FS-TER-004

TECHNOLOGY FACT SHEETS
FOR EFFLUENT TREATMENT PLANTS
OF TEXTILE INDUSTRY

OZONIZATION

SERIES: TERTIARY TREATMENTS

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INDITEX

OZONIZATION (FS-TER-004)

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1. INTRODUCTION

Ozone is a chemical known for its high oxidizing power. In recent decades, especially due to the progressive tightening of quality limit values in the discharge flows to natural environment, ozone has become an advanced alternative for textile wastewater treatment.

This review paper is mainly based on the *Ozonation text or Water and Wastewater* (Gottschalk et al, 2000), the ozonation chapter present in *Handbook of Environmental Engineering, Volume 3: Physicochemical Treatment Processes* (Nazih et al, 2005) and Design Manual: *Municipal Wastewater Disinfection* (US EPA, 1986), with contributions of articles and other specific documents on the treatment of textile effluents.

2. OZONIZATION FUNDAMENTALS

2.1. Ozone properties (US EPA, 1986)

Ozone is an unstable gas which is produced when oxygen molecules dissociate into atomic oxygen and subsequently collide with another molecule of oxygen. The energy source to dissociate the oxygen molecule can be produced commercially and can also occur naturally. Some natural sources for the production of ozone are from ultraviolet sunlight and lightning during a storm.

The stability of ozone, without being high, is higher in air than in water. In the absence of phosphates and carbonates and at pH 7, it has been observed an average life of 8 minutes in water.

In air or water, ozone stability is very temperature dependent. The half-life at room temperature varies from 20 to 100 h at 250 °C while only 0.04 to 0.4 s. This characteristic is important for ozone design because cooling of the ozone generators is necessary.

Similarly, good ventilation of the location of the ozonator will be required and excess ozone contained in the system must be destroyed.

Ozone is an explosive substance at concentrations above 240 g/m^3, although ozone generators rarely procude concentrations higher than 50 g/m^3.

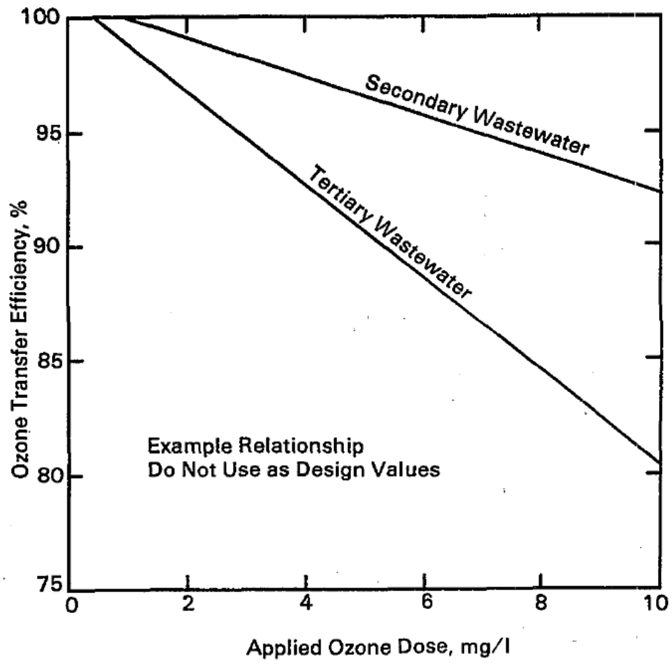


Figure 1.- Ozone transfer efficiency will decrease as applied ozone dosage increases.

Ozone is a parityally water soluble substance. Transfer efficiency (TE - transfer efficiency) has been shown as a function of the area of the two phases in contact (gas-liquid), the transfer potential (depending on the concentration of the gas in the liquid) and a transfer coefficient. At a certain dose of ozone, a low-quality wastewater presents a greater demand for ozone and TE will be higher.

Ozone is a strong oxidizing agent, having an oxidation potential of 2.07 V. It will react with organic and inorganic compounds present in natural or wastewater. These reactions are typically called "ozone demand" reactions.

2.2. Advantages and disadvantages of the use of ozone

Ozonation systems use have the following advantages (Nazih et al., 2005; Sharma, 2013)

- Possesses strong oxidizing power and requires short reaction time, which enables the pathogens to be killed within a few seconds.
- Produces no taste or odor.
- Provides oxygen to the water after disinfecting.
- Requires no chemicals.
- Oxidizes iron and manganese.
- Destroys and removes algae.
- Reacts with and removes all organic matter.
- Decays rapidly in water, avoiding any undesirable residual effects.
- Removes color, taste, and odor producing compounds.
- Aids coagulation by destabilization of certain types of turbidity.

Some of the disadvantages are listed below:

- Toxic (toxicity is proportional to concentration and exposure time).
- Cost of ozonation is high.
- Installation can be complicated.
- Ozone-destroying device is needed at the exhaust of the ozone reactor to prevent toxicity.
- May produce undesirable aldehydes and ketones by reacting with certain organics.
- No residual effect is present in the distribution system, thus postchlorination may be required in case of disinfection use.
- Much less soluble in water than chlorine; thus, special mixing devices are necessary.
- It will not oxidize some refractory organics or will oxidize too slowly to be of practical significance, which would need adicional reactives.
- It will not eliminate dissolved minerals and salts.

3. OPERATION AND DESIGN OF OZONE SYSTEMS

The five main elements of an ozonation system are (Figure 2):

1. Air preparation or oxygen feed.
2. Power supply
3. Ozone generation
4. Ozone contacting
5. Ozone contactor exhaust gas destruction

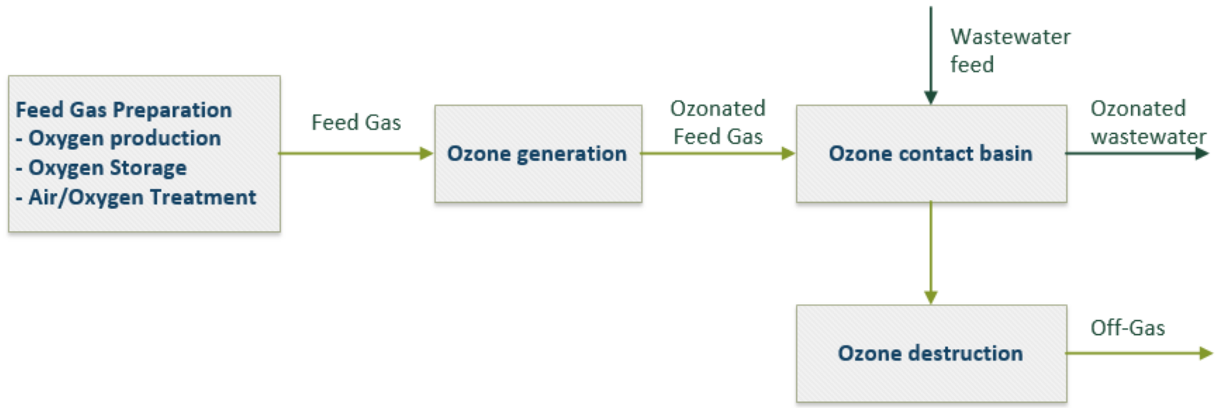


Figure 2. Simplified ozone process schematic diagram (mod. From US EPA, 1986)

3.1. Air preparation

It is recommended to dry ambient air to a maximum dewpoint of -65 ° C before being used in an ozonation system.

Post-desiccant filters are installed to remove particulates less than 0.3–0.4 µm in diameter. Two-stage filtration is recommended. The first-stage filter removes particulates greater than 1 µm and the second stage removes particulates less than 0.3–0.4 µm in diameter.

Air feed systems can dry ambient air or use pure oxygen.

Ambient air-feed systems used for ozone generation are classified by low, medium, or high operating pressure. The most common type is a system that operates at medium pressures ranging from 0.7 to 1.05 kg/cm^2. The decision to use a high-, medium-, or low-pressure air preparation system often is based on a qualitative evaluation of potential maintenance requirements, as well as an evaluation of capital and operating costs.

Schematic diagrams of low- and high-pressure feed gas pretreatment systems are shown in Figs. 3a and 3b. It must be pointed out that Fig. 3a is also representative of a medium-pressure system, but may require a pressure reducing valve (PRV) upstream from the ozone generator as shown in Fig. 3b. Each diagram illustrates a dual component process, and depicts the desired flexibility for the provided equipment.

Pure oxygen use as feed gas

Using pure oxygen has certain advantages that have to be weighed against its added expense. Most suppliers of large-scale ozone equipment consider it cost effective to use ambient air for ozone systems having less than 1,590 kg/d generating capacity. Above this production rate, pure oxygen appears to be more cost effective. Systems that dry ambient air consist of desiccant dryers, commonly used in conjunction with compression and refrigerant dryers for generating large and moderate quantities of ozone. Very small systems (up to 0.044 m^3/s) can use air-drying systems with just two desiccant dryers (no compression or refrigerant drying). These systems use silica gel, activated alumina, or molecular sieves to dry air to the necessary dew point (–65ºC).

Pure oxygen use is considered as an alternative to air feed in many applications due to a number of reasons (Nazih, 2005):

- It has a higher production density (more ozone produced per unit area of dielectric).
- It requires lower energy consumption (energy supplied per unit area of dielectric).
- Essentially double the amount of ozone can be generated per unit time from oxygen than from air (for the same power expenditure); this means that ozone generation and contacting equipment can be halved in size when using oxygen, to generate and contact the same amount of ozone.
- Smaller gas volumes are handled using oxygen, rather than air, for the same ozone output; thus, costs for ancillary equipment are lower with oxygen feed gas than with air.
- If used in a once-through system, gas recovery and pretreatment equipment are eliminated.
- Ozone transfer efficiencies are higher due to the higher concentration of ozone generated.

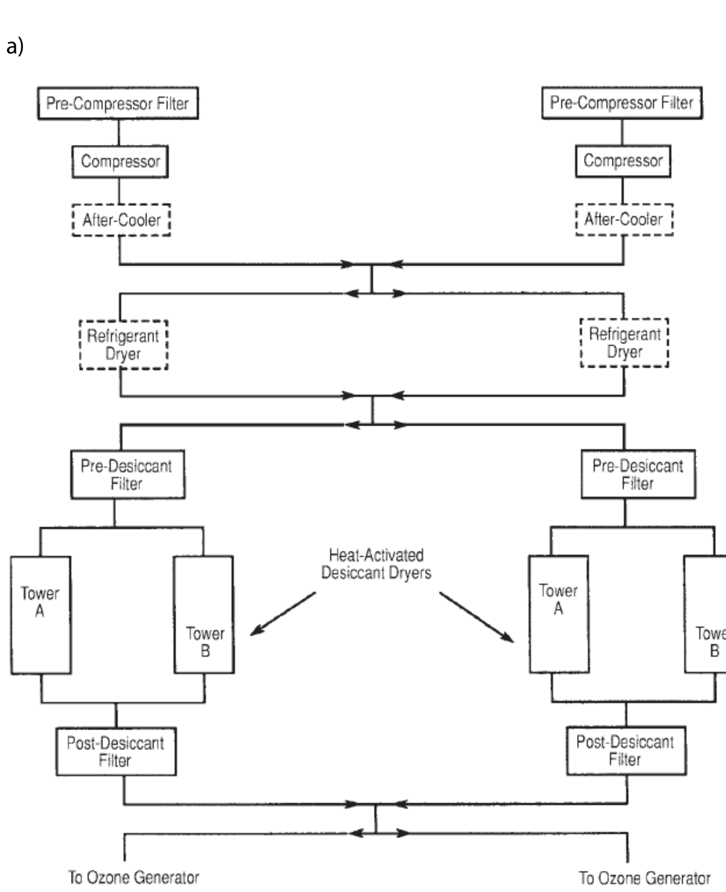


Figure 3.- Schematic diagrams for low (a) and high (b) pressure air feed gas treatment system (US EPA, 1986).

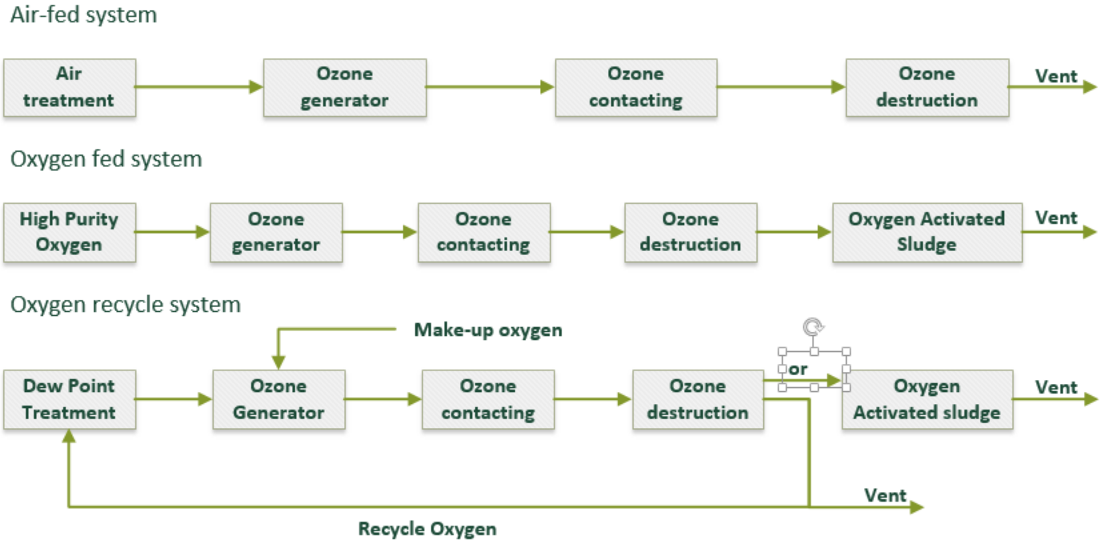


Figure 4 Diagram with different ozonizator feed options (mod. from US EPA, 1986).

A common practice of O_2 fed systems is the use of waste gas ozonator blowers in a conventional biological treatment, if such treatment has ETP (Figure 4). This practice adds the difficulty of balancing the flow of oxygen to fit the needs of both processes (US EPA, 1986)

3.2. Electric supply

The voltage and frequency supplied to the ozone generator is variable as a mean of controlling the amount and flow of ozone produced.

Ozone generators employ high voltages (> 10,000 V) or high frequency electric current (up to 2000 Hz), requiring a number of technical considerations. Electrical cords must be properly insulated; the high voltage transformers should be kept in a cool environment and transformers must be, at the same time protected from ozone pollution, which can occur with small leaks of ozone. High frequency and voltage transformers must be high quality units specific to the operation with ozone. The supplier of the ozonator should be responsible for the design and supply of electrical subsystems.

3.3. Ozone generation

There are actually some different technologies involved in ozone generation, although, the most common is electrical discharge, wich consists in oxygen molecule ionization by applying high power alternating current to the gas. As explained before, it uses air or pure oxygen as feed gas, either at ambient or elevated pressure.

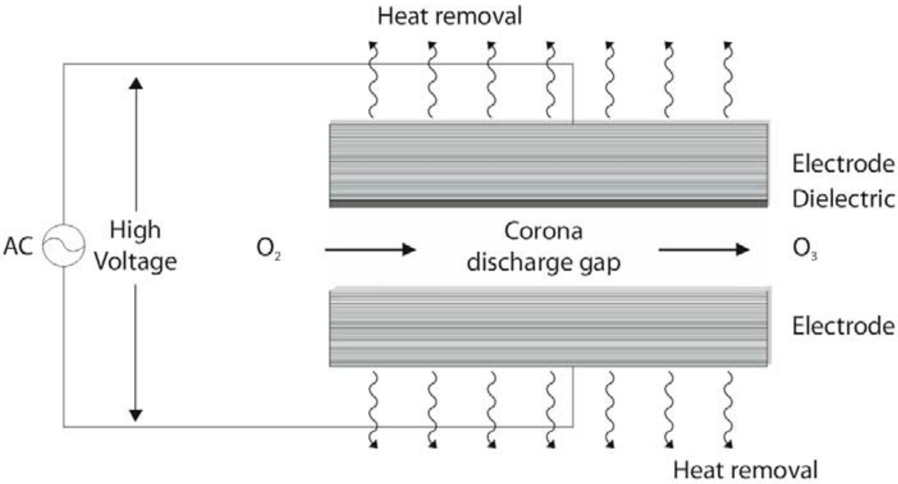


Figure 5.- Principal elements of a corona discharge ozone generator (mod. from US EPA, 1986).

Ozone can be generated by UV radiation techniques, but only to maximum concentrations of 0.25% by weight.

The discharge cell, as shown in Figure 5, consists of two dielectric material electrodes separated by a discharge gap, through which high potential differences are maintained. A dry and cold flow oxygen-enriched air, pure oxygen or air are passed through the electrodes and ozone is produced.

Types of ozone generators
They are normally classified by the control and cooling mechanism and the physical arrangement.

- Horizontal tube, Voltage controlled, water cooled.
- Vetical tube, voltage controlled, water-cooled.
- Vertical tube, Frequency-controlled, double cooled.
- Lowther plate, frequency-controlled, air-cooled or water cooled.

The starting conditions of these generators can be divided into:

- Low frequency (60 Hz) with high voltage (> 20.000V);
- Medium frequency (600 Hz), medium voltage (<20,000 V);
- High frequency (> 1000 Hz) and low voltage (<10,000 V)

Where low frequency units are the most common.

Ozone contactor

Ozone is mainly generated as a gas, so wastewater and gas have to be brought into contact. This unit involves two operation: the ozone absorption within water and the reaction between the ozone and the materials contained in the fluid, whether this two operations can occur in a single or in multiple chambers.

There are several types of ozone gas-liquid contactors, being Bubble columns (BCs) and Stirred tank reactors (STRs) the most commonly used.
Bubble columns reactors consist in a chamber which contains a heterogeneuous system with the water phase and ozone fine bubbles provided by bottom diffusers or sidestream gas-injection systems. As the gas moves upwards, ozone dissolves and reacts with the substances contained in the liquid. Ozone is normally added to the tank through fine bubble diffusers or sidestream bubble injectors (Figure 6). Bubble contactors are commonly constructed with 5.5 to 6.7 m in water depth to achieve 85 to 95 percent ozone transfer efficiency (EPA, 1999). The larger the number of contact chambers connected in series, the closer to plug-flow will be the ozone reaction.

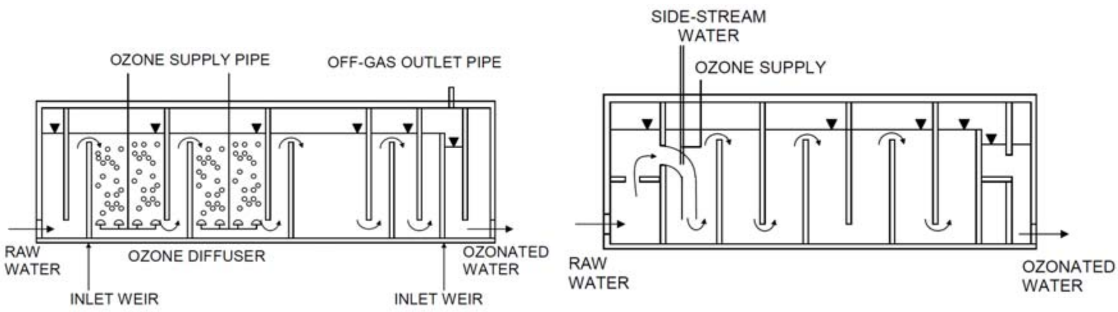


Figure 6.- Sectional view of a fine bubble diffuser (left) and a sidestream water injector ozone contactor (Kim, 2007) .

Turbine mixers are simple chambers with the liquid phase where gas is introduced through a sparger (Figure 7). Ozone solution and distribution in the chamber is aid by a mixer sometimes aid by baffles made of corrosion-resistant materials. Ozone transfer efficiency in turbine mixers can result over 90 percent (Gottschalk, 2002; EPA, 1999).

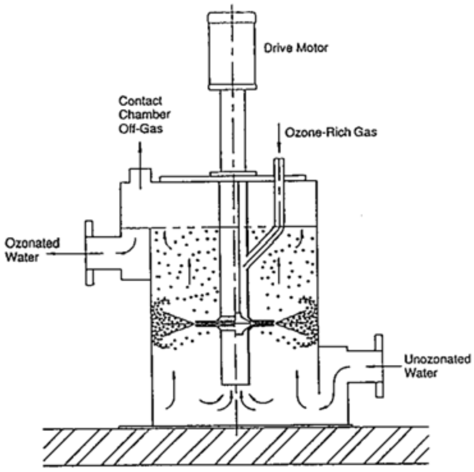


Figure 7. Turbine mixer ozone contactor (EPA, 1999)

Besides, systems with diffusers and tower modules contactors do not require more energy than that used for the generation of ozone. The high-speed stirrers, static mixers and all negative pressure contactors do require additional energy.

In order to develop a proper ozone control, it is important to measure the ozone gas level both in the inlet and the outlet of the reactor, as well as the liquid ozone concentration. Larger systems implement continuous analytical methods like amperometric and UV-absorption to control O_3 level, while smaller scale ones can also use discontinuous titrimetric and photometric methods.

3.4. Exhaust gas destruction

As ozone off-gas is still over toxic levels, it requires to be transformed again to atmospheric oxygen before leaving the system. Ozone is destroyed at temperatures higher than 350°C or by catalized reactions over 100°C. Gas outflow will contain ozone levels below 0.1 ppm in volume.

When pure oxygen is used, the economic possibility of recirculating the oxygen flow after ozone destruction should be studied.

3.5. Design indications (EPA, 1986; Nazih, 2005)

- Air compressors should be located in a separated area or in a proper housing to avoid noise disruption
- Follow manufacturer's indications in compressors and dryers sealings
- When bottom diffusers are used, contact chamber max. depth is indicated by the maximum ozone generator pressure (around 103 kPa).
- Each contact stage should present a drainage to improve maintenance operations (once or twice per year). It is recommended to install more than one
- The contactor should leave 1.2-1.8 m headspace to allow foam formation.
- Each set of diffusers must have a flow control valve in the pipe and separate flow meter.
- Wastewater flow is recommended to work countercurrent to gas flow to maximize transfer efficiency of ozone.
- The media contact should be made of concrete for waterproof and resistant to ozone construction.
- The pipe system for gas-ozone must be made of stainless steel ozone systems (304L or 316L) prepared for positive pressure. Weldings are recommended to be Tungsten inert Gas, or even flanged connections without weldings can be performed.
- Ozonized feed-gas and contact basin off-gas sample lines should be stainless steel tubing. Teflon tubing may be considered for short runs.

4. APPLICATIONS IN TEXTILE EFFLUENTS TREATMENT

Applications ozonation processes as part of water treatment in the textile industry can be found as:

- Process water preparation. Color and organic matter removal and disinfection.
- Effluent treatment of industrial wet processes: Where ozonation is used for color, toxicity and biodegradable organic matter reduction as well as to raise dissolved oxygen levels in effluent discharge. With the same purposes, it also appears as part of effluent recycling processes.

4.1. Applications in the treatment of natural waters for preparation of process water

It is recommended in situations where there are problems with DBPs (disinfection by products). It has been found effective in Cryptosporidium and microcystins inactivation.

In turn, it has been proved that ozone is an effective tool to prevent pest ingrowth within the supply network, such as the zebra mussel or to control of certain algae growth and zooplankton inactivation which can subsequently be removed by flocculation and filtration (Nazih, 2005).

Ozone can also be used to precipitate metals in groundwater containing iron and manganese and low concentration of organic substances.

It is considered that the humic substances are the main cause of the color presence in natural waters. According to different authors, **a dose of 1-3 mg O_3/ mg C leads to almost complete color removal.** Doses of ozone applied to obtain high color elimination can be very high. Interestingly, when the ozone dose is sufficient, organic structure is modified so that the final chlorine demand decreases in disinfection process.

It can be found in natural water treatment as a way to eliminate synthetic organic chemicals, obtaining, usually CO_2 and VOCs, both gases can be removed by stripping.

4.2. Wastewater treatment

It is proposed as an industrial scale unit in wastewater facilities that can use ozonation systems with ozone generation capacity above 0.5 kg per hour. These treatments can be classified as (Gottschalk, 2000):

- As one step of the overall effluent treatment (in chemical processes, or combinations: chemical/biological and physical-chemical).
- As part of an industrial pretreatment in water reuse line or indirectly or leading to discharge to a waste water treatment plant.
- As a mean to comply with effluent discharge quality in certain specific compounds due to their toxicity; or to increase que removal capacity in color, aggregated parameters (COD or BOD) or solids reduction and in disinfection uses.

- **Color removal**

The use of chemical oxidants such as ozone, chlorine or hypochlorite, hydrogen peroxide or potassium permanganate can be used to destroy the dye and generate a colorless solution, whose oxidation products can be removed by biological treatment. Ozone eliminates the color of all types of dye, with the exception of vat and dispersed non soluble dyes, which have slow reaction rate (Vandevivere, 1998).

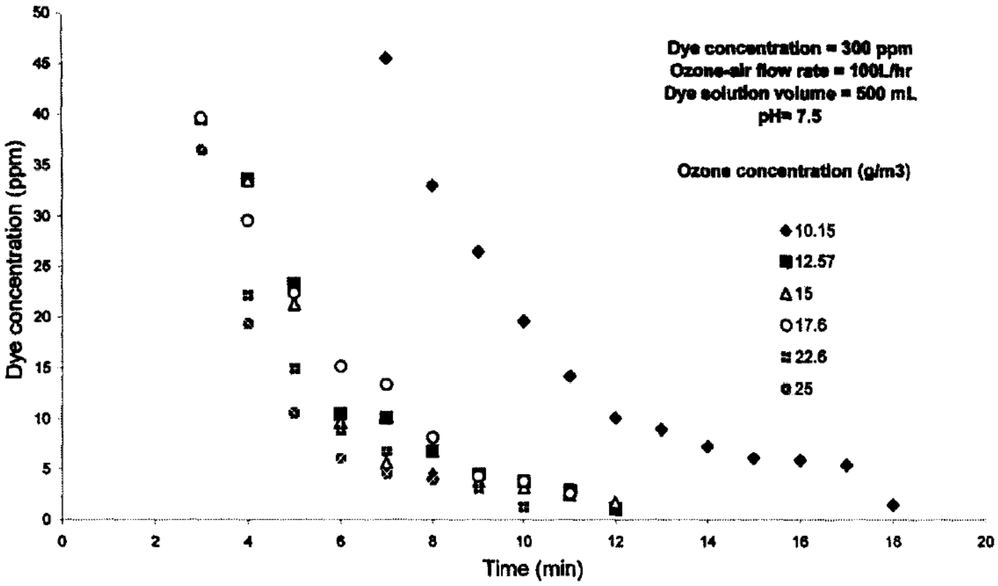


Figure 8.- Time vs concentration data at different ozone concentrations in a direct dye treatment (Konsowa, 2003).

In Figure 8, the evolution of the concentration of a direct dye versus time can be observed at different concentrations of ozone. In this example, the specific dose of ozone for bleaching is approx. 0.02 g O_3/g removed dye.

- The determining parameters in the study of color removal of textile wastewater are:
- Ozonated air flow
- Concentration of dye/s
- pH
- Concentration of ozone in the gas
- Contact time

- **Proposed treatment lines**

Ozone units with disinfection targets are proposed. In many situations, textile mills combine process effluent with human consumption wastewater needing bacterial reduction before discharge to natural media. As ozonization is a quite expensive technique, different levels of pre-treatment should be considered before disinfection unit. Although it is not applicable to wastewater with important industrial loads, in EPA (1986) appears the example of 4 different municipal WWTP effluents to be treated with ozone. The best disinfection performance was achieved with an influent nitrite-nitrogen concentration less than 0.15 mg/L and dissolver COD lower than 10 mg/L (Figure 9).

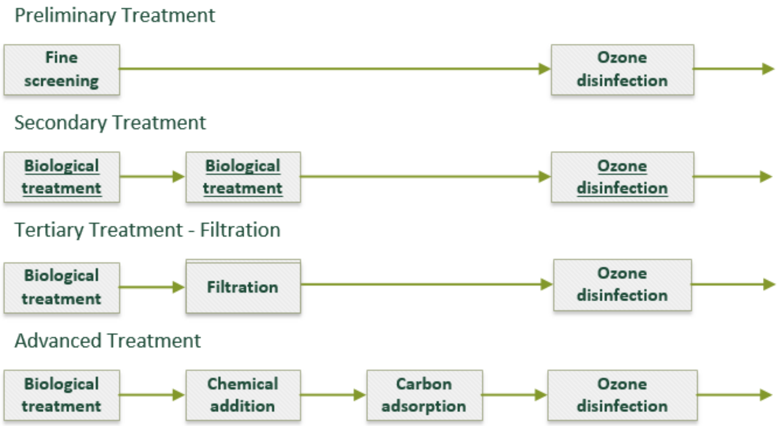
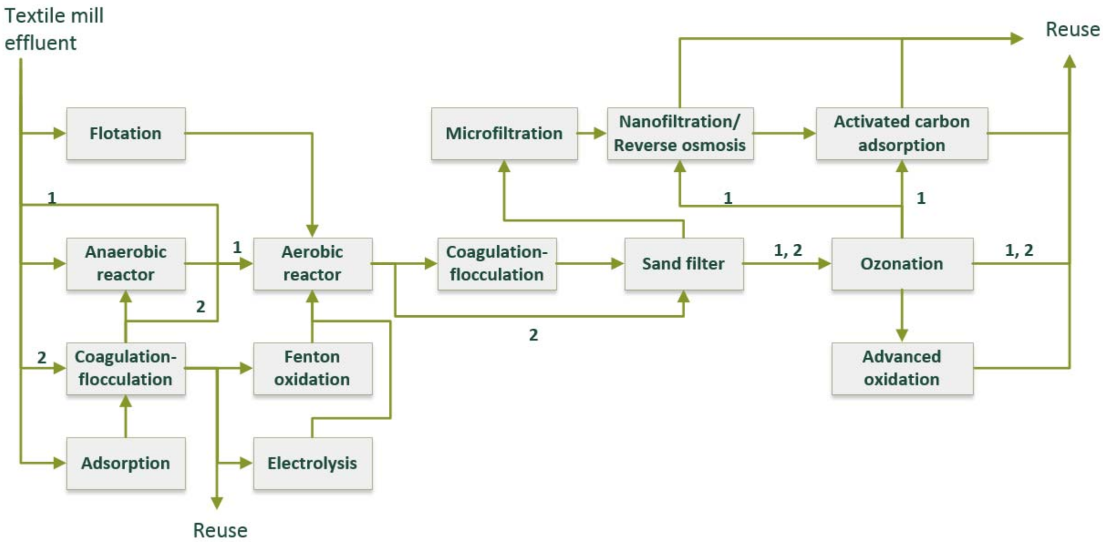


Figure 9 Ozone use schematic treatment examples (mod. from US EPA, 1986)

Also, when the treatment target is effluent reuse, some possible plant configurations can be described (Figure 10).



1: High Lura WWTP managed by CIDA Srl in the area of Como (Italy) treating 22,000 m³/d of a mixture consisting of 17% domestic WW, 32% rainwater, 51% textile finishing mill equalized effluent (cellulose, wool, silk and synthetic fibers. 2: Levi's Strauss WWTP, Wervik (Belgium).

Figure 10.- Compilation of possible technological, commercial and experimental processes for textile effluent treatment and reuse with ozonation units (modified from Vandevivere, 1998).

- **Multi-stage processes: ozonization and biodegradation**

The biological treatment is usually more efficient in terms of the removal process of organic contaminants. Unfortunately, not all contaminants are biodegradable. Designs combining treatment with chemical oxidation biological processes are based on the observation that many of the oxidation products of biorefractory contaminants become biodegradable. The purpose of the combination of both processes is to minimize the amount of oxidant needed and therefore reduce operating costs (Gottschalk, 2000).

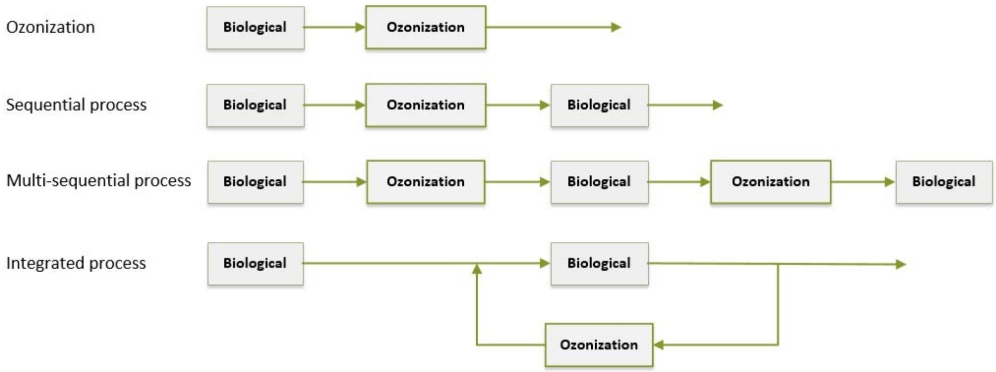


Figure 11.- Possible biological/chemical treatment schemes after biological treatment (Gottschalk, 2000).

In Figure 11 different configurations that are currently working on the combination of ozone oxidation processes with biological processes are shown. In several recent studies integrated system has proven to be the most efficient in the consumption of ozone per g of DOC removed in process.

According to studies Jochimsen (1997), the industrial water from tanning industry, including wet treatments and finishing, may be treated with a combination of oxidation processes with ozone followed by aerobic treatment. Ozone oxidation allows to increase the bioavailable organic matter through degradation of refractory organic matter, which is removed in the biological unit. Thus the removal efficiencies of COD and DOC (dissolved organic carbon) are increased.

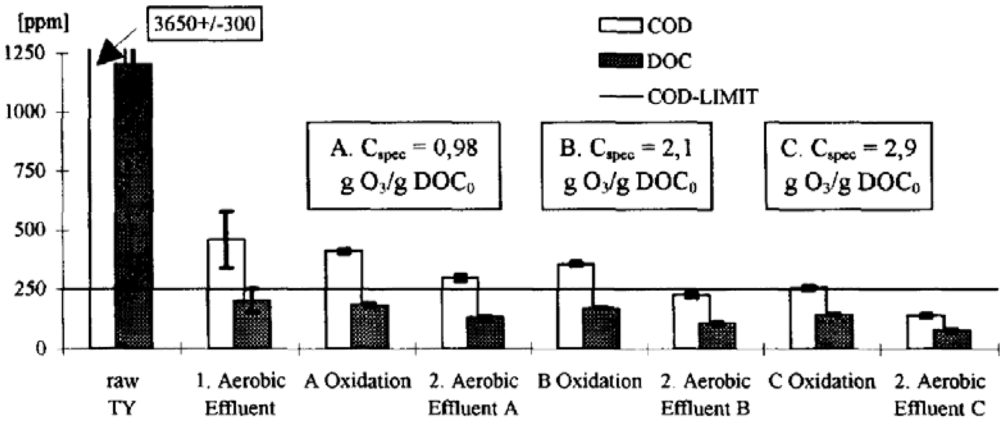


Figure 12.- Effluent quality parameters in successive stages with combined biological + ozone treatment to tannery effluent (Jochimsen, 1997).

In many real scale applications energy and oxygen cost variables are contemplated as decisive economically. Ozone savings are usually achieved by the application of combined ozonation/biodegradation systems. Today, in many cases, ozonation of wastewater is included as part of a multi-stage system employing biodegradation at least before and often posteriorly to chemical oxidation step (O_3-Bio-O_3 systems) system (Figure 12). In Table 1, characteristics of various textile treatment plants are summarized in Germany, primarily with direct discharge to natural media. It is observed that the color removal, for example, can be obtained with specific doses of ozone at low operating costs. On the other hand, the use of technology with high DOC removal (> 80%) in small plants can be very expensive.

In Germany, in 1999, 6 ozone treatments were found in textile industry, with a typical consumption greater than 0.13 g O_3/g ΔCOD.

Most used contactors in full scale ozonation systems are reactors with bubble column (BC) equipped with diffusers or injectors, mainly operating in a countercurrent reactor in series with continuous flow. Many of them work at high pressures (2-6 bar) to achieve high ozone transfer rates, increasing the efficiency of the process.

Table 1 Summary of technological characteristics, operation parameters and treatment costs in textile effluent treatment plants with ozonation (Gottschalk, 2000)

Treatment type	No. And tipe of ozone reactor (op. Pressure)	Ozone production capacity	nominal/ real liquid flow rate	Ozone yield coefficient $Y(O_3/M)$ (M=COD)	Invetment ozonation stage	Specific costs (without annuity)	Remarks
		Kg O_3 h/h	m^3/d	Kg O_2/kg ΔM	€ (1999)	€/m^3	
O_3/UV	1 BC (3 bar_{abs})	1-2	240 // 200-400	n. d.	n. d.	1.8	Sludge forming: valves stuck
Bio-O_3	4 BC (1 bar_{abs})	160	120.000	n. d.	n. d.	0.11	Mainly decolourization, oxidation of surfactants to <1.5 mg/L, wáter reuse in textile factories
Bio-O_3-Bio	1 BC	12	110 // 160	0.127; 0.343 * (M=DOC)	n. d.	n. d.	Decolorization, ozone not used for COD removal
TM-Bio-O_3	3 BC & 3 Bio en serie	5	1750 // 500	aprox.: 1.4	2.3 Mill.	approx. 2.5	Decolorization, ozone not used for COD removal

MT – mechanical treatment (filtration, sieving)
1 US $ - 1.8 DM

*specific ozone input (dosage given in kg O_3/kg MO
**cost for equipment, but without costs for construction of reactors (1999)

5. PARAMETERS AND CONTROL STRATEGIES

Ozonization requires several control on each unit of the system (EPA, 1986):
Air feed treatment systems:

- Low pressure air compressors monitoring
  - Discharge: pressure, temperature, flow
  - Seal water: temperature, flow
- After cooler monitoring
  - Feed gas: temperature
  - Discharge: temperature
  - Cooling water: inlet/outlet temperature, flow rate
- Dessicant dryer monitoring
  - Feed-gas: flow rate, inlet/outlet temperature and pressure
  - Purge gas: flow rate
  - Discharge gas: dew point

Energy supply

- Energy consumption meter generator.

Ozone generator

- Generator monitoring:
  - Inlet gas: flow rate, temperature, pressure.
  - Ozone discharge: concentration, temperature.
  - Electrical inlet: voltage, amperage, frequency, and energy consumption.
  - Cooling water: inlet/outlet temperature, flow rate.
  - Cooling air system: ambient air and inlet temperature, dew point
- Cooling water is prepared for worst conditions
- Gas ozone detectors should be placed in the areas where personnel work, as well as in the ozone off-gas destroyer outlet.

Ozone contactor

- Contactor monitoring:
  - There should be installed dissolved ozone sensors in the water and in the outlet of the contact chamber.

Ozone destructor equipment

- Process monitoring:
  - Inlet/outlet gas: temperature, flow rate, ozone concentration

In order to assess the performance of the unit, the control parameters that have been shown to be more efficient in assessing the performance of the process are (Gottschalk, 2000; US EPA, 1986; Sharma, 2013):

Oxidation process:

- Dissolved organic carbon or: Shows both increased bioavailable organic matter and subsequent biodegradation in combined processes.
- Biological oxygen demand (BOD_5).
- UV absorption (256 nm): Monitoring of dissolved organic matter

Color removal: Atomic absorption spectrophotometry: In case of known dyes, there is a specific wavelength to which study their degradation.
Disinfection: Fecal coliforms
Toxicity: Biological analysis.

6. PERFORMANCE

In the case of wastewater, common disinfection yields range between 99.9% - 99.99% so as to comply with discharge targets into natural media.
As for color removal, the usual objective is a colorless water discharge, which means a color removal> 99%.
The removal of organic matter, as seen above, varies depending on the parameter analyzed and the characteristics of the water to be treated. Generally, the COD decreases by mineralization while dissolved organic matter increases due to a greater amount of biodegradable available substances (Jochimsen, 1997).
Ozone use do not usually remove trace organics, but it commonly degrades them into their main metabolites, with a higher polar nature and smaller size. In order to achieve a more removal efficiency, this technique is combined with others like activated carbon adsorption or advanced oxidation processes (Gottschalk, 2000)
In Annex A1 examples of configurations used in ozonation stages in textile ETPs are included.

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ANNEX1

REAL SCALE EXAMPLES

Example 1 (Vandevivere, 1998)

The sewage works at Leek receives 60% of its total load (76000 inhabitant equivalents) from seven dyehouses. Following pilot-scale investigations showing that required color removal could be achieved with 9.5 ppm O_3 with an HRT of 20 min, a tertiary treatment involving lagoons, sand fillters and an ozonation plant was built at a capital cost of £5 million. In about 1 year, only four samples out of 39 analyzed have failed the color consent.

Example 2 (Vandevivere, 1998)

The Alto Lura WWTP (Como, Italy) treats a mixture of 75% textile and 25% municipal wastewater with a sequence of pre-denitrification, activated sludge, sand filtration and ozonation. The ozonator was built in 1992 in order to reduce surfactants and color in the final effluent. These goals are being achieved with 20 mg/L O_3 but unwanted by-products are formed, especially aldehydes in the range 0.5-2 mg HCHO/L. Plans are made to reduce aldehyde formation by adding a tertiary flocculation unit before the sand filters.

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ANNEX 2
GRAPHICAL DESCRIPTION OF UNIT PROCESSES

Figure 1.- Cooling system and oxygen storage (ETAP-Lugo).

Figure 2.- In-situ ozone generation equipment (left) and pressure and gas controllers (right). ETAP-Lugo.

Figure 3.- Detail of ozone generator. ETAP-Lugo.

Figure 4.- Ozone contactor. ETAP-Lugo.

Figure 5.- Commercial ozone generator example (Allied Power). 3 Kg/h of ozone generation with a 35 mg/L concentration – Current cost 65.000 – 81.000 US$ (Sept-2014).

Figure 6.- Horizontal ozone generators at industrial scale.
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