Univerza v Mariboru

Fakulteta za kemijo
in kemijsko tehnologijo

Bachelor thesis

ANALYSIS OF FLOCCULATION/FLOTATION AS WASTEWATER PRETREATMENT

September 2021

Julia Olmedo Gómez

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WASTEWATER PRETREATMENT

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Student: Julia Olmedo Gómez

Student program: Engineering in industrial technologies, specialization in chemical
engineering, Erasmus+ international exchange program.

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Maribor, 2021

UNIVERSITY OF MARIBOR

FACULTY OF CHEMISTRY AND CHEMICAL ENGINEERING

Place and date: MARIBOR, 14.07.2021

On the basis of Article 330 of the Statute of the University of Maribor (Official Gazette of the Republic of Slovenia, No. 41/2021-UPB13), I issue the following

DECISION ON FINAL WORK

JULIA OLMEDO GOMEZ, student of 1st cycle ABS study programme Izmenjava UN, has satisfied all requirements and is allowed to compose the final work.

The topic of the final work is predominantly from the field of the chair/department/institute: KATEDRA ZA KEMIJSKO TEHNOLOGIJO.

Supervisors:

doc. ddr. ANDREJA NEMET, univ. dipl. inž. kem. tehnol., supervisor
ALEN ERJAVEC, mag. inž. tehn. varstva okolja, somentor/-ica

Title of final work:
Analiza flokulacije/flotacije kot predobdelave odpadne vode

Title of final work in English:
Analysis of flocculation/flotation as wastewater pretreatment

The deadline for submission of the final work is 30.09.2021. Final work shall be prepared in line with requirements of the guidelines: *Predloge z navodili za pisanje zaključnega dela* and submitted at the Student Affairs Office. Number of copies: 1. At the same time, a statement from the supervisor is submitted (and possible co-supervisor) on final work adequacy.

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red. prof. dr. Zdravko Kravanja, Dean

Po pooblastilu dekana:
prodekan
prof. dr. Matjaž Finšgar

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UNIVERZA V MARIBORU

FAKULTETA ZA KEMIJO IN KEMIJSKO TEHNOLOGIJO

Kraj in datum: MARIBOR, 14.07.2021

Na osnovi 330. člena Statuta Univerze v Mariboru (Uradni list RS, št. 41/2021-UPB13) izdajam

SKLEP O ZAKLJUČNEM DELU

JULIA OLMEDO GOMEZ, študent/-ka študijskega programa 1. stopnje UN Izmenjava UN, izpolnjuje predpisane pogoje in se mu/ji dovoljuje izdelati zaključno delo.

Tema zaključnega dela je pretežno s področja katedre/oddelka/inštituta: KATEDRA ZA KEMIJSKO TEHNOLOGIJO.

Mentorji:

doc. ddr. ANDREJA NEMET, univ. dipl. inž. kem. tehnol., mentor/-ica
ALEN ERJAVEC, mag. inž. tehn. varstva okolja, somentor/-ica

Naslov zaključnega dela:
Analiza flokulacije/flotacije kot predobdelave odpadne vode

Naslov zaključnega dela v angleškem jeziku:
Analysis of flocculation/flotation as wastewater pretreatment

Rok za izdelavo in oddajo zaključnega dela je 30.09.2021. Zaključno delo je potrebno izdelati skladno z navodili: *Predloge z navodili za pisanje zaključnega dela*, in ga oddati v pristojnem referatu članice. Število izvodov: 1. Hkrati se odda tudi izjava mentorja/-ice (in morebitne/-ga somentorja/-ice) o ustreznosti zaključnega dela.

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red. prof. dr. Zdravko Kravanja, dekan

Po pooblastilu dekana:
prodekan
prof. dr. Matjaž Finšgar

Obvestiti:
- kandidata/-ko,
- mentorja/-ico,
- somentorja/-ico,
- arhiv.

INDEX

DECLARATION

I declare that I have written this thesis myself. Any contribution made by others are indicated separately. I have reviewed the literature in the field of my thesis under the following keywords:

Source: Science Direct (https://www.sciencedirect.com/)

Keywords:	Number of references
AOP	27,335
n-Hexane extractable material	31
Wastewater pretreatment	530
Flocculation/flotation	730

Source: COBISS+ (https://plus.si.cobiss.net/opac7/bib/search/advanced?db=cobib)

Keywords:	Number of references
AOP	1,188
n-Hexane extractable material	4
Wastewater pretreatment	74
Flocculation/flotation	12

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Maribor, September 2021   Julia Olmedo Gómez

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ANALYSIS OF FLOCCULATION/FLOTATION AS WASTEWATER PRETREATMENT

ABSTRACT

The purpose of this bachelor thesis is to optimize pretreatment of a wastewater from cosmetic industry. The current cleaning process in industry consists of two steps, namely i) AOP (Advanced Oxidation Process), and ii) flocculation/flotation (FF). However, as the HEM (n-Hexane Extractable Materials) decreases the efficiency of the AOP process by heavily blocking the light to penetrate in the wastewater to be cleaned, a first FF in acid environment is introduced in the process as pretreatment. The parameters that were varied to carry out the optimization were i) $Fe_2(SO_4)_3$ content added in first FF and, ii) H_2O_2 content. To precise, JAR tests were carried out to make the FF and the sample was put in a 1.8 L reactor and subjected to UV light in the AOP treatment. For the better distribution of the H_2O_2 and $Fe_2(SO_4)_3$ addition to the cleaning process the design of experiment was used. The HEM measured was carried out according to standard method "United States Environmental Protection Agency (EPA), Method 1664, Revision B: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry". We found that the percentage of removal is very high, it goes from 96.3% till more that 99.9%. In average, with this new first FF, the percentage of removal is 98.61% which represents a very high enough removal rate of HEM to achieve the WW disposal limits in most cases. Only 3 out of 9 experiments exceeded the emission limit of 100 mg/L of HEM due to the type of surfactant, which we can conclude it has a strong influence in the removal of HEM.

Keyword: AOP, n-hexane extractable material, wastewater pretreatment, flocculation/flotation.

UDK: 628.335+628.349(043.2).

Analiza flokulacije/flotacije kot predobdelave odpadne vode

Povzetek

Namen tega diplomskega dela je optimizacija predobdelave odpadne vode iz kozmetične industrije. Sedanji process čiščenja v industriji je sestavljen in dveh korakov: 1) napredni oksidacijski procesi (ang. Advanced Oxidation Process-AOP) in 2) flokulacije/flotacije (FF). Prisotnost težkohlapnih lipofilnih snovi (TLS) znižuje učinkovitost AOP z preprečevanjem penetracije svetlobe v odpadno vodo, ki jo čistimo, zato smo uvedli predobdelavo FF v proces v kislem okolju. Parametri, ki smo jih spreminjali tekom optimizacije so bile: 1) dodatek $Fe_2(SO_4)_3$ v prvi FF in 2) dodatek H_2O_2. FF process smo izvedli v t.i. Jar testu in potem smo 1.8 L vzorca smo dali v reaktor, ki smo ga izpostavili UV svetlobi v AOP obdelave. Za boljo razporeditev dodatka H_2O_2 in $Fe_2(SO_4)_3$ v process čiščenja smo uporabili načrtovanje eksperimentov. Meritev LS smo izvedli glede na standard United States Environmental Protection Agency (EPA), Method 1664, Revision B: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry". Ugotovili smo, da je bila uspešno odstranjevanja zelo visok, od 96,3% pa vse do 99,9%. V povprečju z novo začetno FF kar predstavlja dovolj visoko odstranjevanja TLS za doseganje mejne vrednosti za izpust odpadne vode v večini primerov. Samo 3 od 9-ih poskusov presegajo mejno vrednost TLS 100 mg/L, zaradi tipa površinsko aktivnih snovi, za katere lahko zaključimo, da ima velik vpliv na odstranjevanj TLSa.

Key words: AOP, TLS, predobdelava odpadne vode, flokulacija/flotacija.

UDK: 628.335+628.349(043.2).

LIST OF TABLES

LIST OF FIGURES

APPLIED SYMBOLS AND ABBREVIATIONS

Symbols

γ^{HEM}	mass concentration of HEM (mg/L)
m^{FLASK}	tare mass of the flask (mg)
m^{HEM}	mass of the dried HEM (mg)
$m^{HEM+FLASK}$	mass of the flask with the dried HEM in it (mg)
V^{WW_sample}	sample volume (L)

Abbreviation

AOP	Advanced oxidation process
BOD	Biochemical oxygen demand
COD	Chemical oxygen demand
EPA	United States Environmental Protection Agency
FF	Flocculation/flotation
HEM	n-Hexane extractable material
LLS	Low-volatile lipophilic substances
S1	Surfactant 1
S2	Surfactant 2
SS	Suspended solids
TOC	Total organic carbon
UV	Ultraviolet
W2FF	Wastewater after the second flocculation/flotation process
WW	Wastewater
WWE	Wastewater for experiments

1 Introduction and problem statement

1.1 Water consumption overview

Nowadays, water pollution is, without any doubt, one of the principal factors involved in human development. Over the years, urban areas become larger and larger and, with them, the demand for water has grown, as has water consumption per person per day. Water scarcity is a huge problem that affects not only human beings, but flora, fauna and climate. For that reason, it requires of investigations about how to increase water quality and the wastewater treatment, by environmentally sustainable processes. In this regard, as difficult as it may seem, more than one million people in Europe do not have access to clean water sources. This means that approximately 2 % of the population does not have access to adequate water supply and sanitation. According to the World Health Organization (WHO), 50-100 liters of water per person per day are needed to ensure that basic needs are achieved. (Fundación AQUAE, 2021). Economic activities in Europe consume an average of around 243,000,000 liters of water annually, according to the Water Exploitation Index. Although, most of this water (more than 140,000,000 liters) is returned to the environment, it often contains impurities or pollutants, including hazardous chemicals.
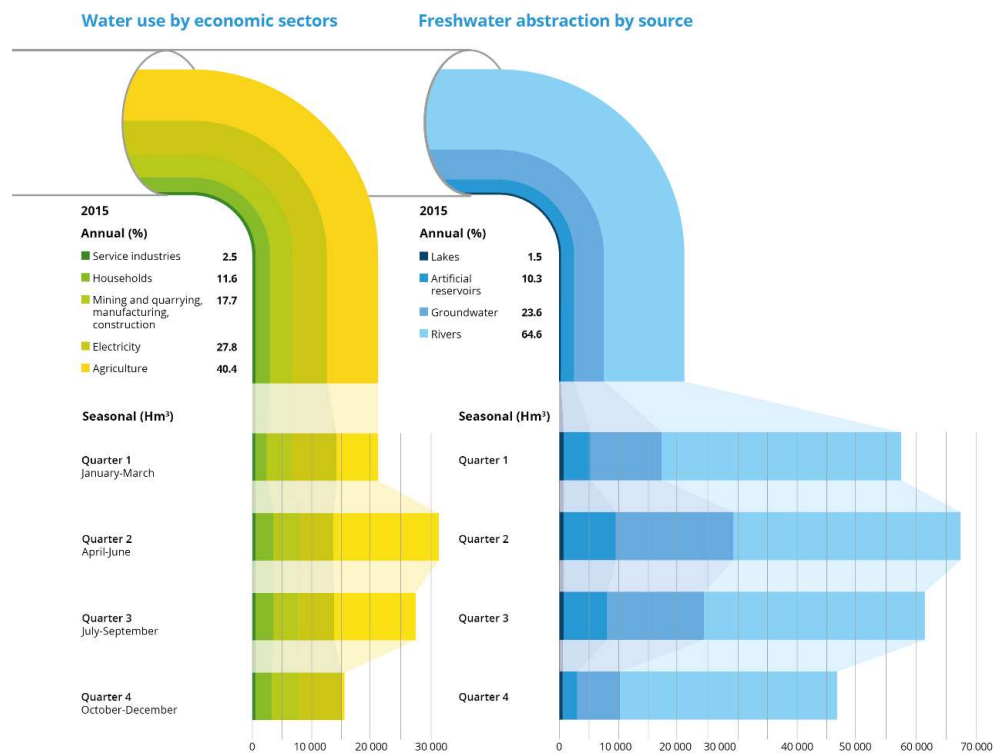
Figure 1-1: Water use in Europe (after European Environmental Agency, 2019)

In the Figure 1-1, we can see, in summary, water use in Europe and the freshwater abstraction by source both on an annual and quarterly basis. The largest consumption of water corresponds to agriculture: around 40 % of the total water consumed annually in Europe. Despite efficiency improvements in the sector since the 1990s, agriculture will remain the largest consumer over the coming years, increasing water stress in Europe. Power generation also consumes a large amount of water, representing about 28 % of its annual consumption. Water is mainly used as a cooling medium in nuclear and fossil fuel power plants. It is also used to generate

hydroelectricity. Mining and manufacturing industry account for 18 %, followed by domestic consumption, which accounts for about 12 %. An average of 144 liters of water per person per day is supplied to European households. (Agencia Europea de Medio Ambiente, 2018)

1.2 Wastewater treatment

We can consider wastewater (WW) as water that have been used at homes, industries or business and that must be treated before it is released back to the environment. It also includes water from the rain. (USGS, 2021). Conventional WW treatment plants often include the following steps (see Figure 1-2):

- Pretreatment: it refers to the transport of wastewater to the treatment plant and includes processes such as roughing, screening, desanding and homogenizing. In this stage, the inflow is measured and controlled, and larger floating solids are removed.
- Primary treatment: it includes the processes of decantation, flotation, clarification, filtration and neutralization. At this stage, flocculation/flotation (FF) is included to remove suspended solids. This process will be the one in which the thesis will go in depth and the one that will be optimized.
- Secondary treatment: or biological treatment, is characterized by the use of microorganisms to remove pollutants from water either aerobically or anaerobically. These bacteria or algae carry out a process of oxidation of the pollutants.
- Tertiary treatment: its aim is to remove the organic load and nutrients by ion exchange, adsorption, disinfection (e.g. chlorination). It also removes some specific pollutants such as phosphates. (Marcela Fúquene & Viviana Yate, 2018)

European Union has legislated in terms of water quality: on 23 October 2000, the "Directive 2000/60/EC of the European Parliament and of the Council establishing a framework for the Community action in the field of water policy" or, for short, the EU Water Framework Directive (WFD) was finally adopted. (European Comission, 2021).

This thesis is dealing with wastewater treatment in a cosmetic industry. The current cleaning process in industry consist of two steps, namely i) AOP (Advanced Oxidation Process), and ii) FF. The sequence of treatment follows the logic of first oxidizing large organic molecules and after removing them in the FF process. However, it might not be the most optimal way as the HEM (n-Hexane Extractable Materials) decreases the efficiency the AOP process by heavily blocking the light to penetrate in the WW to be cleaned. The idea of this work was to perform additional FF process as pretreatment before AOP process. From previous experiment it was concluded that FF process in acid environment might be a successful way to decrease the HEM content.
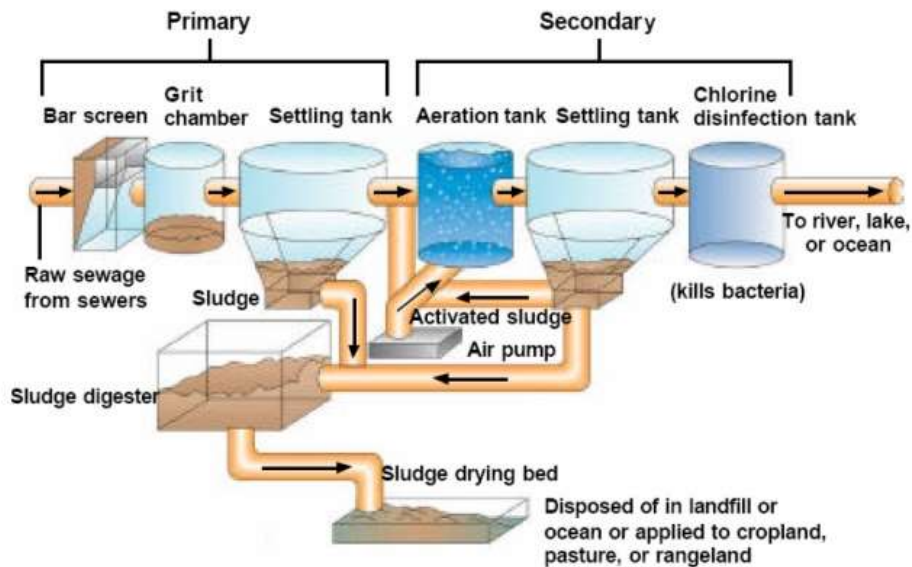
Figure 1-2: Scheme of a wastewater treatment plant (after TUHH, 2004)

Few industrial applications are found dealing only with AOP WW treatment. In Plataforma Solar de Almería in Spain, this process is applied to the pretreatment of wash water of pesticide containers. It is said to be economical and to be applicable to industrial pharmaceutical process (Vogelpohl, 2007).

The hypothesis is that if a first FF is carried out and optimize, the AOP could increase its efficiency because the UV light could penetrate deeper in the WW and decompose the remaining pollutants more successfully. That way, the stages that follow the treatment concerned to the thesis are:

i. **First FF in an acid environment**. In this stage, iron sulphate (III) ($Fe_2(SO_4)_3$) and a drop of polyelectrolyte are added to achieve the FF.

ii. **Advanced oxidation process (AOP).** The pH is maintained between 2-2.7 by adding hydrochloric acid (HCl). In this stage, hydrogen peroxide (H_2O_2) is added. In addition, the sample is subjected to UV radiation by means of a lamp in the reactor. The hydroxide (OH^-) ion oxidizes the organic compound present in the water into smaller molecules. Ideally, the end products are CO_2 and water, however this is rarely the case.

iii. **Second FF in an alkaline environment.** The pH has to be maintained above 8, so lime is added. Also, iron sulphate (III) ($Fe_2(SO_4)_3$) and a drop of polyelectrolyte is added to achieve FF.

1.3 Aim of the thesis

The aim of the thesis was to analyze the efficiency of flocculation/flotation as wastewater pretreatment before the AOP and second FF treatment. In this context the addition of $Fe_2(SO_4)_3$ in first FF and H_2O_2 before AOP process was investigated in order the assess the efficiency of the entire treatment process. The amount of H_2O_2 added before AOP and the amount of $Fe_2(SO_4)_3$ added before first FF were varied. The design of experiments was developed with the computer program Design-Expert. After the three-step: i) first FF, ii) AOP and iii) second FF WW treatment process was performed. The measure of HEM removal and sludge formation were the measures to assess the efficiency of the WW treatment.

2   Theoretical background

In this section, we will review the theoretical part of the problem that we will apply later for the experimental work.

2.1   Flocculation/Flotation

As we have seen FF is the principal process of this work. This covers two distinct stages:

  i.   Rapid mixing of dispersed coagulant into WW
  ii. Flocculation for agglomeration of small particles into large flocs by gentle agitation

Then, flocs formed are allowed to settle down/float and removed as sludge while the treated water is transferred into the next treatment process or for discharge into a watercourse. (Teh et al., 2016). The aim is the elimination of suspended solids (SS) and as much of the organic materials possible. To remove SS and organics, a floc forming chemical is needed. This is what is called the coagulant. Then, it will be separated from water by flotation, settling or adsorption. In this treatment, compounds such as ferric chloride or iron sulphate and/or polymer are added to the WW to destabilize the colloidal materials and this cause the small particles to join into larger settleable flocs (Amuda & Amoo, 2007). Coagulation is defined as the destabilization of suspension by making particles to aggregate. However, flocculation describes the process when the destabilized particles are made to contact each others, in order to form larger aggregates. Because of the size of colloidal particles (approximately 0.01 to 1 μm), attractive forces (Van der Waals) between particles are less strong than the repelling forces of the electrical forces. Due to this, particles tend to remain separated and dispersed in the suspension. To remove colloidal particles by settling or floating, the only practical way is if they settle/float rapidly. This means that particles must be larger. One destabilization method is the charge neutralization. It is achieved by adsorption of mononuclear and polynuclear metal hydrolysis species or polyelectrolytes on the surface of the particles. The most commonly used metal coagulants can be divided into aluminum and iron coagulants. The whole process can be seen in Figure 2-1.
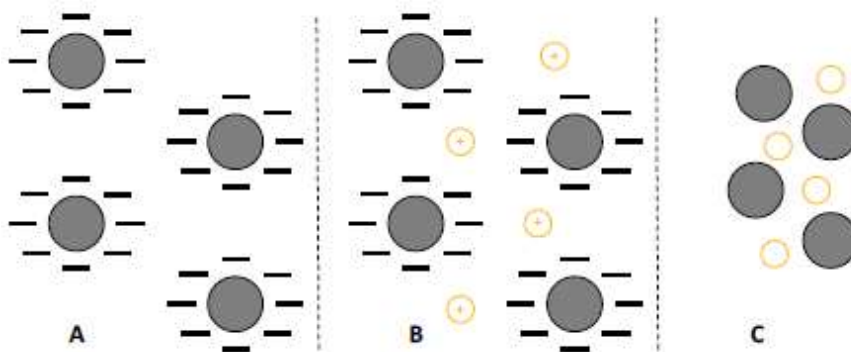

Figure 2-1: Scheme of coagulation. A) Colloidal particles, B) Addition of coagulant, and C) Destabilized colloidal particles (after Florencia Cevallos, 2015)

In this work, in the laboratory, we would use polyelectrolyte and iron coagulants: ferrous sulphate.

In addition to destabilization, flocculation is required as the next step to induce aggregation and settling/floating of large agglomerates. This is achieved by a gentle mixing of the suspension followed by letting aggregates to settle down/float. (Fig. 2-2) (Teh et al., 2016) (Marcela Fúquene & Viviana Yate, 2018)

Colloidal particles   Polyelectrolyte   Larger aggregates
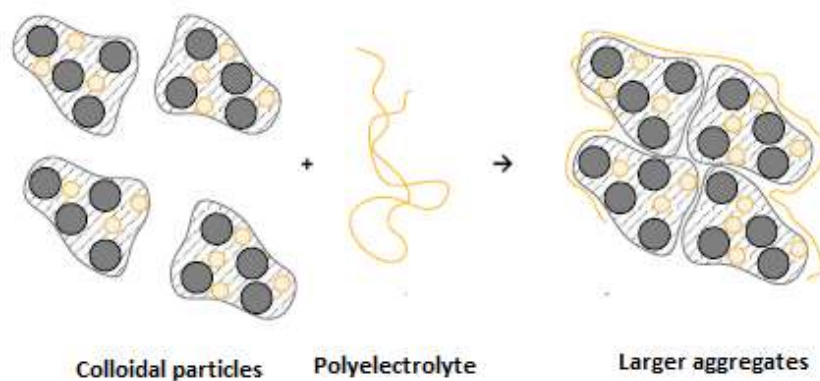
Figure 2-2: Diagram of how the addition of polyelectrolyte works (after Florencia Cevallos, 2015)

2.2 Advanced Oxidation Process (AOP)

The goal of the process is the elimination of non-degradable, poorly soluble compounds in wastewater. It consists of an oxidation reaction at atmospheric pressure and temperature. The necessary oxidant agent is the hydroxyl radical (OH·) that owns a very high capacity as oxidant, with short time of reaction. For this, reaction's speed is controlled by the diffusion (Florencia Cevallos, 2015). Due to high reactivity of hydroxyl radical, it is possible to remove organic and inorganic compounds achieving a reduction of BOD, COD, TOC and toxicity in wastewater. The process is particularly useful for solutions containing aromatics, pesticides, petroleum constituents, and volatile organic compounds. In addition, AOP could be used to treat effluents of secondary treated WW which then go to the tertiary treatment. The pollutants are converted into smaller inorganic compounds such as water, carbon dioxide and salts, and finally they undergo sedimentation (Liu et al., 2021). Hydroxyl radicals are produced with the help of one or more primary oxidants (ozone, hydrogen peroxide, oxygen) and/or energy sources (UV light) or ferric/ferrous salts. AOPs are characterized by its low selectivity and its high speed (Florencia Cevallos, 2015). The whole reaction of oxidation could be divided into three parts:

    i. Formation of $OH^{·-}$.
    ii. Initial attacks on target molecules by OH^{-} and their breakdowns to fragments.
    iii. Subsequent attacks by OH^{-} until ultimate mineralization.

The mechanisms of OH• formation depend on the technique of AOP that will be used. In this thesis the Fenton like process is assumed to happen. It presents high efficiency and low cost. The process consists of mixing hydrogen peroxide with iron salts (Fe^{2+} or Fe^{3+}). The ferric/ferrous ion acts as a catalyst for the formation of hydroxyl radicals (OH•). The mechanism of the reaction is the following one:

    1. $Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{-} + ·OH•$ (1)
    2. $Fe^{2+} + ·OH \rightarrow Fe^{3+} + OH^{-}$ (2)
    3. $R\text{-}H + HO• \rightarrow$ oxidated products   (3)

Reaction number 1 corresponds to the formation of hydroxyl radicals. Then, they react by oxidating the ferrous cation (reaction 2) and, finally, they attack the organic compounds (reaction 3). The formation of radicals is catalyzed by the use of radiation UV (Photo-Fenton like Process). This increases the degradation speed of pollutants. The process is homogenous type as both chemical species are in aqueous phase. Usually, the increase in the concentration of reactants increase the efficiency and the kinetics of the reaction. However, an excess in iron concentration produces secondary reactions such as the formation of iron hydroxide; and an excess in hydrogen peroxide may result in a degradation of itself by the hydroxyl radicals (Florencia Cevallos, 2015). In our work, we will combine the Fenton like process with UV light: Photo-Fenton like process.

3   METHODS

In this section the laboratory equipment and methods are describe. The first part focuses on the WW treatment, while the second part is related to the analysis of the WW treatment, namely the HEM content and the mass of dried sludge formed within FF process during WW treatment.

3.1   Design of experiments

An experiment is a systematic technique performed under controlled conditions to find an undiscovered effect, test or establish a hypothesis, or demonstrate a recognized effect. We know between which intervals the concentrations of the reactants move and the design of experiments will tell us the recipe we should follow according to a logic to validate our hypothesis. For experimental design it is normally used a program. The inputs in the program will be the factors that are controllable and the output that will be obtained are the responses analyzed. This design of experiments help to minimize the design costs, product material and labor complexity (MoreSteam, 2021). It represents a plan for carrying out the experiments in order to test the synergistic effects of various different parameters with acceptable statistical accuracy.

3.2   Wastewater treatment

The laboratory inventory needed for this part was:

5 jars of 1L	Pipettes of 5 and 10 mL	4-gang stirrer
Separatory funnels	Flasks of 250, 100, 50 mL	pH-meter
Reactor	UV lamp	Cooling system
Jar of 2L	Magnetic stirrer	Syringes
Oven	Laboratory balance	Desiccator

3.2.1   First FF

When the wastewater for experiments (WWE) is ready, we add the designed volume of $Fe_2(SO_4)_3$. This concentration of $Fe_2(SO_4)_3$ (in mL/L) has been established by design of experiment and is multiplied by the volume of WWE to obtain the volume (in mL) that is needed to add of this reactant to every jar and 1 mL/L of polyelectrolyte is added to all jars. The jars are placed in the 4-gang stirrer and are left during 10 minutes to 100-140 rpm (Figure 3-1).

Figure 3-1: Jars in the 4-gang stirrer.

After this mixing, the water is poured in separatory funnels and left there for one day for the FF to take place.

Sludge of this first FF is collected and dried in small jars in an oven at a temperature of 105 ⁰C for 4-5 days.

3.2.2 Advanced Oxidation Process (AOP)

For the AOP, 1.8 L of water from first FF are collected. The water is poured into the reactor (Fig. 3-2) and stirred continuously with a stirring magnet. The corresponding volume of hydrogen peroxide (H_2O_2) is added then. The next step is to set the required pH of the mixture, which should be between 2 and 2.7. If it is higher, then, some drops of HCl (concentration: ≥37%) are added till the pH reaches that interval. The UV lamp is inside the reactor, while the WW sample is stirred around it. When the WW sample is prepared, the UV lamp is switched on. To maintain the AOP process within the required temperature range the UV lamp is cooled via cooling water cycle. The AOP process lasted for 30 mins and it is followed by second FF.

The UV lamp is introduced through this opening at the top.
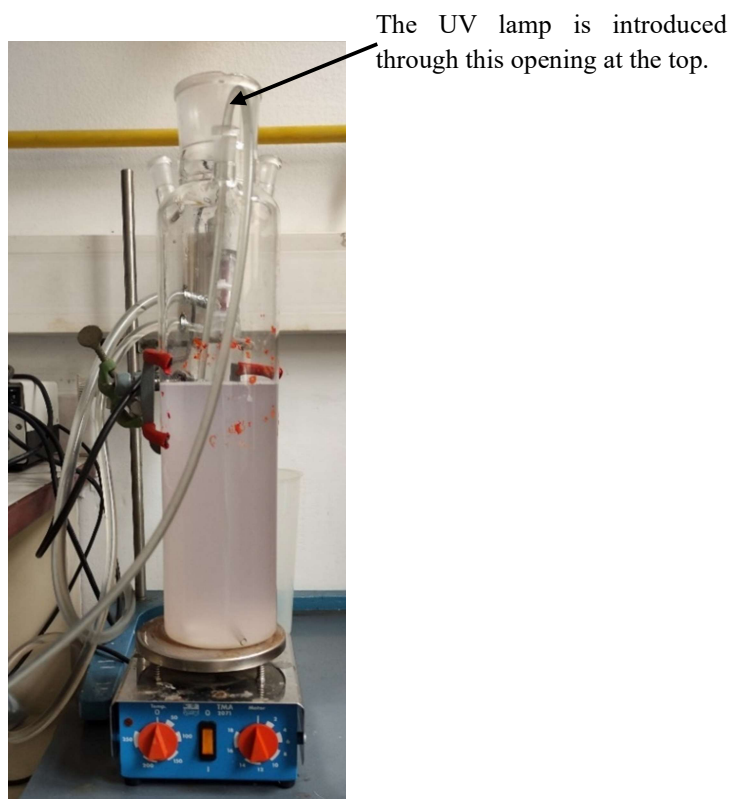
Figure 3-2: Reactor of the AOP.

3.2.3 Second FF

The second FF takes place in alkaline environment. Both the volume and pH of WW sample was measured. To perform FF process, the required volume of $Fe_2(SO_4)_3$ is added. The concentration of $Fe_2(SO_4)_3$ in this second FF to be added was always 1.5 mL/L. After addition of $Fe_2(SO_4)_3$ a lime milk was added to reach the pH above 8, preferably around 8.5. Finally, 1 mL/L of polyelectrolyte was added to finish the FF process. During chemical additions, the mixture is stirred at about 400 rpm. When stirring for 10 minutes, the flocs are let to settle down.

The sludge formed in this FF is also collected and dried in the oven. This sludge dries in approximately 2-3 days until constant weight.

3.3 HEM measurement

The list of the laboratory inventory needed is the following:

Flat-bottomed spherical flasks	Oven	Desiccator
Volumetric flask of 500 mL	Laboratory balance	Flask of 50 and 100 mL
Separatory funnel	Filter funnel	Filter papers
Glass rod	Rotavapor	

After each WW treatment stage, 500 mL of water are collected for HEM measurement. Also, 500 mL of the initial WWE are collected to enable the HEM removal calculation. As the

samples were not measured immediately after treatment their pH was lowered below 2 by addition of H_2SO_4 (4M). The HEM measurement consisted of extraction, solvent distillation and calculations phases.

3.3.1 Extraction

The flat-bottomed flask is first dried in an oven at 105-115 ºC during at least 2 hours and after cooled down to room temperature in desiccator. The weight of the flask is measured at room temperature.

A 500 mL water sample in volumetric flask is used for measured. This is half of the amount of sample required in the method (500 mL instead of 1 L) due to the shortage of it. Because of that, we also use only half of the required amount of n-hexane. First, half of the sample is poured in separatory funnel. To the other half of sample left in flask, 15 mL an n-hexane are added with at least 85 % purity. The volumetric flask is then shaken to allow the extraction liquid-liquid for 1-2 minutes. The sample from flask then poured into the separatory funnel. The volumetric flask is rinsed with small portions (5 mL) of n-hexane and poured into the funnel. For the extraction to take place, the separatory funnel is shaken for 3 minutes. The sample in separatory funnel is left for 10 minutes to allow the organic phase and the aqueous phase to separate. The aqueous phase is the lower one.

After 10 mins the organic phase is collected in a small flask (100 mL). The separatory funnel is rinsed with small portions of n-hexane (5-10 mL) until there are no rests of lipids in the walls of the funnel. In the Figure 3-3, we can observe the two phases: the aqueous is more amount and has a purple color in our case, while the organic one is only a thin whitish layer.
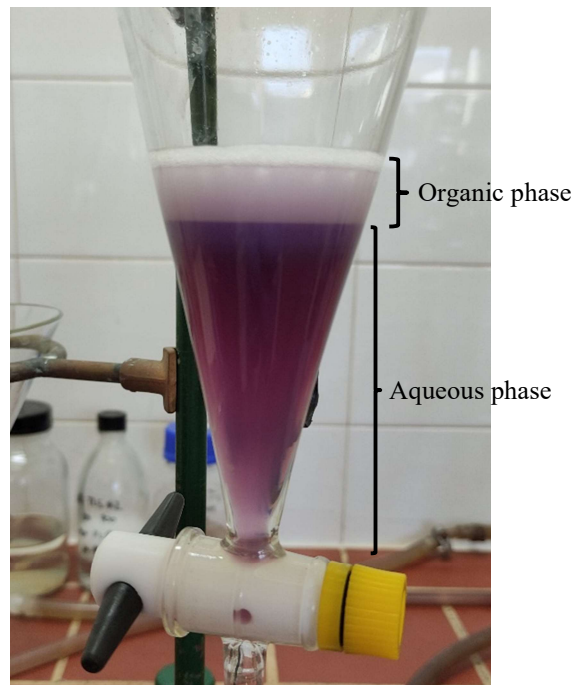
Figure 3-3: HEM measurement extraction phase.

A filter paper (MN 615 cellulose filter, ff) is placed in a filter funnel and filled to about half full of Na_2SO_4 anhydrous. The organic phase is then drained through the Na_2SO_4 into the pre-weighed flask. If needed additional filter papers with Na_2SO_4 anhydrous was used. When the Na_2SO_4 anhydrous cannot absorb more water, it hardens and that indicates that another filter

with Na₂SO₄ anhydrous should be used. The small flask is rinsed with n-hexane and pour it into the pre-weighed spherical flask.

3.3.2 Solvent distillation

This part of the measure starts by connecting the spherical flask to the rotavapor (Fig. 3-4). The lower half of the flask is immersed in the water bath with a temperature of 50 ⁰C. The speed of the rotavapor is 40 rpm.

Figure 3-4: Rotavapor.

The pressure in rotavapor is first set to 900 mbar, after to 700 mbar, nest to 500 mbar and finally to 350 mbar. At each pressure level the sample was left for 10 minutes. After distillation on rotavapor the sample in flask is dried in oven at 70 ⁰C for 30 minutes. After, it is placed in a desiccator with silica gel until it cools to room temperature. Finally, the flask with the dried residue is weighed.

3.3.3 Calculations

The mass of the dried HEM m^{HEM} is calculated as the mass of the flask with the dried HEM $m^{\text{HEM+FLASK}}$ minus the tare mass of the flask m^{FLASK} (Eq. 1):

$$m^{\text{HEM}} = m^{\text{HEM+FLASK}} - m^{\text{FLASK}} \tag{1}$$

The mass concentration of γ^{HEM} HEM in the sample is calculated as the mass of dried HEM divided by the volume of wastewater sample (Eq. 2).

$$\gamma^{\text{HEM}} = \frac{m^{\text{HEM}}}{V^{\text{WW\_sample}}} \tag{2}$$

(U.S. Environmental Protection Agency, 2010)

3.4   Mass of dried sludge

To enable reliable comparison of sludge formation at various samples the mass of dried sludge has been measured. The sludge formed during the FF was dried in the oven and then weighted. That way, we determined the mass of sludge on the whole sample, so we can calculate the sludge formation in g/L.

4   EXPERIMENTAL WORK

4.1   Design of experiment

Firstly, the experiment design was done by using the program called Design-Expert. (StatEase, 2021). It is a program for analyzing and planning laboratory experiments. It also offers optimization, comparative tests, screening, robust parameters design, characterization. It has graphical tools help to identify the impact of each factor. The statistical significance of the factors is established with analysis of variance (ANOVA). After opening the program and starting a new experiment, we choose *Response Surface* from the list of designs. We start by introducing how many factors we are going to study and vary. In the experiment, two factors will be varied i) concentration of H_2O_2 in L/m^3 (or mL/L) and, ii) concentration of $Fe_2(SO_4)_3$ in L/m^3 (or mL/L). We also need to introduce the interval between which the concentrations are going to move. In the Figure 4-1 we can see all these data as they would be entered in the program itself. We obtain 13 runs or experiments.

Central Composite Design

Each numeric factor is set to 5 levels: plus and minus alpha (axial points), plus and minus 1 (factorial points) and the center point. If categoric factors are added, the central composite design will be duplicated for every combination of the categorical factor levels.

Numeric factors: 2   (2 to 50)   ● Horizontal   ● Enter factor ranges in terms of ±1 levels
Categoric factors: 0   (0 to 10)   ○ Vertical   ○ Enter factor ranges in terms of alphas

	Name	Units	Low	High	-alpha	+alpha
A [Numeric]	Fe2(SO4)3	L/m3	1	2	0.792893	2.20711
B [Numeric]	H2O2	L/m3	5.5	10	4.56802	10.932

Type: Full ∨   Blocks: 1 ∨

Points
Non-center points: 8
Center points: 5

alpha =   1.41421   Options...   13   Runs

Figure 4-1: Factors for experimental study.

The response was the HEM content in units of concentration (mg/L). After pressing the button of *Finish*, we obtained the following design of experiment having the information about the required amount of chemicals to be added to each run (Figure 4-2).

Std	Run	Factor 1 A:Fe2(SO4)3 L/m3	Factor 2 B:H2O2 L/m3
1	2	1	5.5
2	11	2	5.5
3	3	1	10
4	7	2	10
5	6	0.792893	7.75
6	9	2.20711	7.75
7	1	1.5	4.56802
8	12	1.5	10.932
9	4	1.5	7.75
10	10	1.5	7.75
11	8	1.5	7.75
12	5	1.5	7.75
13	13	1.5	7.75

Figure 4-2: Layout of the design of the experiment.

Figure 4-3 presents the distribution of $Fe_2(SO_4)_3$ and H_2O_2 dosage (x-axis presents $Fe_2(SO_4)_3$, while y-axis H_2O_2).
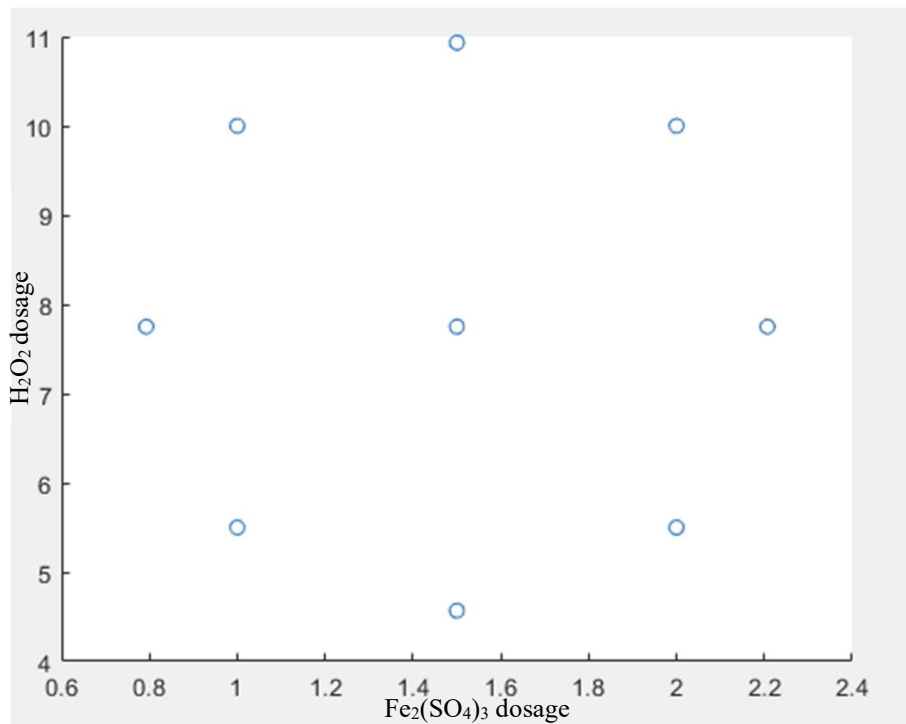

Figure 4-3: Distribution of addition of reactants.

There are five levels of dosage for each component, and they are combined to obtain the different runs. The central points should be measured more times, to reach statistical confidence in results of experiments, as the optimal results is expected to be within the selected region.

4.2 Wastewater treatment process

4.2.1 Preparation of the wastewater for experiments

Three different types of WWE were prepared. The industry provided for the whole work with two different surfactants before they run out. The results and removal would be different depending on which one has the WWE. That way we will have what we will call surfactant 1 (S1) and surfactant 2 (S2), to differentiate them. Two more WWE had to be prepared without surfactants (due to its lack).

In the table 4-1, a schematic summary can be seen for the final experiments done.

Table 4-1: Surfactants used in the WWE.

STD	SURFACTANT USED
1	S1
2	S1
3	S2
4	S1
5	S2
6	S2
7	0
8	S2
9	0

For all experiments, five jars of 1 L were prepared using this recipe:

For the experiments of STD 1, 2, 3, 4, 5, 6 and 8, the WWE was prepared by mixing the following quantities:

- 50 g of lipids (TLS)
- 150 g of surfactants
- 600 g of distilled water

That way, we obtain 800 g of final mixture that are approximately 0.8 L.

Besides, for the experiments of STD 7 and STD 9, the WWE was prepared without surfactants by mixing the following quantities:

- 50 g of lipids (TLS)
- 700 g of distilled water

So, we obtain 750 g of WWE that are approximately 0.75 L.

To sum up, depending on the surfactants, we have:

- WWE 1: with S1
- WWE 2: with S2

- WWE 3: without surfactants

4.2.2 First flocculation/flotation

The following table (Table 4-2) shows the final volume (mL) of $Fe_2(SO_4)_3$ added before the jars are placed in the gang-stirrer.

Table 4-2. Volume of $Fe_2(SO_4)_3$ added.

STD	mL of $Fe_2(SO_4)_3$
1	0.8
2	1.6
3	0.8
4	1.6
5	0.6
6	1.8
7	1.1
8	1.2
9	1.1

As we have seen, when the jars are removed from the gang-stirrer, the mixture is poured in the separatory funnels. Figure 4-4 shows how the WW looks like when it is has been recently poured.

Figure 4-4: WW poured in the separatory funnel before separation of flocs.

Figure 4-5 represents the separation of the aqueous layer (upper layer) from the sludge (lower layer). This is the case of S1. In the case of S2 (figure 4-6), we can see that the sludge is in the upper layer and the WW, in the lower layer. Also, the color of the water is different: with S1, the water becomes a dark purple color.
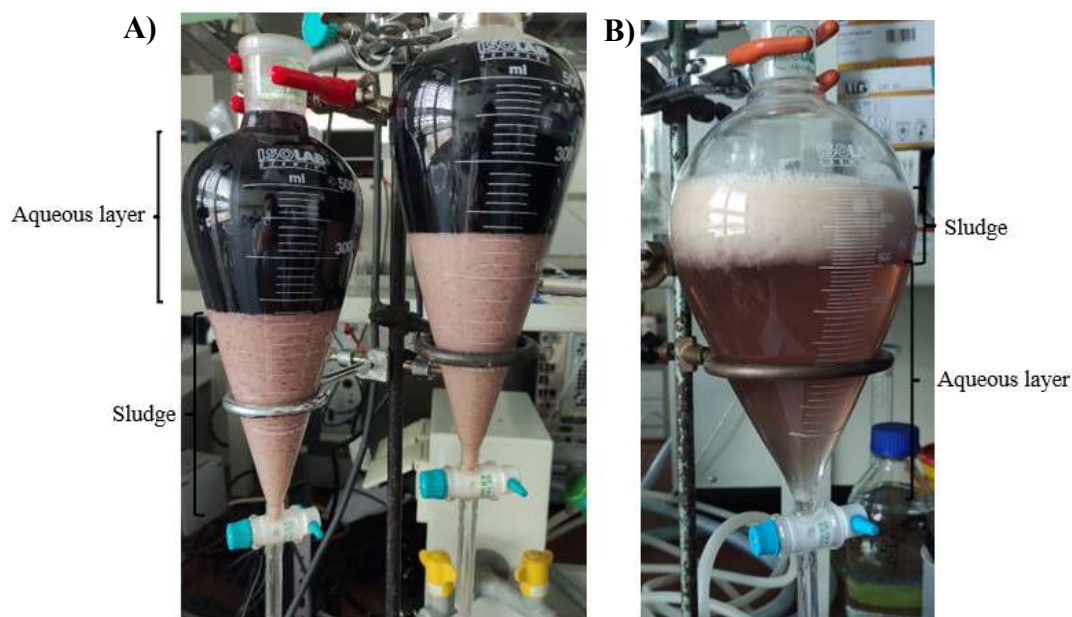
Figure 4-5: After first FF takes place A) S1, B) S2 and no surfactants.

4.2.3 Advanced Oxidation Process (AOP)

The following Table 4-3 shows the final volume (mL) of H_2O_2 added and the pH before the reaction in 1.8 L of sample from aqueous layer after first FF.

Table 4-3. Value of each variable for AOP.

STD	mL of H_2O_2 (35%)	pH
1	9.9	2.7
2	9.9	2.5
3	18.0	2.5
4	18.0	2.4
5	13.9	2.5
6	13.9	2.6
7	8.2	2.4
8	19.7	2.5
9	13.9	2.3

4.2.4 Second flocculation/flotation

The following Table 4-4 shows the final volume of water collected from the AOP, the final volume (mL) of $Fe_2(SO_4)_3$ added and the final pH after the addition of lime:

Table 4-4: Value of each variable for second FF.

STD	Volume of water (L)	mL of $Fe_2(SO_4)_3$	pH
1	1.30	2.0	8.82
2	1.30	2.0	11.53
3	1.30	2.0	10.02
4	1.20	1.8	8.64
5	1.10	1.7	10.15
6	1.10	1.7	9.31
7	1.30	2.0	8.66
8	1.40	2.1	9.91
9	1.25	1.9	9.61

After the second FF, as we have seen, the flocs are let to settle down. With S1, this does not occur till one day after. However, with S2, flocs settle down in half an hour (Figure 4-7).

Figure 4-6: Sludge of second FF.

4.3 HEM measurement

This work only covers the final degree of lipid removal and therefore only the HEM of the three WWE samples and the HEM of the samples obtained after the second FF (W2FF) will be measured. The influence of surfactants as well as their removal is not our concern in this work. Also, the level of removal after each stage is out of the scope of this work.

For the measurement of the samples after second FF, the method described in section 3.3 was followed. However, for HEM measurement of WWE, the WWE is diluted 1:10. We mix 50 mL of WWE with 450 mL of distilled water. Due to this, a recalculation of HEM will be necessary to be done. That means that equation 2 shall be multiplied by 10 due to the dilution to obtain the real concentration. To achieve higher accuracy, the flask was weighted two times and then, the average was done. In addition, V_s of equation 2 corresponds to the volume of the volumetric flask which is equal to 0.5 L.

5 RESULTS AND DISCUSSION

About the sludge formed in the first FF, we can analyze the following Table 5-1. It shows the total mass of sludge formed in the separatory funnels before putting it in the oven and the final dry mass after the complete drying in the oven.

Table 5-1: Sludge removed in the first FF (wet and dry).

STD	V^{init}(L)	m^{sludge} (g)	V^{sludge} (L)	m^{dry_mass} (g)
1	4.00	1928.27	1.92	29.61
2	4.00	769.10	0.70	17.96
3	4.00	1154.93	1.10	8.90
4	4.00	1348.19	1.38	26.09
5	4.00	1472.07	1.40	9.54
6	4.00	1188.35	1.10	8.44
7	3.75	1226.99	1.10	8.69
8	4.00	1214.96	1.15	9.00
9	3.75	1524.98	1.35	10.35

If we compare results, STD 1, 2 and 4 used S1 and we can observe that the dry mass formed is much higher (it goes from almost 18 g till almost 30 g) than the other STD that used S2 or no surfactant. With the ones that used S2 or no surfactant the dry mass is around 9 g. This confirms the hypothesis that the type of surfactant significantly affects not only the FF but the whole process.

In table 5-2 we have the results of the dry mass after the oven of the sludge of the second FF.

Table 5-2: Dry mass removed in the second FF.

STD	Dry mass (g)
1	0.39
2	1.55
3	1.52
4	0.62
5	0.92
6	2.61
7	5.55
8	2.94
9	3.27

As we expect, the dry mass in the second FF is much lower than the dry mass of the first FF. However, we cannot see differences related on the type of surfactant used. Only we can comment that without surfactants the dry mass values obtained are a little bit higher than the others (5.55 g and 3.27 g).

From the HEM measurement the following results of Tables 5-3 and 5-4 are obtained:

Table 5-3: HEM calculations for WWE.

WWE	Tare weight (g)	Final weight (g)	γ^{HEM} (mg)	HEM (mg/L)
1	134.7645	135.2087	444.25	8885.0
2	134.7612	134.9053	144.10	2882.0
3	170.9804	171.0709	90.55	1811.0

Table 5-4: HEM calculations for W2FF.

STD	Tare weight (g)	Final weight (g)	γ^{HEM} (mg)	HEM (mg/L)
1	170.9786	171.1435	164.85	329.7
2	134.7598	134.8461	86.30	172.6
3	99.4280	99.4315	3.50	7.0
4	100.8799	100.9793	99.35	198.7
5	99.4303	99.4311	0.75	1.5
6	100.8717	100.9051	33.35	66.7
7	100.8739	100.8745	0.65	1.3
8	100.8673	100.8733	6.00	12.0
9	178.1160	178.1294	13.35	26.7

The cosmetics industry has set the limit of emissions of HEM in 100 mg/L after the whole process. If we analyze the table 5-4, we observe that 3 out of 9 experiments does not fit this limit. But we can realize that these three experiments use WWE 1 with S1. Obviously, the sample with S1 had much higher content of HEM compared to other samples. Despite having very high HEM content the HEM concentrations were nearby the limits.

Also, literature *(Method 1664, Revision B: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry)* set the method detection limit of HEM in 1.4 mg/L. As we can see, the value of HEM at the end of experiment STD 7 is lower than this value. We can conclude that the experiment 7 has almost eliminated all the HEM but to calculate the final removal we will change this value to 1.4 instead of 1.3.

After determining of HEM concentration before and after treatment, the percentage of removal was calculated using the Equation 3.

$$\eta = \frac{HEM\ in\ WWE - HEM\ in\ W2FF}{HEM\ in\ WWE} \cdot 100 \tag{3}$$

That way, the removal is obtained (Table 5-5):

Table 5-5: Percentage of removal after the whole process.

STD	η
1	96.29
2	98.06
3	99.76
4	97.76
5	99.95
6	97.69
7	99.92
8	99.58
9	98.53

When Table 5-5 is analyzed, we can conclude that the percentage of removal is very high, it goes from 96.3% till more that 99.9%. In average, with this new first FF, the percentage of removal is 98.61%. By comparison of the dried sludge formation, it can be concluded that the vast majority of HEM is removed in first FF. That way, we can state that introducing this new FF the cleaning of WW is efficient. However, four experiments were still to be carried out so the results of optimization of concentrations of reactants could not be done and we cannot predict which exact concentration of $Fe_2(SO_4)_3$ and H_2O_2 are the optimal.

To sum up, main results are reflected in the following Table 5-6. Additionally, the sludge formation per initial sample volume before FF1 $\gamma^{sludge\text{-}init}$ was calculated. The initial volume, when surfactants were added was 4 L, while the volume of sample with no surfactant addition was 3.75 L. It is clearly visible, that samples with higher initial HEM content had higher mass of sludge formed between 4.07 g/L up to 6.25 g/L. Interestingly enough, the lower addition of $Fe_2(SO_4)_3$ resulted in higher sludge formation, however, the HEM removal rate was lower. The samples with lower HEM content, where S2 was used, resulted in lower sludge formation, which is expected as there is less pollutant to remove.

Table 5-6: Summary of results.

STD	Surfactant type	$V^{Fe2(SO4)3}$ (mL)	V^{H2O2} (35%)/mL	$m^{1.\,FF}$ (g)	$m^{2.\,FF}$ (g)	m^{total} (g)	$\gamma^{sludge\text{-}init}$ (g/L)	HEM initial (mg/L)	HEM final (mg/L)	η (%)
1	1	0.8	9.9	29.61	0.39	30	7.50	8885.0	329.7	96.29
2	1	1.6	9.9	17.96	1.55	19.51	4.88	8885.0	172.6	98.06
3	2	0.8	18.0	8.90	1.52	10.42	2.61	2882.0	7.0	99.76
4	1	1.6	18.0	26.09	0.62	26.71	6.68	8885.0	198.7	97.76
5	2	0.6	13.9	9.54	0.92	10.46	2.61	2882.0	1.5	99.95
6	2	1.8	13.9	8.44	2.61	11.05	2.76	2882.0	66.7	97.69
7	0	1.1	8.2	8.69	5.55	14.24	3.80	1811.0	1.3	99.92
8	2	1.2	19.7	9.00	2.94	11.94	2.98	2882.0	12.0	99.58
9	0	1.1	13.9	10.35	3.27	13.62	3,63	1811.0	26.7	98.53

The HEM removal rate was in all cases high. It could be seen that in this case lower addition of $Fe_2(SO_4)_3$ resulted in higher HEM removal and lower sludge formation. What it is interesting to note is that samples with no additional surfactants (7 and 9) had lower HEM content compared to samples with S2 content (3,5,6,8) but the sludge formation was higher. This clearly indicates that surfactant type and content have impact on HEM removal in the entire process.

6 CONCLUSIONS

The goal of this work was to analyze the efficiency of the HEM removal throughout the entire process of wastewater treatment consisting of flocculation/flotation process in acid environment; followed by advanced oxidization process carried out with H_2O_2 with UV lamp and flocculation/flotation process in alkaline environment. Each experiment was prepared with different concentrations of $Fe_2(SO_4)_3$ and H_2O_2 with addition of different samples with high surfactant content or samples with only high HEM content. As can be concluded, all experiments that were carried out had the HEM removal higher than 96.29 % representing a very successful HEM removal.

The limit of 100 mg/L HEM after treatment was achieved in 6 cases, only in samples with enormously high initial HEM content was not below, however even in the worst case the HEM content decreased from 8885 mg/L to 329.7 mg/L. In cases where the HEM content was in line with the usual content of HEM in WW all the samples were well below the set limit.

Another important conclusion of this work is the impact of the type and content of surfactants being present in the samples. As the removal rate and sludge formation was lower in case with added surfactant it can be interpreted that surfactants or at least this one type of surfactant can have a negative impact on the HEN removal. This is in accordance with the theory of flocculation/flotation and the surfactant composition. In these cases, one should be aware of additional charges in the surface of surfactants when designing the FF process.

In the future an economic analysis would have been useful to be done t if the introduction of this new stage is profitable.

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8   CURRICULUM VITAE

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DRIVING LICENCE

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VOLUNTEERING

11/2018 – CURRENT

Volunteering in Grupo Scout Impeesa 379

Valladolid
Non-formal education with children between 6 and 20 years old. Activities in nature and helping others. The basic motto is to leave the world better than you found it.

LANGUAGE SKILLS

MOTHER TONGUE(S): Spanish

OTHER LANGUAGE(S):

English

Listening	Reading	Spoken production	Spoken interaction	Writing
B2	B2	B2	B2	B2

French

Listening	Reading	Spoken production	Spoken interaction	Writing
A2	A2	A2	A2	A2

EDUCATION AND TRAINING

09/2017 – CURRENT – Pº del Cauce, 59, Valladolid, Spain

Chemical engineering
Universidad de Valladolid

https://www.eii.uva.es/

22/02/2021 – 05/07/2021 – Smetanova ulica, 17, Maribor, Slovenia

Chemical engineering (ERASMUS+ programme)
Univerza v Mariboru

https://www.fkkt.um.si/

09/2012 – 06/2017 – Av. del Real Valladolid, 2, Valladolid, Spain

Professional Dancer, specialising in Spanish Dance
Professional Dance School of Castilla Y León

https://fuescyl.com/epdcyl

PROJECTS

10/2020 – CURRENT

Ballet Español de la UVa

https://danza.uva.es/ballet-uva/
This project begins when there is a desire to continue dancing despite having finished dance studies. University studies do not allow me to be 100% dedicated to dancing in a professional company. This is the solution to be able to continue enjoying dancing.

WORK EXPERIENCE

13/07/2020 – 31/07/2020 – Valladolid, Spain

Leisure and Free Time Monitor

La Canica Verde

Urban camps with children from 7 to 15 years old. My task consisted in organising adventure activities such as climbing, orienteering, hiking...

19/07/2021 – 23/07/2021 – Valladolid, Spain

Leisure and Free Time Monitor

Pebetero

Urban camps with children between 8 and 13 years old. My task consisted of organising activities and games related to different sports.

DIGITAL SKILLS

Programme knowledge

Design-Expert software /   - good command of Microsoft Office tools (Word, Excel, Power Point and Outlook).

Priloga 6 – IZJAVA O AVTORSTVU IN ISTOVETNOSTI TISKANE IN ELEKTRONSKE OBLIKE
ZAKLJUČNEGA DELA

UNIVERZA V MARIBORU
Fakulteta za kemijo in kemijsko tehnologijo
(ime članice UM)

IZJAVA O AVTORSTVU IN ISTOVETNOSTI TISKANE IN ELEKTRONSKE OBLIKE ZAKLJUČNEGA DELA

Ime in priimek študent-a/-ke: Julia Olmedo Gomez
Študijski program: Izmenjava UN
Naslov zaključnega dela: Analysis of flocculation/flotation as wastewater pretreatment

Mentor: Andreja Nemet
Somentor:

Podpisan-i/-a študent/-ka Julia Olmedo Gomez

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