



Waste sourced bio-based substances for solar-driven wastewater remediation: Photodegradation of emerging pollutants



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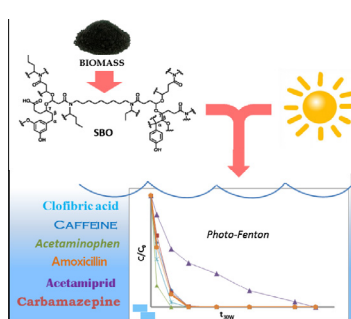
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HIGHLIGHTS

- Bio-based organic substances (SBO) have been used in photochemical processes.
- A mixture of six emerging pollutants has been used as target effluent.
- SBO-mediated reactive species generation cannot compensate for the screen effect.
- SBO have been demonstrated to enhance mild photo-Fenton (pH = 5.2).

GRAPHICAL ABSTRACT



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ABSTRACT

The effect of soluble bio-based organic substances (SBO) isolated from urban wastes on photochemical wastewater treatments was investigated using a mixture of six emerging pollutants (EPs), namely acetaminophen, caffeine, acetamiprid, clofibric acid, carbamazepine and amoxicillin. Solar simulated experiments showed that although SBO were able to enhance the indirect photolysis of the EPs, they could not compensate for the light screening effect produced by these colored materials. In contrast, SBO were able to enhance photo-Fenton at slightly acidic conditions (pH = 5.2), most probably increasing iron availability. In the later case, the order of reactivity (amoxicillin \geq carbamazepine \geq clofibric acid $>$ caffeine $>$ acetaminophen $>$ acetamiprid) was similar to that of the rate constants determined for all six EPs with $\cdot\text{OH}$. Finally, experiments performed at pilot plant scale with real sunlight at pH = 5.2 and SBO showed that 5 of the EPs were removed in only 25 min and only acetamiprid was more resistant to the process. The overall results show not only how we can use waste to clean out wastes, but also contribute to the studies which show how wastes can become a source of revenue through the industrial exploitation of their chemical value.

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1. Introduction

Solar photochemical processes are receiving increasing attention because of their ability to remove toxic or non-biodegradable compounds from the environment. They are relevant from two

different points of view: (a) photochemical processes are among the most important pathways for the removal of pollutants in natural aquatic systems [1] and (b) solar photocatalytic processes have been demonstrated as an emerging green technology for wastewater treatment [2]. In both approaches, the influence of natural dissolved organic matter (DOM), particularly humic substances (HS), seems to play a relevant role.

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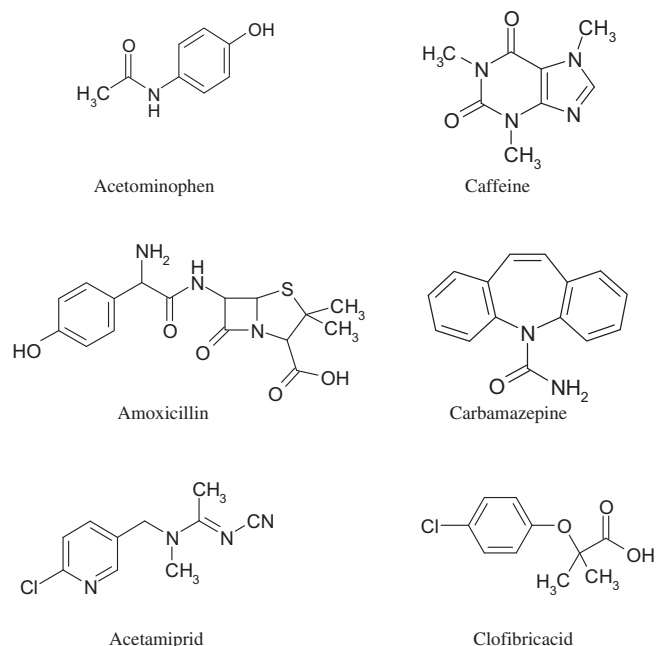
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In the case of natural aquatic ecosystems, two different mechanisms have been proposed for sunlight mediated reactions: direct substrate photolysis or indirect reactions promoted by reactive species generated by natural photosensitizers like nitrate or DOM [1,3]. In particular, HS contribute to the photochemical removal of pollutants [4], as upon irradiation they generate species such as hydroxyl radical (HO^\bullet), singlet oxygen ($\text{O}_2(^1\Delta\text{g})$), superoxide, or triplet-excited states of HS [5–9], which can potentially react with pollutants. However, HS may also negatively affect the process by screening sunlight due to their capacity to absorb in a wide range of wavelengths [10].

The applicability of HS in photochemical processes for wastewater treatment has also been investigated. They can be potentially used as photocatalysts; in fact, there is some information reporting on the use of some photochemically active organic compounds in wastewater treatment [11]. However, its major application is as complexing agent for iron in a photo-Fenton process at neutral or mild acidic conditions. This is an efficient photo-oxidative method in which a mixture of catalytic iron salts and sacrificial amounts of hydrogen peroxide are able to generate highly oxidizing species, such as hydroxyl radical [12]. One major drawback of this process is the highly acidic condition required to prevent the precipitation of iron as non-active oxide or hydroxide. Some recent papers have indicated that HS can form photochemically active iron complexes at circumneutral pH [13,14], thus allowing implementation of photo-Fenton-like processes at milder conditions. Unfortunately, in spite of these interesting properties, exploitation of HS as auxiliaries for the photoremediation of contaminated waters at commercial scale is not feasible due to their rather low concentration in natural ecosystems.

Recently, urban bio-wastes have been shown as a potential cost-effective renewable source of soluble bio-based substances [15]; these materials have similar chemical nature and properties as HS present in natural waters and soils [16,17]. SBO have been produced in relatively large scale by alkaline hydrolysis of urban bio-wastes sampled from various process streams and they have been shown rather effective in a wide number of applications, such as polluted soil washing [18], agriculture [19], animal husbandry [20] and material chemistry [21]. At laboratory scale, successful results have been obtained in the SBO-mediated photodegradation of aromatic sulfonic acids [22], phenols [23], as well as azo [24] and cationic dyes [25] dyes. Proving the performance of these substances as auxiliary for larger scale direct solar light photoremediation of a wide number of pollutants is needed to assess the actual viability of SBO assisted photoremediation processes.

With this background, the aim of this work is to assess the applicability of SBO as sensitizers, both, enhancing the degradation of pollutants by photolysis and in mild photo-Fenton process. Degradation of environmentally relevant target compounds, namely emerging pollutants (EPs), will be investigated. These are a group of chemical substances that have been recently detected in fresh waters as a result of human activities. Among EPs can be included pharmaceuticals, hormones, preservatives and bactericides, sunscreens, plasticizers and flame retardants or illicit drugs [26,27]. They are commonly present in natural ecosystems or wastewater treatment plant effluents at concentrations of a few $\mu\text{g L}^{-1}$ or ng L^{-1} and photochemical processes have been demonstrated to be useful for their elimination from the natural waters [28]. For this purpose, a mixture of six EPs belonging to different families has been chosen to test the performance of SBO in their photodegradation. These were acetaminophen (analgesic), caffeine (stimulating agent), acetemiprid (insecticide), clofibric acid (metabolite of clofibrate, also employed as herbicide), carbamazepine (psychiatric drug) and amoxicillin (antibiotic) (see Scheme 1 for structures). They have been chosen because information on their photodegradation is available [10] and the role of HS on their



Scheme 1. Chemical structures of EPs employed in this work.

photolysis and in a neutral solar photo-Fenton process is also well established [10,29]. Herein, the photolysis and mild photo-Fenton in the presence of SBOs is studied at laboratory scale. In addition, in order to better evaluate the feasibility of the process, selected experiments have also been performed in a pilot plant for wastewater detoxification under solar irradiation.

2. Materials and methods

2.1. Reagents

Acetaminophen, caffeine, amoxicillin, clofibric acid, carbamazepine and acetamiprid were purchased from Sigma–Aldrich. Hydrogen peroxide (30% v/v) and ferrous sulfate, used in the photo-Fenton reactions, were supplied by Panreac. Water employed in all the experiments was Milli-Q grade.

The SBO employed in this study, namely CVT230, was obtained from urban bio-wastes sampled from the process lines of ACEA Pinerolese waste treatment plant in Pinerolo (Italy), as described elsewhere [25]. Briefly, the starting refuse was mature compost obtained from gardening-park trimming residues. The collected refuse sample after 230 days of composting was digested 4 h at 60 °C, pH 13 and 4 V/w water/solid ratio. The resulting suspension was centrifuged for 20 min and the supernatant liquid phase filtered through the ultrafiltration membrane (5 kD molecular weight cut-off). The concentrated retentate was then dried at 60 °C to yield the final SBO product. The chemical composition of this material is given in Table 1.

2.2. Reactions

Laboratory scale experiments were performed by irradiating aqueous solution containing the six EP at an initial concentration of each pollutant of 5 mg L^{-1} . Eventually, SBO in the range 1–200 mg L^{-1} and 5 mg L^{-1} of iron ion were added (as iron(II) sulfate); this is a concentration of iron commonly used in photo-Fenton experiments at mild conditions, as at higher amounts it cannot be easily kept in solution and fast precipitation occurs [29–31]. In experiments involving H_2O_2 , the stoichiometric

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