oxidation processes

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## Abstract

Synthetic wastewater samples containing a model surfactant were treated using two different Fenton-like advanced oxidation processes promoted by solar radiation; the photo-Fenton reaction and Co/PMS/UV processes. Comparison between the different experimental conditions was performed by means of the overall surfactant degradation achieved and by obtaining the initial rate in the first 15 min of reaction ( $IR_{15}$ ). It was found that, for dark Fenton reaction, the maximum surfactant degradation achieved was 14% under low iron and oxidant concentration. Increasing Fenton reagents by one magnitude order, surfactant degradation achieved 63% in 60 min. The use of solar radiation improved the reaction rate by 17% under same conditions and an additional increase of 12.5% was obtained by adjusting initial pH to 2.  $IR_{15}$  values for dark and irradiated Fenton reactions were 0.143 and 0.154 mmol/min, respectively, for similar reaction conditions and this value increased to 0.189 mmol/min when initial pH was adjusted. The use of the Co/PMS system allow us to determine an increase in the degradation rate, for low reaction conditions (1 mM of transition metal; 4 mM oxidant) similar to those used in dark Fenton reaction. Surfactant degradation increased from 3%, for Fenton reaction, to 44.5% in the case of Co/PMS. When solar irradiation was included in the experiments, under same reaction conditions described earlier, surfactant degradation up to 64% was achieved. By increasing Co/PMS reagent concentration by almost 9 times under irradiated conditions, almost complete (>99%) surfactant degradation was reached in 5 min. Comparing  $IR_{15}$  values for Co/PMS and Co/PMS/UV, it allow us to observe that the use of solar radiation increased the degradation rate in one magnitude order when compared with dark experiments and further increase of reagent concentration increased reaction rate twice.

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**Keywords:** Co/PMS; AOPs; Fenton reagent; Degradation; Wastewater treatment; Soil restoration; SDS

## 1. Introduction

Surface active agents or surfactants are molecules with a hydrophilic head and a hydrophobic tail [1] that increase the aqueous solubility of hydrophobic compounds by solubilization. Surfactants are widely used in soap and detergent formulations, as active ingredient, like shampoos and dishwashing liquids [1,2]. They play an important role in many practical applications and products in different types of industries such as paper, food, polymers, cosmetics, food, pharmaceuticals and oil recovery [3,4].

Most of the commercial surfactants currently used are synthetic organic compounds considered as xenobiotic compounds.

Surfactants are essentially non-toxic and harmless; however, recent studies have revealed that some of the synthetic surfactants and their degradation products indeed possess potential health and environmental effects. Environmental effects associated with the presence of synthetic surfactants in the aquatic environment are considered a serious problem because these compounds are often toxic to aquatic vertebrates and invertebrates, have a potential to disrupt hormonal systems of aquatic organisms and may subsequently alter the ecosystem [5].

Sodium dodecyl sulphate (SDS) is a common representative of synthetic surfactants with a simple hydrocarbon chain widely used in commercial detergent formulations and nowadays for environmental remediation [3,4,6,7]. Systems involving surfactant–oil–water are being studying due to their high oil recovery potential. Soil washing studies showed greater oil removal with SDS than other surfactants employed [2,6,8].

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Domestic and industrial wastewaters are one of the major pollution sources of a receiving water body. Both included a significant amount of surfactants produced by the households and diverse industries. Wastewater treatment plants can have operational difficulties with the excess of foam generated by these substances.

With the growing interest of surfactants' application in environmental remediation, several authors [9–11] have studied the removal of single and double components of petroleum hydrocarbons through soil washing using aqueous surfactants solutions and conclude that this approach is a promising remediation technique [12,13]. Many papers related with surfactant enhanced soil washing deals with aspects such as the selection of ideal surfactant and doses, soil washing mechanisms and modelling of the process [14,15], the use of different driving forces for surfactant enhanced soil washing (i.e. air sparging and flotation) [16,17] and other engineering aspects [18].

Our research group has been studying for years this remediation technique at laboratory, pilot plant and full-scale levels, for decontamination of soil containing diesel, gasoline, crude oil and other petroleum fractions [12,13,19,20]. We have found that sodium dodecyl sulphate posses greater petroleum hydrocarbon removal than other surfactants employed [6–8,23]. Its use for restoration of soil contaminated with TPHs and PAHs has been proved to be as efficient as the use of biopiles or *in situ* flushing pilot plant (41 m<sup>3</sup>) with similar operational costs (140, 94 and 104 US\$/m<sup>3</sup>, respectively) [21,22].

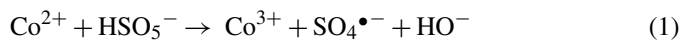
Biodegradation is the most frequent technology used for the removal of surfactants and other pollutants from wastewater as reported by Scott and Jones [24] and Torres et al. [25]. Nevertheless, the problem of the removal of pollutants, specifically in soil washing water, becomes more complicate since it concerns the biodegradation of both, the surfactant and extracted contaminants obtained from the soil. In this case, the presence of pollutants with high toxicity and persistence may inhibit the applicability of biological treatment [26]. To date, only few studies dealing with the treatment of surfactants and chemicals present in effluents from soil washing processes have been reported and even fewer studies have dealt with the complete detoxification of the complex mixtures generated during these procedures.

Advanced oxidation processes are being used as alternative processes for water treatment applications with pesticides, dyes and surfactants [27–30]. They are based on the generation of strong oxidizing species capable to react non-selectively with any organic compounds. This oxidizing species can be generated by both, non-photochemical and photochemical processes.

The Fenton reaction is a common oxidation process that includes the formation of hydroxyl radicals (2.8 V of oxidation potential) by the combination of ferrous salt and hydrogen peroxide. The addition of radiation, like UV or visible light, accelerates the generation of these radicals and it is known as photo-Fenton process. Under radiation, Fe<sup>3+</sup> ions are constantly reduced to Fe<sup>2+</sup> and the photo-Fenton process is improved by the participation of photogenerated Fe<sup>2+</sup>.

The use of other transition metals with the combination of oxidants had being studied. The cobalt/peroxymonosulphate

(Co/PMS) system has been tested for the degradation of several organic pollutants [28,29,31,32]. It generates sulphate radicals highly reactive as shown in Eq. (1)



The combination of UV light (using lamps or solar radiation) to promote the formation of radical species in this system has been recently explored [29,33,34].

Just a few studies dealing with Fenton and photo-Fenton process for the pre-treatment of wastewater with surfactant had been reported [35,36] and, to our knowledge, no reports dealing with the removal of surfactants using solar driven cobalt/PMS/UV process have been published. The aim of this study is to determine the degradation of a model surfactant, SDS, with Fenton and solar driven Fenton process and compare it with Co/PMS and solar driven Co/PMS/UV process as alternative technologies for the treatment of surfactant containing soil washing wastewater.

## 2. Experimental

### 2.1. Reagents

Sodium dodecyl sulphate (SDS) was obtained from Farco Chemical Supplies, ferrous sulphate heptahydrate (FeSO<sub>4</sub>·7H<sub>2</sub>O) was purchased from Baker and cobalt acetate (CoAcO<sub>2</sub>) from Aldrich. The oxidants employed were hydrogen peroxide (Aldrich, 50% stabilized) and sodium peroxymonosulphate (PMS), active component of OXONE<sup>®</sup> manufactured by DuPont, was obtained from Aldrich. All reagents were ACS reagent grade and were used as received.

### 2.2. Analytical methods

The amount of anionic surfactant in each sample was determined using a spectrophotometer UV–Vis Hewlett Packard 8452 A by using the methylene blue active substances (MBAS) analysis, according to the standard methods for the examination of water and wastewater [37].

### 2.3. SDS degradation

#### 2.3.1. Non-irradiated experiments

Three different transition metal concentrations (Cobalt or Fe<sup>2+</sup>: 0, 1 and 10 mM) and two oxidant agent initial concentration (H<sub>2</sub>O<sub>2</sub>, 4, 60 mM and PMS, 4 and 32 mM) were tested for the experimental runs using an initial concentration of sodium dodecyl sulphate of 1000 mg/L in all cases. SDS concentration (1000 mg/L) was chosen based on the surfactant concentration in real soil washing wastewater. SDS concentrations in the beginning of the process are about 5000 mg/L, but due to the water-rinsing steps, SDS concentration at the end of the process should be about 1000–2000 mg/L.

The experiments were carried out in a 2 L Erlenmeyer flask covered with aluminum foil. Enough transition metal (Fe<sup>2+</sup> or Cobalt) to reach its initial concentration was added in 2 L of water spiked with 1000 mg/L of surfactant. A sample of 100 mL,

considered the initial time ( $t=0$ ), was collected and preserved with NaOH 12N for Fenton and methanol (pesticide grade) for Co/PMS process. Then the appropriate volume of the oxidant ( $H_2O_2$  or PMS) to reach the desired concentration was added to the mixture. The solution remained under constant agitation during the experiment. Sampling and preservation was performed after 5, 10, 15, 30 and 60 min of reaction and obtained samples were immediately analyzed with the MBAS method. The experiments were carried out without pH adjusting.

### 2.3.2. Irradiated experiments

For irradiated experiments, a bench-scale study was carried out using a tubular pyrex glass reactor located in the focus of a compound parabolic concentrator (CPC) detailed elsewhere [27,34,38]. The system was bended  $19^\circ$  (local latitude) and had a total collection surface of  $0.1\text{ m}^2$ . Experimental conditions for solar driven process were the same as the non-irradiated experiments. In the same way, 2 L of synthetic solution was prepared by adding the necessary volume of SDS and transition metal stock solution to achieve the desired concentration. The reaction solution was put in circulation in the reactor for 5 min to achieve homogeneous conditions. A sample of 100 mL, considered zero time, was taken and preserved appropriately for each process. Afterwards, the oxidant was added to reach the initial concentration and samples were taken after 5, 10, 15, 30 and 60 min of reaction. Global radiation from 280 to 2800 nm was measured during the experiments using a Li-Cor pyranometer (LI-200SA) and was placed at the same sloping than solar collector to avoid angle adjustments. Since the photo-Fenton reaction allows the use of wavelengths from 300 to 650 nm for solar driven processes, the actual incoming irradiation was estimated using as reference an AM1.5 standard, from which a 0.35 factor was obtained for the radiation included in this wavelength range as proposed by Chacon et al. [27]. In the case of the Co/PMS system, able to use the UV part of the solar spectrum [34], global solar UV radiation was obtained using a total UV radiometer (Eppley, TUVR) which is able to measure in a wavelength range between 295 and 385 nm. Accumulated energy, defined as the total amount of irradiative energy reaching the reactor since the beginning of the experiment up to a given time by unit volume, was determined using the relation previously reported by Goslich et al. [39]:

$$Q_n = Q_{n-1} + \Delta t G_n (A/V), \quad \Delta t = t_n - t_{n-1} \quad (2)$$

where  $\Delta t$  is the time between radiation measurements,  $Q_n$  the accumulated energy (kJ/L),  $G_n$  the adjusted global radiation ( $W/m^2$ ) measured in the radiometer in each experiment,  $A$  the module area ( $m^2$ ) and  $V$  is the total system volume (L).

Additional to the experiments using solar radiation, test using UV lamp were also carried out in order to determine, under controlled UV irradiation conditions, the comparability of the tested AOPs. In order to do so, a 36 W UV lamp (WHI-36W-PLL, Phillips Lighting Co.) with a spectral output at 365 nm was used. The reaction mixtures were transferred into 11 borosilicate glass screw-cap 150-mL bottles and exposed to UV radiation until reaching the desired irradiation time. After that, each sam-

ple was immediately analyzed for surfactant concentration using the analytical method described earlier. Experimental conditions tested in these experiments were performed at the best conditions (transition metal and oxidant agent concentration) determined during solar experiments.

## 3. Results and discussion

### 3.1. Dark Fenton process

Results obtained for the experiments using Fenton process without radiation are shown in Fig. 1. As seen, no surfactant degradation was determined when SDS was put in contact with  $H_2O_2$  in the dark during 60 min of reaction time. The effect of the use of Fenton reagents in the degradation of the surfactant is evident. When soft conditions are employed (1 mM  $Fe^{2+}$ ; 4 mM  $H_2O_2$ ), only a 14% of degradation is achieved in 60 min. By increasing the iron and the oxidant concentration around one magnitude order, the removal efficiency of SDS improved 63% approximately in the same reaction time. Other works [35] had reported surfactant removal over 95% for wastewater containing an anionic surfactant with an initial concentration of 10 mg/L using 0.33 mM  $FeSO_4$  and 1.76 mM  $H_2O_2$  and an initial pH around 3. Marked differences between the results reported by Lin et al. [35] and those showed in this work are probably due to the initial SDS concentration. In this work, SDS synthetic solution was prepared with an initial concentration of 1000 mg/L, because this is similar to the quantity found in soil washing wastewater. Furthermore, experiments reported here were carried out without pH adjusting (initial pH value ca. 5), whereas Lin et al. [35] worked at pH of 3. The improvement of Fenton reagent performance at low pH values is well known [33,35].

### 3.2. Solar-driven Fenton process

Fig. 2 shows the results of application of solar radiation to Fenton reaction. The figure depicts the behavior of SDS concentration using the conditions that obtained higher overall removal of SDS for dark Fenton experiment. In order to demonstrate the

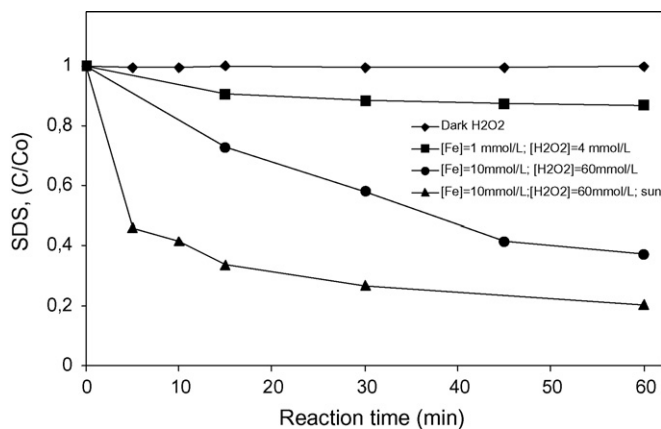


Fig. 1. Degradation of SDS using different conditions of the Fenton reagents under non-irradiated conditions. For comparison, the result with solar radiation is also included.

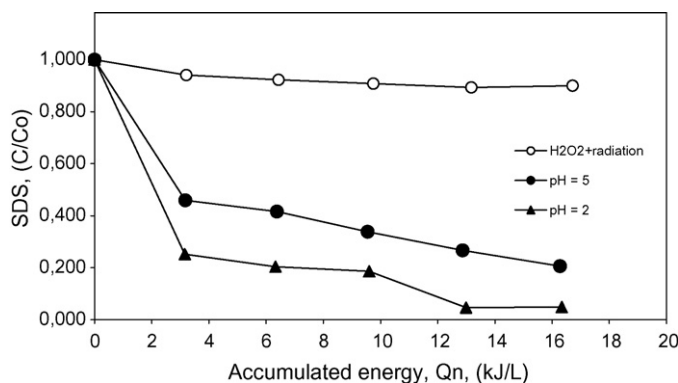


Fig. 2. SDS degradation using Fenton reaction (10 mM FeSO<sub>4</sub>; 60 mM H<sub>2</sub>O<sub>2</sub>) under solar radiation at two pH values as a function of accumulated energy  $Q_n$  (kJ/L). SDS degradation achieved using H<sub>2</sub>O<sub>2</sub> under solar radiation is also shown.

effect of pH on the reaction kinetic, data is shown for the same photo-Fenton reagent concentrations at two different pH values as a function of accumulated energy.

Comparing Figs. 1 and 2, the effect of solar radiation on the reaction kinetic can be observed with the increment of SDS final degradation. At the same reaction conditions tested (10 mM FeSO<sub>4</sub>; 60 mM H<sub>2</sub>O<sub>2</sub>), 63% of SDS degradation was achieved in the absence of solar radiation whereas a 79% of SDS removal was obtained in the CPC reactor; an increment of 17%. As mentioned earlier, the effect of pH in Fenton reagent is significant in the reaction rate. With the same concentrations employed, an improvement of 12.5% in the final degradation was achieved when pH was set to a value below 2 at the same reaction time.

Attempts to fit the experimental values to first order kinetic failed for dark experiments so initial rate at 15 min (IR<sub>15</sub>, mmol/min) was determined as kinetic constant for each experiment (see Table 1). As expected, the value of the kinetic constant when pH was less than 2 is greater than with a pH almost neutral.

### 3.3. Non-irradiated Co/PMS process

Fig. 3 shows the result of the experiment made in non-irradiated conditions with the Co/PMS process. Final degradation of the ionic surfactant under these soft conditions (1 mM Cobalt; 4 mM PMS) was 44.5%. As seen before, using the same concentrations for Fenton (1 mM Fe<sup>2+</sup>; 4 mM H<sub>2</sub>O<sub>2</sub>), only 3% of SDS removal was achieved at the same reaction time. This means an increment on the degradation of 41% employing Co/PMS system. No further experiments were realized with a different metal or oxidant concentration because the intention of the study was to make this comparison of both advanced oxi-

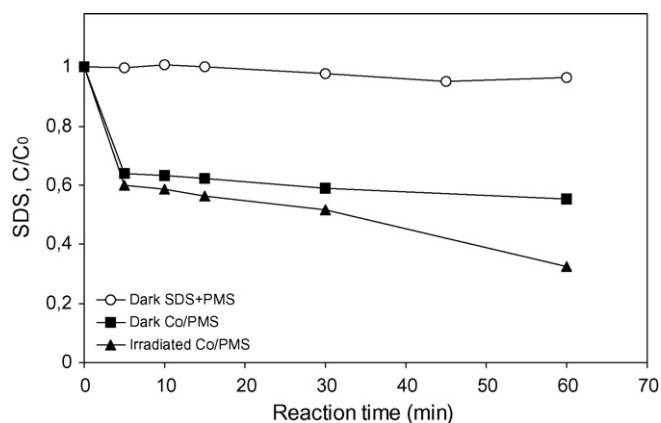


Fig. 3. SDS degradation with dark Co/PMS system. The initial concentrations were 1 mM cobalt and 4 mM PMS.

dation processes and the use of solar radiation for promoting them.

Just like in dark Fenton process, an adjustment for a first order reaction to the data obtained failed because they did not present a linear behavior under this condition or were not enough to complete the reaction kinetics. For comparison, IR<sub>15</sub> values for the dark Co/PMS process are depicted in Table 2.

As shown, the effect of the oxidant agent on the SDS degradation under dark conditions generate the lowest IR<sub>15</sub> value (0.093 mmol/min), as expected. The addition of low concentrations of the transition metal (1 mM) to the reaction mixture, produced an improvement in the dark process initial reaction rate near to seven times. By increase of the reactive concentrations by almost one magnitude order, the IR<sub>15</sub> value was also improved up to one magnitude order when compared with dark PMS test.

### 3.4. Solar-driven Co/PMS process

In the presence of solar radiation, using 1 mM Cobalt and 4 mM PMS, 64% of overall removal of the surfactant was obtained after 60 min of irradiation (see Fig. 4). In contrast, under a non-irradiated environment, 44.5% of degradation was achieved during the same conditions and reaction time with Co/PMS system; an improvement of almost 20% in the final degradation of SDS. The effect of solar radiation is evident in promoting the Co/PMS system. Furthermore, an increment of 10 times to the concentration of the transition metal and almost 9 times the oxidant gave a result of >99% of degradation in the first 5 min of reaction.

Even though photo-Fenton reaction generated a removal of 79% of the surfactant in 60 min of reaction, solar Co/PMS/UV system requires lower concentrations of reagents (1 mM Cobalt;

Table 1  
Initial reaction rates for different 10 mM FeSO<sub>4</sub> and 60 mM H<sub>2</sub>O<sub>2</sub> with solar radiation at different pH values

Reaction conditions (dark experiments)	IR <sub>15</sub> (mmol/min)	Reaction conditions (irradiated experiments)	IR <sub>15</sub> (mmol/min)
[H <sub>2</sub> O <sub>2</sub> ] = 60 mM	0.000	[H <sub>2</sub> O <sub>2</sub> ] = 60 mM	0.00
[Fe <sup>2+</sup> ] = 1 mM; [H <sub>2</sub> O <sub>2</sub> ] = 4 mM; pH 6	0.033	[Fe <sup>2+</sup> ] = 10 mM; [H <sub>2</sub> O <sub>2</sub> ] = 60 mM; pH 6	0.154
[Fe <sup>2+</sup> ] = 10 mM; [H <sub>2</sub> O <sub>2</sub> ] = 60 mM; pH 6	0.143	[Fe <sup>2+</sup> ] = 10 mM; [H <sub>2</sub> O <sub>2</sub> ] = 60 mM; pH <2	0.189



Table 2  
Initial reaction rates for different dark and irradiated Co/PMS conditions tested

Reaction conditions (dark experiments)	IR <sub>15</sub> (mmol/min)	Reaction conditions (irradiated experiments)	IR <sub>15</sub> (mmol/min)
[PMS] = 4 mM	0.0093	[PMS] = 4 mM	0.018
[Cobalt] = 1 mM; [PMS] = 4 mM	0.0646	[Cobalt] = 1 mM; [PMS] = 4 mM	0.1014
[Cobalt] = 10 mM; [PMS] = 32 mM	0.1014	[Cobalt] = 10 mM; [PMS] = 32 mM	0.230

4 mM PMS) to achieve a 64% degradation of SDS in the same reaction time. In comparison, photo-Fenton system needs 10 times more of metal and almost 17 times of oxidant.

Comparison of the kinetic data (IR<sub>15</sub>, see Table 2) shows also the same improvement in the process by the use of irradiation. PMS alone in the presence of solar radiation produced an increase in the reaction rate over one magnitude order when compared with dark PMS. The application of irradiation over the Co/PMS reagent at soft experimental conditions ([Cobalt] = 1 mM; [PMS] = 4 mM) generated a IR<sub>15</sub> value equal to the highest value obtained under dark conditions (0.1014 mmol/min). Increasing the reagent's concentration around 10 times, the highest IR<sub>15</sub> value (0.23 mmol/min) is obtained.

### 3.5. Fenton and Co/PMS promoted by artificial UV radiation

Results of the application of artificial UV radiation, generated using the UV lamp, are depicted in Fig. 5. On the figure, a comparison between the two AOPs tested is shown in order to demonstrate the differences suggested in previous sections of this work. No SDS degradation was observed when UV radiation was used alone (data not shown). When comparing each one of the processes using artificial or natural radiation, it is not easy to figure out which one produced the best results. It could be reasonable to think that solar radiation could produce best results by considering the actual radiation intensity delivered by the two energy sources. Whereas UV solar radiation impinges the reaction mixture at up to 50 W/m<sup>2</sup>, depending on the weather conditions, the UV lamp used for these experiments is able to release only around 5 W/m<sup>2</sup>. Nevertheless, this trend

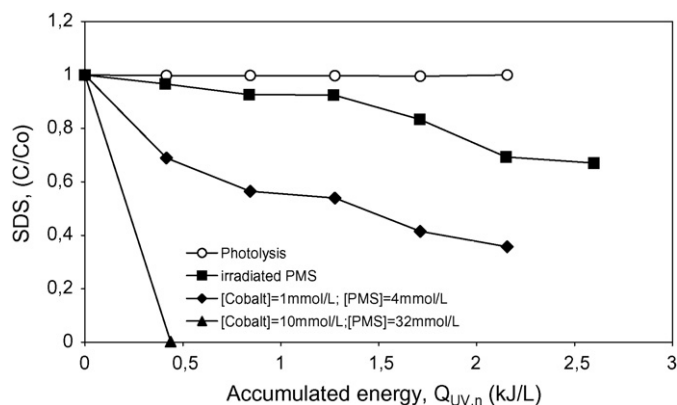


Fig. 4. Solar-driven removal of SDS in synthetic wastewater at two different Co/PMS concentrations as function of accumulated energy ( $Q_n$ ). Experimental results for SDS degradation with photolysis and irradiated PMS are also shown.

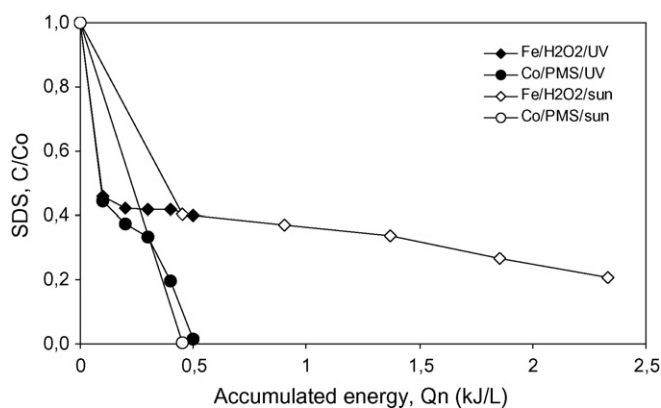


Fig. 5. Effect of the use of solar and artificial UV radiation on the tested processes as a function of accumulated energy ( $Q_n$ ).

is not observed but generation of similar degradation kinetics in both cases. From Fig. 5 it is noticeable that Co/PMS/UV system carried out the highest SDS degradation, compared to Fe/H<sub>2</sub>O<sub>2</sub>/UV, independently of the radiation source (solar or artificial UV).

A very interesting result obtained from the experiments using UV lamp is the confirmation of the availability for the Co/PMS process to use UV radiation. It is worthy to note that this methodology was able to almost completely degrade SDS (final degradation over 99%) in the presence of UV radiation as the only energy source. Under these specific experimental conditions, Co/PMS presented a better performance when compared with photo-Fenton reagent (>99% of SDS degradation using cobalt/PMS versus 60% using photo-Fenton) which provide evidence that, despite photo-Fenton reaction is very attractive because its capability to use UV and a part of the visible solar spectrum, Co/PMS is able to use UV radiation with higher efficiency.

## 4. Conclusions

Fenton and Fenton-like advanced oxidation processes were tested for the degradation of a common surfactant, SDS, employed in commercial detergent formulation and soil washing procedures. The effect of solar radiation as an alternative energy to promote both processes employed in this work was studied. Results showed that degradation efficiency of Fenton and Co/PMS increase considerably with the use of solar radiation. This is the first report of the use of the solar driven Co/PMS/UV in the degradation of surfactants in water.

From experimental conditions, Co/PMS system requires lower concentration of the transition metal and the oxidant to obtain a final degradation of almost 50%. Fenton needs higher

concentrations (10 times of metal and almost 17 times of oxidant) of concentrations reagents to reach 63% approximately of surfactant removal. Despite low cobalt catalyst concentrations were proved to effectively activate PMS and degrade SDS, cobalt is not regulated for water applications and the possibility of adverse effects in cases of high metal release to natural water is present. Current research in our group is devoted to the development of immobilized cobalt catalyst in order to improve the application of this technology.

The advanced oxidation processes promoted with solar energy are an alternative for the degradation of SDS in wastewater, a common active ingredient in detergents and soil washing. In a second step of the project, we are working with oxidation of solutions containing both SDS and petroleum hydrocarbons, which are the main components of the soil washing wastewaters. In a subsequent study, the preliminary results of our work with a real soil washing wastewater, containing SDS, TPH and other salts, will be presented.

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