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Chapter 3

Agro-industrial Wastewater: One of the Challenges of Sustainable Development Goal 6

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The objective of this chapter is to present a brief contextualization about water uses and the generation of wastewater, relating them to some indicators and challenges of Sustainable Development Goal (SDG) 6: clean water and sanitation. Additionally, important parameters for water analysis and the proposal of advanced oxidative treatment are presented[4].

Water Uses

Discussing environmental safety and protection is extremely important, mainly due to water scarcity in some regions and the rapid population growth. These facts have significantly reduced ecosystems, including the aquatic ecosystem, where we have found various new chemical pollutants in recent years. These factors have drastically altered the environment, and we need to understand more deeply and quickly how humans relate to nature.

According to the National Water Agency (ANA), only 2.5% of the planet's water resources are fresh water, and a large part is found in glaciers and headwaters. Brazil has the largest reserve of fresh water in the world, with approximately 12% of available fresh water (Brasil, 2021). However, it is not equally distributed across Brazilian territory, with 9.6% in the Amazon region and 2.4% in other regions, where 95% of the Brazilian population is located.

Currently, the country faces structural difficulties in the political and administrative management of water resources, making it necessary to adopt national strategies and regulations to provide not only access to quality drinking water for consumption but also for various productive economic sectors (Brasil, 2021; Lima, 2018).

In this context, the comprehensive report "Conjuncture of Water Resources in Brazil 2021" (Brasil, 2021) described that in 2020, 1,947.55 m^3/s of water were withdrawn and distributed among the following

[4] This text is based on: FRIGULIO, M. A. P. Application of pre-treatment for the reduction of organic pollutants in effluents from an agro-industry. Dissertation (Master's) - São Paulo State University (Unesp), Faculty of Sciences and Engineering, Tupã, 2023.

sectors: (a) irrigation 50%, (b) urban supply 25%, (c) industry 9%, (d) animal husbandry 8%, (e) thermoelectric plants 5%, (f) rural supply 2%, and (g) mining 2%. Over the past decades, there has been an 80% increase in water use, and it is estimated that by 2040 this percentage will increase by 42%. This history is mainly related to the country's urban, agricultural, and economic expansion. Concerning the expansion of industry, the sectors highlighted for water use in Brazil in 2020 include the sugar and ethanol production sector with 40% of industrial demand, followed by the pulp and paper, meat production, and alcoholic beverage industries. It is worth noting that the sugar-energy sector stands out for reusing its effluents in the irrigation and fertigation of sugarcane fields (Brasil, 2021).

However, until the 1990s, water used by the industrial sector was considered an irrelevant input, both economically and in terms of availability. Consequently, water resources were used without parsimony and without adequate control mechanisms, either for meeting demand or for the final disposal of effluents (Santos, 2009). Currently, however, various economic sectors, as well as civil society, are effectively concerned with adopting concrete measures to reduce water consumption and effluent generation (wastewater).

Producing sustainably, that is, mitigating environmental degradation and using limited natural resources—among them water—consciously, are challenges currently faced not only by industries but by all of society. These challenges are directly linked to the Sustainable Development Goals (SDGs) established by the United Nations General Assembly in 2015, specifically SDG 6: clean water and sanitation, ensuring the availability and sustainable management of water and sanitation for all (UN, 2016).

SDG 6 outlines eight targets and eleven indicators to be effectively achieved by 2030 (UN, 2016). In this chapter's context, Target 6.3 of the United Nations (UN, 2016) is highlighted:

> By 2030, improve water quality by reducing pollution, eliminating dumping, and minimizing the release of hazardous chemicals and materials, halving the proportion of untreated wastewater, and substantially increasing recycling and safe reuse globally (UN, 2016).

In Brazil, the target was adapted to:

> By 2030, improve the quality of water in water bodies by reducing pollution, eliminating dumping, and minimizing the release of hazardous substances, halving the proportion of untreated effluent discharge, and substantially increasing local recycling and safe reuse (Brasil, 2019).

The term "water bodies," according to the Institute for Applied Economic Research (IPEA) (Brasil, 2019), was added to indicate that the target does not only address water use but also the management of water resources. The terms "release of hazardous chemicals and materials," "untreated wastewater," and "globally" were respectively changed to "release of hazardous substances," "untreated effluent," and "locally," as these terms better fit the context of Brazilian legislation.

This target has two evaluation indicators: (a) 6.3.1 "Proportion of wastewater safely treated" and (b) 6.3.2 "Proportion of water bodies with good ambient water quality" (Brasil, 2019).

However, according to the National Water Agency, the 6.3.1 indicator still lacks systematic data on a national and regional scale for industrial effluent treatment, as this indicator includes data on industrial, domestic, and total effluents. In Brazil, the data used to calculate this indicator comes from national surveys conducted with service providers in each municipality, covering (a) urban effluents, (b) economic activity effluents (services and commerce), and (c) a small portion of industries located within urban perimeters (Brasil, 2022).

These data, along with data on septic tanks not connected to the public sewer system, are aggregated and used for calculation (Brasil, 2022). Thus, in 2019, for example, only 58.3% of effluents were safely treated in the country, an improvement of 15.5% since 2019 (Brasil, 2022), highlighting the need for enhancements in monitoring effluent treatment in the country.

Regarding the second indicator of Target 6.3 (6.3.2), it closely relates to the previous indicator (6.3.1), as it monitors water quality. Improper discharge of untreated effluents will impact receiving water bodies. According to the National Water Agency (Brasil, 2022), in Brazil, the 6.3.2 indicator data show that in 2018, 77.45% of water bodies had good environmental water quality. The monitoring from 2010 to 2018 indicated a 12.11% improvement during that period.

The fourth target of SDG 6, target 6.4, states:

> By 2030, substantially increase water-use efficiency across all sectors and ensure sustainable withdrawals and supply of freshwater to address water scarcity and substantially reduce the number of people suffering from water scarcity (UN, 2016).

In Brazil, the text of the target was simplified for clarity without changing its meaning, removing the repetitive term water scarcity, defining it as: Target 6.4: "By 2030, substantially increase water-use efficiency across all sectors, ensuring sustainable withdrawals and supply of freshwater to substantially reduce the number of people suffering from water scarcity" (Brasil, 2019).

The indicators responsible for data collection for this target are: (a) 6.4.1 "Change in water-use efficiency over time" and (b) 6.4.2 "Level of water stress: proportion of freshwater withdrawal to total renewable freshwater resources" (Brasil, 2019). Indicator 6.4.1 aims to evaluate water-use efficiency in the following sectors: services, agriculture, and industry. Being an economic indicator—a higher efficiency reflects a reduction or increase in Gross Value Added (GVA)—this evaluation reflects to what extent a country's growth depends on water resource use. According to the National Water Agency, from 2010 to 2018, there was a reduction in water-use efficiency, with recovery in recent years, ranging from 80.93 R$/m³ in 2010 to 78.02 R$/m³ in 2018 (Brasil, 2022).

The second indicator of Target 6.4 (6.4.2) estimates the consumption pressure on the country's water resources and considers the environmental need for water to conserve aquatic ecosystems. Between 2006 and 2019, the evolution of water stress levels in Brazil ranged from 1.33% to 1.72%, respectively, according to estimates by the National Water Agency (Brasil, 2022), results considered satisfactory by the United Nations (UN), which considers a percentage below 10% as satisfactory. Nevertheless, continuous and effective monitoring is essential, as changes in demand intensity or unfavorable balances can lead to scarcity and conflicts over use in certain regions.

Wastewater: important parameters

To achieve the challenges described so far, the precise and efficient management of water resources is required, before, during, and after agro-industrial production, including raw material production to the final product's realization. The liquid and solid wastes generated by processing industries must necessarily be classified and identified according to their specificities before final disposal, considering the country's current laws. For industrial effluents, the Federal Resolution of the National Environmental Council—CONAMA No. 430, of May 13, 2011—establishes national conditions and standards for effluent discharge into receiving water bodies. Additionally, each federation state has its legislation, which is usually stricter than federal legislation.

In the State of São Paulo, Decree No. 8.468 of September 8, 1976, updated by Decree No. 54.487 of June 26, 2009, provides parameters for treated effluent discharge into rivers or sewage networks, specifically Articles 18 and 19 of Decree No. 8.468/76, which address each case in detail. The decree also defines that, where a public sewage system is available, effluents from any polluting source must be discharged into it and establishes that the State Basic Sanitation Technology and Environmental Protection Company (CETESB) will supervise and define, and, when necessary, indicate the appropriate means for the enterprise to discharge its effluent (São Paulo, 1976).

In addition to legal aspects, it is important to note—concerning technical aspects—that the various components present in wastewater that alter its purity are defined and quantified through parameters that evaluate its quality. These parameters include: (a) physical characteristics, (b) chemical characteristics, and (c) biological characteristics. According to Von Sperling (2005), the main parameters to be analyzed in raw and treated wastewater are presented in Box 1.

Regarding temperature, it must be below 40°C according to effluent emission standards (São Paulo, 1976), as increased temperature reduces viscosity and surface tension, while thermal conductivity and vapor pressure increase. The reduction in surface tension of the medium can interfere with aeration rates (O_2 replenishment), causing air bubbles to stay in contact with the aquatic medium for a shorter time. Additionally, the solubility of a gas in a liquid is inversely proportional to temperature, so increased temperature reduces gas concentrations in water, including dissolved oxygen. According to CONAMA Resolution 430/2011, for the preservation of aquatic life, 5.0 mg/L of dissolved oxygen is necessary, as species tolerance varies (Metcalf; Eddy, 2016). Temperature variation also affects aquatic organisms, as they have optimal temperatures for growth, migration, spawning, and egg incubation (Metcalf; Eddy, 2016). Changes in surface temperature depend on the seasons, time of day, altitude and latitude, flow rate, and depth, but are also caused by industrial effluent discharges.

It should also be noted that pH changes influence aquatic ecosystems due to their effects on the physiology of various species. Although each aquatic organism has an ideal pH, most require pH values between 6.5 and 8.0 for growth, reproduction, and survival (Parron; Muniz; Pereira, 2011). The CONAMA Resolution 430/2011 establishes pH values between 6 and 9 for the protection of aquatic life for various classes of natural waters and values of 5 to 9 for effluent discharge. In addition to directly affecting the physiology of aquatic organisms, other aspects of lake dynamics are influenced by pH. Low pH can cause the release of toxic elements and compounds from sediments into the water, where they can be absorbed by animals or aquatic plants. Changes in pH also influence the availability of nutrients for plants, such as phosphate, ammonia, iron, and toxic metals in water (Addy; Green; Herron, 2004).

Box 1 – Main Parameters to be Evaluated in Wastewater

Characteristics	Parameters	Wastewater	
		Raw	Treated
Physical	Temperature (°C)	x	
Chemical	Hydrogen Potential (pH)	x	x**
	Alkalinity	x	
	Nitrogen	x	x
	Phosphorus	x	x
	Dissolved Oxygen (DO)		x**
	Organic Matter (COD and BOD)	x	x
Biological	Indicator Organisms	x	x
	Algae (various)		x**
	Decomposer Bacteria (various)		x**

Source: Von Sperling, 2005. Adapted by the authors. Notes: **process control, during treatment.

Alkalinity is a measure of the water's capacity to neutralize acids, that is, the number of substances in the water that act as a buffer, the capacity to resist pH changes, with the main constituents being bicarbonate ions (HCO_3^-), carbonate (CO_3^{2-}), and hydroxides (OH^-). Alkalinity comes from rocks and soils, salts, certain plant activities, and industrial wastewater discharges (detergents and soap-based products are alkaline). If the geology of an area contains large amounts of calcium carbonate ($CaCO_3$, limestone), water bodies tend to be more alkaline. The addition of lime as a soil amendment to reduce acidity in domestic lawns can run off into surface waters and increase alkalinity. Higher levels of alkalinity in surface waters mitigate acid rain and other acidic wastes, preventing pH changes. Alkalinity is also important considering wastewater and drinking water treatment because it influences treatment processes such as anaerobic digestion and coagulation (Metcalf; Eddy, 2016).

In relation to chemical characteristics, parameters such as nitrogen, phosphorus, and potassium (N, P, and K) are essential nutrients for plant growth, but when discharged in excess into the aquatic environment, they can cause eutrophication, i.e., the excess of nutrients causes excessive growth of aquatic plants (planktonic and adhered) leading to the deterioration of water body quality by accumulating decomposing organic matter. This accumulation hinders light penetration and decreases dissolved oxygen, causing the death of aquatic animals. The sources are sewage, industrial effluents, and fertilizers washed off by rainwater from agricultural areas (Fugita, 2018).

As previously mentioned, dissolved oxygen is essential for maintaining life forms and is crucial for water quality control. In addition to temperature, the discharge of effluents into a water body directly affects the oxygen balance in the system. This discharge causes a decrease in dissolved oxygen as microorganisms use it to degrade organic matter. Therefore, near the discharge point, bacteria proliferate, dissolved oxygen decreases, resulting in zones of decomposition and septic areas where there are no fish. With the natural reaeration process (oxygen from the atmosphere and photosynthesis) and the absence of new effluent discharges, the water body can recover its initial dissolved oxygen conditions kilometers after the discharge point, a process called self-purification (Manahan, 2013).

Indirectly, two analyses are performed for quantifying organic matter: Biochemical Oxygen Demand (BOD) and Chemical Oxygen Demand (COD). BOD measures the amount of oxygen used by microorganisms during the biochemical oxidation of organic matter: high BOD values indicate pollution as a large amount of oxygen is needed for the biodegradation of organic matter. According to CONAMA Resolution 430/2011, the maximum permitted BOD for effluent discharge is 120 mg/L, with this limit only being exceeded in the case of effluent from a treatment system with a minimum removal efficiency of 60% BOD (Brazil, 2011), but in the state of São Paulo, by State Decree 8468/1976, it is 60 mg/L O2 or a minimum overall treatment process efficiency of 80% (São Paulo, 1976). COD is the amount of O2 needed for the oxidation of organic matter by a chemical agent, and when COD is high, it represents that a large amount of oxygen is needed for the degradation of organic matter. There is no Maximum Permitted Value (VMP) for COD, but it is very useful when used together with BOD.

Water Pollution: Organic Compounds

Water pollution by organic chemical compounds is extensive, with these compounds being largely responsible for aquatic pollution due to their constant use and presence in domestic, industrial, and agricultural effluents. Many of these compounds are toxic, persistent, have high Chemical Oxygen Demand (COD), and are not treated by conventional methods. Among the organic compounds present in effluents are biodegradable and recalcitrant or refractory compounds.

Biodegradable compounds are chemicals that, after a certain period, are decomposed by the action of microorganisms. Examples of these compounds include proteins, carbohydrates, lipids, and soaps. There are two types of biodegradation pathways: (1) aerobic pathway: decomposition by microorganisms using O_2 + carbon source (glucose) and nitrogen source (NH_3) + essential nutrients (P, S, Fe) producing biomass + CO_2 and H_2O; (2) anaerobic pathway: decomposition by microorganisms using carbon source (glucose) and nitrogen source (NH_3) + essential nutrients (P, S, Fe) + electron acceptors (NO_3^-, SO_4^{2-}, Fe^{3+}) producing biomass + CH_4 and H_2O.

Non-biodegradable organic compounds or those with very slow biodegradation rates are called recalcitrant or refractory. These compounds are present in most agro-industrial effluents. Due to their complex chemical structure, many are stable (persistent), and in cases of prolonged exposure to very low concentrations (chronic toxicity), some can be carcinogenic, mutagenic (alterations in genes and chromosomes), or teratogenic (problems in newborns), and can also cause kidney and liver dysfunctions, sterility, and neurological problems. Moreover, they can affect non-target organisms (cattle, bees, humans), and often their partial degradation byproducts are also toxic and persistent (Manahan, 2013).

Organic compounds referred to as micropollutants or emerging contaminants are substances that have been used for a long time and new substances that are part of our daily routine, such as pesticides, dyes, pharmaceuticals, personal care products, cosmetics, cleaning products, chemical additives, and plastics/microplastics. The recent focus on these contaminants is due to access to new technologies capable of detecting compounds at very low concentrations, on the order of micrograms (µg/L) or nanograms (ng/L), thus enabling the quantification of hundreds of compounds in different environmental areas. The main analytical techniques that made these quantifications possible are chromatography, especially liquid chromatography coupled with mass spectrometry.

Many of these micropollutants are not included in environmental control regulations or legislation and are not part of routine monitoring programs by environmental and health agencies. Therefore, there is a need for studies aimed at treating refractory organic molecules in water, as traditional water and sewage treatment methods do not show satisfactory efficiency for these compounds, which are increasingly present in our waters.

Alternative for Treatment

Among the different processes available (physical, chemical, or biological), Advanced Oxidation Processes (AOPs) have been successfully studied for the degradation of toxic and persistent organic pollutants. AOPs are considered a highly competitive technology for water treatment to remove recalcitrant organic pollutants that are not treatable by conventional methods (Wang; Zhuan, 2020). The development and research of such AOP applications have been stimulated due to the pollution of water resources through agricultural and industrial activities and the requirement for industries to meet effluent discharge standards.

AOPs are based on the generation of hydroxyl radicals ($HO^{\bullet}$), a strong (E^{o} = 2.80 V) and non-selective oxidant that reacts with most organic compounds very quickly, ensuring the effectiveness of AOPs both in terms of oxidation capacity and kinetic standpoint (Oturan; Aaron, 2014). These radicals attack carbon chains, potentially degrading them completely into CO_2, water, and inorganic ions or partially, producing fewer toxic compounds that are more degradable by conventional processes. In AOPs, the generation of $HO^{\bullet}$ can be achieved by chemical, electrochemical, photochemical, and the more recent Sono chemical and Sono electrochemical methods. They are divided into homogeneous and heterogeneous processes (which use solid catalysts, electrodes), which can occur in the presence or absence of Ultraviolet (UV) light, which can be of artificial or natural (solar) origin.

The generation of $HO^{\bullet}$ can be achieved through the reaction between iron ions and hydrogen peroxide, known as the Fenton reaction or reagent (Fenton, 1894). The classic application of the Fenton reagent is a homogeneous system, requiring only the mixing of reagents at ambient temperature and pressure, not requiring sophisticated equipment. Therefore, it is considered safe for handling and has a low environmental impact (Oturan; Aaron, 2014).

Fenton's catalytic reactions basically consist of the oxidation of Fe^{2+} to Fe^{3+}, producing $HO^{\bullet}$, which will oxidize any organic compound present in the solution. The reactions of the Fenton Reagent and its interaction with the organic molecule (R), in a simplified form, are shown in Table 1.

Table 1 – Fenton Reagent Reactions

Reaction		
$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{-} + HO^{\bullet}$	(Start of the reaction chain)	(1)
$RH + HO^{\bullet} \rightarrow R^{\bullet} + H_2O$		(2)
$R^{\bullet} + Fe^{3+} \rightarrow R^{+} + Fe^{2+}$		(3)
$Fe^{2+} + HO^{\bullet} \rightarrow Fe^{3+} + OH^{-}$	(End of the reaction)	(4)

Source: Moravia; Lange; Amaral, 2011. Note: Fe^{2+} and Fe^{3+} represent hydrated molecules; R represents an organic molecule

The efficiency of the degradation of organic pollutants during the application of the Fenton process will depend on some operational factors, such as the concentration of reagents, operating pH, temperature, and the concentration of contaminants in the wastewater (Zhang *et al.*, 2019).

The molar concentration ratio between Fe^{2+}/H_2O_2 is extremely important because the efficiency of the degradation of organic compounds will depend on this ratio. If the concentrations of Fe^{2+}/H_2O_2 are excessive, there will be low concentrations of $HO^\bullet$ to oxidize the organic matter, as unwanted reactions may occur with excess reagents causing their elimination (Aarslan-Alaton; Kabdaşli; Teksoy, 2007; Kallel *et al.*, 2009).

Despite numerous studies, there is no consensus on the appropriate molar ratio for oxidation between Fe^{2+}/H_2O_2, as it may vary depending on the type of effluent/compound to be treated. Aarslan-Alaton; Kabdaşli and Teksoy (2007) report that for removing color in effluents containing dyes, the ratio 1:3 was efficient, while Lange *et al.* (2006) report that the concentration range of Fe^{2+}/H_2O_2 can vary from 1:5 to 1:25. Araújo *et al.* (2016) conclude that this ratio varies according to the type of effluent to be treated.

Regarding the optimal pH range for the application of this technique, studies report values of 2 to 4, as with the increase in pH, H_2O_2 decomposes rapidly into water and oxygen, and iron precipitation may also occur, reducing the production of HO• and, consequently, decreasing the efficiency of the oxidation process (Zhang *et al.*, 2019; Ziembowicz; Kida, 2022). Bello, Raman, and Asghar (2019) report that pH above 3.5 promotes the precipitation of Fe^{3+} in the form of iron hydroxide, which decreases its interaction with H_2O_2 and, consequently, reduces the production of HO•. Additionally, large amounts of chemicals are spent to adjust organic wastewater to pH 2-4 before decontamination, which is a disadvantage that needs to be evaluated and improved.

Studies have been conducted on the application of the Fenton process to various types of effluents, such as tannery effluents (Kalyanaraman *et al.*, 2012), olive mill effluents (Kallel *et al.*, 2009; Lucas; Peres, 2009), paper and pulp effluents (Jamil *et al.*, 2011), yeast effluents (Pala; Erden, 2005), slaughterhouse effluents (Almeida *et al.*, 2015), water with humic substances (Júlio *et al.*, 2006), coke plant effluents (Jiang *et al.*, 2011), landfill leachate (Lange *et al.*, 2006; Moravia; Lange; Amaral, 2011), and pesticide effluents (Forti *et al.*, 2020; Tadayozzi *et al.*, 2021; Da Silva *et al.*, 2022). In all these varieties, the method applied at the laboratory scale was efficient, showing a reduction in phytotoxicity and a reduction in COD. The reduction also ensured improvements in other parameters such as color and BOD in all the studies.

The combination of the Fenton process with biological treatment was applied and evaluated by Kalyanaraman *et al.* (2012). The Fenton reagent was applied as a pre-treatment for tannery effluents before the biological process and showed satisfactory results after the treatability of this combination of methods. The pre-treatment improved the biodegradability of the tannery effluent, resulting in the formation of short-chain hydrocarbons and reducing its COD and BOD load.

The application of the Fenton process to various types of industrial effluents is extensive; therefore, the study and deepening of the AOP by the Fenton process has become a subject of constant improvement and investigation, given its easy applicability and high benefit, which can make industries more competitive and improve the biodegradability of difficult-to-treat effluents.

One way is to apply the Fenton reagent with irradiation addition, called photo-Fenton, to increase the production of HO•. Under light irradiation, $[Fe(OH)]^{2+}$ is excited, regenerating Fe^{2+} that catalyzes the decomposition of $_{d}$e H2O2 and producing HO• that degrades organic pollutants, according to reaction 5. In addition, direct photolysis of H_2O_2 also produces HO• (reaction 6).

$$[Fe(OH)]^{2+} + h\nu \rightarrow Fe^{2+} + HO\cdot \qquad (5)$$

$$H_2O_2 + h\nu \rightarrow 2\ HO\cdot \qquad (6)$$

The essence of the photo-Fenton process is to accelerate the reduction of Fe^{3+} to Fe^{2+} using the energy provided by light. Since the use of artificial light makes the process expensive, utilizing sunlight can remedy this inconvenience. The combination of ultraviolet or visible light with conventional Fenton can increase the efficiency of organic pollutant degradation, reducing sludge formation.

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