AN OVERVIEW OF THE AVAILABLE WATER TEXTILE EFFLUENT TREATMENTS

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Abstract

The textile industry is one of the most significant contributors to water pollution, with the dyeing and finishing processes being major culprits. The effluent generated from these processes contains various chemicals and dyes that can harm aquatic life and pose a significant threat to human health. Several effluent treatments have been developed to minimise the impact of these effluents on the environment. Fashion is vital to the textile industry supply chain, which determines consumer demand and industrial production. Sustainability requires action from all parts of the chain. Communication and understanding, however, are not always possible due to the different fields of knowledge involved. This overview discloses the available solutions for liquid effluent treatment from textile dyeing and finishing providing a fast, precise and deep understanding of methods such as the Physicochemical and Biological treatments and the recent Advanced Oxidation Processes.

Keywords: *textile wastewater, water pollution, effluent treatment, sustainable fashion*

1. INTRODUCTION

The textile industry is one of the most significant contributors to water pollution, with the dyeing and finishing processes being major culprits. To put things in perspective, 200-350 m^3 of wastewater is produced per ton of finished product (Gozálvez-Zafrilla *et al.*, 2008) and as such, yielding the average pollution levels of 100 kg COD per ton of fabric (Verma, Dash and Bhunia, 2012)

The effluent of these chemical processes, pre-treatments, dyeing and finishing, contains an immense volume of significant pollutants such as a higher concentration of chemical oxygen demand (COD), biological oxygen demand (BOD), suspended solids (SS), as well organic nitrogen, traces of heavy metals. The auxiliaries used in the processes include dispersants, levelling agents, salts, carriers, acids, alkalis and various dyes. (Velusamy *et al.*, 2021) Heavy metals, elements of nitrates, nitrites and phosphates and dyes are harmful to both aquatic and human life due to toxicity, carcinogenicity and absence of biodegradability and cause eutrophication, as they interrupt the photosynthesis of marine plants (Berradi *et al.*, 2019) (Verma, Dash and Bhunia, 2012). Halophilic microbes of textile effluent can convert presumably non-toxic azo dye into highly persistent/mutagenic by-products at salinity levels similar to effluent (Rawat *et al.*, 2018).

In the meantime, the water clarity and quality requirement are critical for the uniform dyeing process and, in general, for efficient wet processing. The shortage of clean water, environmental pollution and water abuse threaten global industry and society. Effluent treatment and recycling are promising solutions to the continuously growing water requirements at an alarming rate.

The EU Directive (Directorate-General for Environment, 2023) presents a clear waste framework of basic waste management principles. It is crucial to handle waste in a manner that poses no threat to human health, the environment, water, air, soil, plants or animals and that does not create noise or odour disturbances or harm the countryside. The directive prioritises preventing waste, reuse, recycling, and recovery, with waste disposal as the final option. Furthermore, the Extended Polluter Pays policy puts the onus on producers to take responsibility for their goods' impact on the environment at the end of their useful life. **Fig. 1** gives a visual representation of these priorities.

As prevention, the sustainability measures intend to improve processing methods minimising water usage during wet processing, and lowering water consumption and wastewater effluent. As a result, methods such as anhydrous supercritical dyeing, solvent dyeing, dyeing with liquid ammonia, and reverse microemulsion dyeing methods (Varadarajan and Venkatachalam, 2016), (Lara, Cabral and Cunha, 2022), (Kabir *et al.*, 2019) enzymatic treatments, (Sen *et al.*, 2021) layer by layer techniques (Uğur, 2023) and foam dyeing (Mohsin and Sardar, 2019) have been reported.

Some of these technologies are yet to be commercialised and remain in laboratory practice. However, these technologies are not generic and cannot be applied to all circumstances; for example, unlike disperse dyes, reactive dyes are insoluble in carbon dioxide, and the technique faces the ecological limitation of the hydrocarbon solvents (Banchero, 2013) (Fu *et al.*, 2015). Surface modification of the substrate with plasma treatment (Haji and Kan, 2021) reduce effluent discharge. Although effluent volumes can be reduced with innovative technologies, fabric preparation and further processing still release water effluent that must be treated.

Fig. 1. EU Strategy in waste handling (Source: EU-Directive Waste Framework Directive, https://environment.ec.europa.eu/topics/waste-and-recycling/waste-framework-directive_en, Accessed on 05/07/2023)

Sustainability comprises a joint effort from all members involved in the chain who must communicate and understand the standard strategy and available options to implement it. This work intends to give a comprehensive general overview of the available solutions for liquid effluent treatment from textile dyeing and finishing using Physicochemical and Biological treatments and the recent Advanced Oxidation Processes.

2. PHYSICAL METHODS

2.1. Adsorption

The adsorption method involves the use of adsorbents such as activated carbon, zeolites, and clays which remove pollutants from wastewater by attracting and binding pollutants onto their surfaces which decolourises and removes organic matter and heavy metals (like Cd, Pb, Cr, Zn, Cu, Co, Hg and As) from dyehouse effluents.

Strong adsorbents can be produced by the traditional pyrolytic method or the alternative hydrothermal carbonisation method (HTC). The latter offers several advantages, such as less power consumption due to lower temperatures involved and the lack of drying stage of the biomass raw material as well as being less hazardous, causing reduced wear of materials employed, and lastly, the lack of pyroligneous acid, tar and contaminating gas, products which are released by the environmentally non-friendly pyrolytic process. The process involves carbonisation of the raw material, which is usually biomass, such as glucose, starch, fructose or lignocellulosic materials and industrial waste, such as beer and livestock waste, swine manure or chitin from prawn shells. The process of HTC involves temperatures of up to 300°C where desorption and dehydration occur, unlike the pyrolytic process where temperatures rise to 700°C with a more porous material. The hydrothermal process can be assisted by templates like graphite oxide to obtain monolith (Martín-Jimeno *et al.*, 2015) or by employing the "salt-templating" method with $ZnCl_2$ (Roldán, Marco and García-Bordejé, 2016) and LiCl-$ZnCl_2$ mixture (Alatalo *et al.*, 2016) Activation is necessary to develop the specific surface and volume of the micropores performed as physical activation in a medium of air, inert gas or CO_2 which shows a beneficial effect on the activated material pore size. Chemical activation is performed with liquid or solid reagents, usually KOH but NaOH (Zhao *et al.*, 2016) and H_3PO_4 (Hao *et al.*, 2014). Activation can be performed up to 400°C without damaging any functional groups present or induced by coating at an earlier stage. (Durán-Valle, Botet-Jiménez and Omenat-Morán, 2017) The functionalisation stage is performed in single or two steps to change the surface chemistry of the carbonaceous material, introducing oxygen functional groups and increasing the adsorption capacity, or nitrogen and sulphur atoms, if they are not present in the raw material, for enhanced dye retention (Roldán, Marco and García-Bordejé, 2016). Alternatively, oxidation performed with potassium permanganate in a strongly acidic medium (Li *et al.*, 2016), with nitric acid (Liu *et al.*, 2013) and with hydrogen peroxide (Xue *et al.*, 2012) has been reported in the literature. This process is applied between 200-300°C where oxygen chemisorption is possible, resulting in functional group formation. The functional groups on the surface of the adsorbent promote its regeneration with acid or alkali treatment. (Durán-Valle, Botet-Jiménez and Omenat-Morán, 2017)

It is a cost-effective and efficient method regarding heavy metal removal from textile effluents, as the adsorbent can be regenerated and reused multiple times for different purposes. Various methods, such as electrochemical regeneration, thermal generation, and pressure swing method, are considered environmentally friendly for adsorbent regeneration.

The process's efficiency depends on the polarity of the absorbent, its surface area, pore size distribution and dosage, pH, temperature and mixing speed. Increased interest towards nanotechnology has led recent studies such as the one of (Velusamy *et al.*, 2021) to gravitate towards the usage of graphene as an adsorbent, primarily for its low cost but as well as its high surface area, chemical stability, convenience of regeneration and high range of possible uses.

Additionally, other characteristics render graphene a suitable adsorbent, high impermeability to gases, high porosity, high optical transmittance, high elasticity, tunable bandgap, and biocompatibility. However, graphene cannot be dissolved or dispersed in other solvents, and because of that, the sorption of the metal ions is significantly reduced. For this reason, graphene oxide (GO) is used instead of graphene as it is more hydrophilic and thus has a better affinity with heavy metals. (Velusamy *et al.*, 2021)

2.2. Membrane Filtration

Membrane filtration uses membranes of different pore sizes, such as microfiltration, ultrafiltration, nanofiltration and reverse osmosis, to remove the pollutant. This type of water treatment is one of the most effective in effluents from dyeing and other processes, such as mercerising and bleaching the fabrics. However, it is a costly method due to the membranes' delicate nature and high-power demands. Almost all cases, wastewater must be pre-treated beforehand to prevent membrane fouling and regain effectiveness. After their use, proper cleaning processes are required to prevent damage. Some factors that may cause fouling may be dissolved inorganic or organic compounds, colloids and bacteria. Maintaining and frequently cleaning (back-washing) the membranes is a prerequisite to retaining their efficiency and proper function. (Keskin *et al.*, 2021) Other than that, it is a relatively simple and highly reliable method.

There are different types of filtrations, and each uses a specific type of membrane, **Fig. 2**. Reverse osmosis, ultrafiltration, microfiltration, nanofiltration, and electrodialysis are the main types of membranes. (Velusamy *et al.*, 2021)

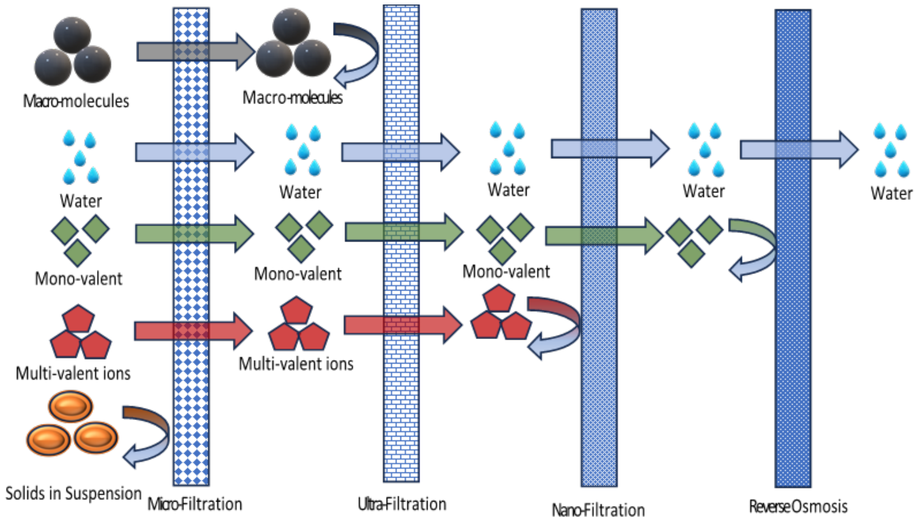

Fig. 2. Types of filtration membranes available

3. CHEMICAL METHODS

3.1. Coagulation – Flocculation

This method uses chemical and physical processes to remove pollutants such as suspended solids, colour, and organic matter from wastewater. The addition of coagulants – flocculants to the wastewater destabilises the suspended particles, causing them to clump together, which in sequence can then be removed by settling or filtration. Adding a flocculant (flocculation) helps the formed particles aggregate and settle more efficiently, removing colour, suspended solids, and organic matter from textile effluent.

Coagulant types, dosage, pH, temperature, alkalinity, and mixing conditions are all important.

Coagulation is very efficient in removing heavy metals from wastewater. However, the major drawback is that it generates secondary pollutants, other than sludge, severely affecting human health and the environment. Therefore, secondary treatment is required in order to make these pollutants safe.

Dyes exhibit low biodegradability due to their high molecular weight and complex structures. The process plays a large part in wastewater characterised by high toxicity, high dissolved solids and high pH. Coagulation–flocculation seems to be the most suitable method of wastewater decolourisation and has been used for many years thanks to its low cost, **Fig. 3**. However, due to their high water solubility and new innovative formulas, difficulty occurs when faced with the choice of the most suitable coagulant. Coagulation–flocculant is a method that produces large quantities of sludge. This phenomenon can be reduced if chemicals that promote coagulation are incorporated into the effluents, such as polyelectrolytes. (Verma, Dash and Bhunia, 2012) Moreover, sludge can have various uses in other fields, functioning as raw material for construction, as fertiliser and other pollutants adsorbent (i.e. fluoride, perchlorate, hydrogen sulphide, sulphur dioxide, and carbon dioxide) (Zhao *et al.*, 2021).

Along with the environmental impact of chemical coagulants – flocculants in mind and the health hazards they are linked with, many studies for green methods emerged. Chitosan, a naturally occurring polymer, can be used as a highly efficient and biodegradable flocculant (Ariffin *et al.*, 2009). Other natural coagulants extracted from corn, green beans, strychnos potatorum (Nirmal seeds) and moringa have also proven highly effective. (Shruthi Keerthi and Mukunda Vani, 2022)

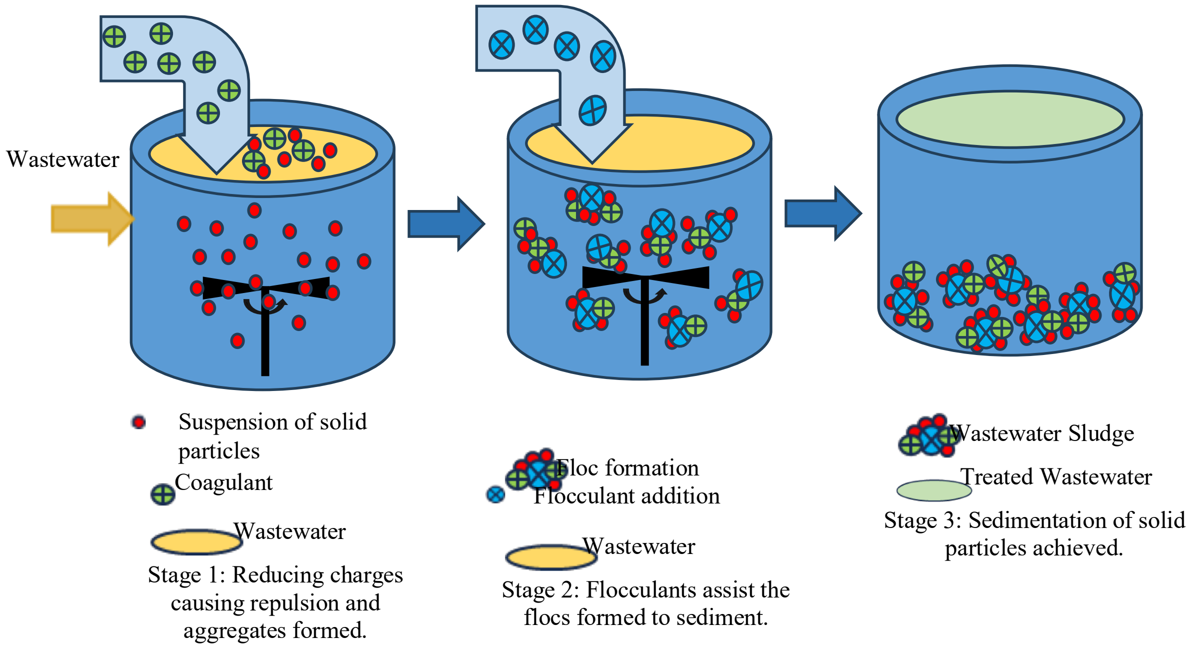

Fig. 3. Simplified diagram of the coagulation-flocculation treatment of wastewater

3.2. Advanced Oxidation Processes

Advanced oxidation processes (AOPs) are a group of chemical treatment methods that involve the generation of highly reactive hydroxyl radicals to oxidise and degrade organic pollutants in the effluent. These processes are effective in treating dye and particularly chromophoric structures (Rather *et al.*, 2019), chemical oxygen demand (COD), and total organic carbon (TOC) from textile effluent. AOPs have the advantage of not requiring the addition of expensive chemicals, which can produce sludge and can be combined with other treatment methods to achieve the desired treatment clarity level. (Paździor, Bilińska and Ledakowicz, 2019)

Zhang (in Table 2 2021) reported different types, including ozonation, photochemical, photocatalysis, Fenton and Photo-Fenton, electrochemical, and ultrasound AOPs, each with its benefits and drawbacks. (Zhang *et al.*, 2021) Generally, the AOPs are highly effective in comparison to the other methods due to their in situ generation of hydroxyl radicals (HO•), which attack pollutants in water at high speeds through hydrogen abstraction, electron transfer, and radical addition (Zhang *et al.*, 2021)

AOP is effective against pollutants. The hydroxyl radical HO• is highly reactive with the organic species in the effluent, while the radical self-eliminates with a short life, hence not imposing subsequent hazards (Gautam, Kumar and Lokhandwala, 2019)

Indicatively, three of the above AOPs will be presented.

Ozonation is an AOP method that can decompose pollutants in two different ways, depending on the pH levels of the solution. The first one involves the immediate dissolving of the components in the water as they are directly attacked by molecular O_3, while. In contrast, the second one uses ozone to liberate the hydroxyl radicals, which oxidise and decompose the pollutants of the wastewater. (Zhang *et al.*, 2021)

Unfortunately, ozonation exhibits low efficiency as ozone has low solubility and escapes the wastewater. Increased ozone doses are needed to sufficiently complete the treatment and avoid the formation of toxic by-products formaldehyde, ketones, phenols, nitromethanes, bromates, and N-nitrodimethylamine. (Miralles-Cuevas *et al.*, 2017; Zhang *et al.*, 2021).

Peroxidation is performed to increase the decomposition rates of O_3 by adding H_2O_2 (O_3/ H_2O_2 systems), favouring the formation of the free radical $HO^{\bullet}$. Catalysts are frequently used to increase oxidation rates and increase treatment efficiency. Dissolved transition metals can be responsible for catalytic effects assisting the homogenous catalytic oxidation. In heterogeneous catalytic oxidation, the catalyst's surface of metal oxides, metals supported on oxides, minerals, and activated carbon aid the reaction. The pH is crucial in the reaction between O_3 and H_2O_2. The reaction is slow at low pH and becomes faster at higher pH. It should be kept in mind, however, that textile dyeing of cellulosics is performed in a strong alkali environment. Hence, the effluent's pH is high and rich in OH^-, which causes strong competition reactions limiting the efficiency of radical formation with O_3. (Zhang *et al.*, 2021)

The mechanism of action is also pH dependent. At acidic pH, the O_3 directly attacks the electron-rich functional groups by cycloaddition, nucleophilic or electrophilic reactions causing cleavage of double bonds, aromatic rings and amines. As a result, aldehydes, carboxylic acids, and other by-products are formed. (Gautam, Kumar and Lokhandwala, 2019) At high pH, the reaction of O_3 with OH- releases radical species HO• via a series of chain reactions. (Gautam, Kumar and Lokhandwala, 2019)

In Electrochemical oxidation, the wastewater is passed through cells with an electric current with no addition of external chemicals. There is no need for adding H_2O_2 as it is produced in situ within the electrolytic medium simply by the O_2 supplied at the cathode surface in lower pH conditions. (Zhang *et al.*, 2021) Oxidation is performed via two different methods. Direct oxidation occurs at the anode's surface, degrading the pollutants in the wastewater, and indirect oxidation occurs when oxidants generate the liquid electrolyte. (Zhang *et al.*, 2021)

The Fenton process mechanism can be seen in the following starting and intermediate equations (Haber and Weiss, 1934 in Gautam, Kumar and Lokhandwala, 2019)

$$Fe^{2+} + H_2O_2 \longrightarrow Fe^{3+} + \bullet OH + OH^-$$

$$Fe^{3+} + H_2O_2 \longrightarrow Fe^{2+} + \bullet O_2H + H^+$$

$$H_2O_2 + \bullet OH \longrightarrow H_2O + \bullet O_2H$$

$$Fe^{2+} + \bullet OH \longrightarrow Fe^{3+} + OH^-$$

$$Fe^{3+} + \bullet O_2H \longrightarrow Fe^{2+} + O_2H$$

$$Fe^{2+} + \bullet O_2H + H^+ \longrightarrow Fe^{3+} + H_2O_2$$

$$2 \bullet O_2H \longrightarrow H_2O_2 + O_2$$

$Fe^{2+}$and H_2O_2 are combined under acidic conditions and a large number of hydroxyl radicals $HO^{\bullet}$ is generated. The excess H_2O_2 causes the rapid oxidation of ferrous ions Fe^{2+} to ferric ions Fe^{3+}. The ferric ion Fe^{3+} is reduced to ferrous ion Fe^{2+} and the reaction propagates rapidly. Over-optimum concentrations of H_2O_2 seem to produce less reactive species that behave as radical scavengers inhibiting the oxidation process. (Zhang *et al.*, 2021)

The electro-Fenton process is a mixed process involving electrochemical oxidation and the Fenton process. Similarly, H_2O_2 is produced in situ at the cathode by the O_2 supplied at low pH conditions. At the same time, hydroxyl radicals $HO^{\bullet}$ are concurrently generated on the cathode by the electrogeneration of H_2O_2 and the electroregeneration of Fe^{2+}. The ferric Fe^{3+} ions are reduced to Fe^{2+}.(Zhang *et al.*, 2021)

The Photo-Fenton process is even more efficient as it uses ultraviolet (UV) light to provide additional energy to the system, activating the hydrogen peroxide (H_2O_2) ferrous (Fe^{2+}) and ferric Fe^{3+}ions which liberates more oxidising hydroxyl radicals $HO^{\bullet}$. This method can be cost-effective and environmentally friendly, using sunlight as the UV source. (Zepp, Faust and Hoigne, 1992) (Zhang *et al.*, 2021)

Whereas the Fenton process is generally a cheap and efficient method, its main drawback is that it can only occur in acidic environments (pH 2-4). In contrast, textile effluents are most of the time alkaline. Therefore, pre-treatment of the effluents is required prior to the procedure. (Zhang *et al.*, 2021)

4. BIOLOGICAL METHODS

The biological treatment uses the activated sludge process, biological aerated filter, and sequencing batch reactor. (Madhav *et al.*, 2018) The activated sludge process involves mixing the wastewater with sludge and aerating the mixture to provide oxygen to the microorganisms, decomposing the organic matter, and finally separating and reusing the active sludge. The biological aerated filter method involves passing the wastewater through a media bed populated with the microorganisms responsible for decomposing the organic matter. Similarly, the sequencing batch reactor method also involves introducing wastewater in an aerating tank for a controlled period, providing oxygen to the microorganisms, which in stages decompose the organic matter, settle the suspended solids, and eventually decant the treated water, **Fig. 4.**.(Madhav *et al.*, 2018)

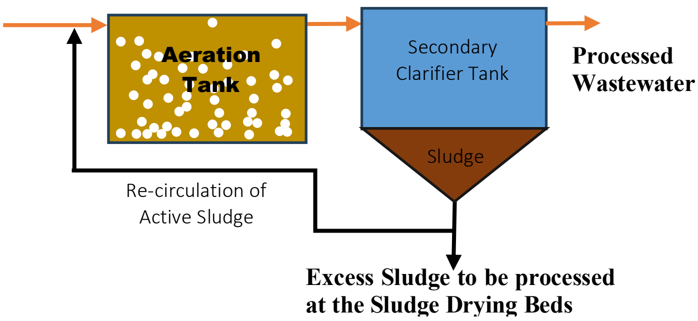

Fig. 4. Simplified diagram of active sludge recirculation in biological methods

Biodegradation in an aerobic environment uses aerobic micro-organisms that utilise oxygen as the hydrogen acceptor during respiration. Anoxic and hypoxic environment conditions lack oxygen, and the micro-organisms can adjust to using other sources, such as sulphates, nitrates, and carbon dioxide, as hydrogen acceptors. (Birch *et al.*, 1989)

The anoxic process entails the digestion of the organics such as carbohydrates, fats or proteins by acidogenic bacteria. As a result of this process produces metabolites of low molecular weight like alcohols and short-chain fatty acids, and the subsequent acetogenic bacteria release acetate, carbon dioxide and hydrogen. Ultimately the methanogenic bacteria reduce acetate and carbon dioxide to methane, releasing biogas. (Slokar and Majcen Le Marechal, 1998)

Azoreductases are enzymes commonly found in many bacteria capable of causing cleavage of the double bond of azo dyes (Kamal *et al.*, 2022) which seizes the single double bond conjugation and affects the colouration of the dye, **Fig. 5.**

$$\text{AzoDye-R}_1\text{-N=N-R}_2 \xrightarrow[\text{by azoreductases enzymes}]{\text{Double bond cleavage}} \text{R1-NH}_2 + \text{R}_2\text{-NH}_2$$

Fig. 5..Cleavage of N=N double bond by azoreductase

The aromatic amines released in the anaerobic environment are fairly bio-transformationally stable and are equally cytotoxic, mutagenic or carcinogenic to azo dyes. However, aromatic amines can be degraded entirely under aerobic conditions. (Sponza and Işık, 2005) Therefore, a sequencing batch of anaerobic/aerobic systems can effectively biodegrade azo dyes in a two stages process of aromatic amine formation and mineralisation by non-specific enzymes through hydroxylation and ring opening of the aromatic compounds. (Zissi and Lyberatos, 1996)

5. COMBINED METHODS

Each of the methods mentioned above has many advantages and disadvantages. Most of the time, a combination of the methods above is used in textile wastewater treatment to achieve the best results possible at a lower cost. When combined with membranes, biological methods yield bioreactors effective in a broader range of dye types and produce smaller quantities of sludge. Membrane filtration (microfiltration and ultrafiltration) can be used after the use of a biological method as a post-treatment (a process known as Membrane BioReactors) if there is a large number of pollutants or if the biological treatment did not yield the desirable results. (Robeson, 2012)

Advanced oxidation/coagulation processes involve the combination of AOPs with coagulation-flocculation processes to remove pollutants. The AOPs oxidise and degrade the pollutants, while the coagulation-flocculation processes remove the formed particles. Hybrid Advanced Oxidation Processes (HAOPs) involve (Hybrid Advanced Oxidation Processes (HAOPs), the combination of two or more AOPs (e.g. ozonation and UV irradiation) to improve treatment efficiency and reduce treatment time. The selection of the appropriate AOP depends on the nature and concentration of pollutants present in the effluent. (Chakma, Das and Moholkar, 2015) Solar-assisted Photo-Fenton processes have been reported in the literature and are a promising treatment for countries with abundant sunshine. (Yasar and Yousaf, 2013)

6. CONCLUSIONS

Many wastewater treatments available today have been presented, some needing improved efficiency to become more commercially attractive. Much information is already published, and new ones are presented daily. This highlights the importance of this topic in attracting industry and academia. The scope was to provide a simple but firm understanding of these technologies to all members of the textile industry chain, with particular reference to fashion that does not master this field. However, they constitute the driving force of the sustainable strategy. A synergistic action is vital for the success of a clean and sustainable fashion.

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