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Application of circular economy principles to a paper manufacturing process

Aplicación de los principios de economía circular a un proceso de fabricación de papel

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Tras realizar este trabajo solo puedo estar agradecida a todas aquellas personas que han hecho posible que lo lleve a cabo.

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Gracias a mis padres, Mabel y Alvaro, que sois los pilares de esta pequeña pero gran familia. Gracias a mi hermana, Belen, que des de la lejanía me seguías alentando. Gracias a mi pareja, Marcos, que en estos meses complicados has sabido apoyarme des de todos los aspectos.

REPORT

CONTENT

1. SUMMARY

This work is based on the importance of water consumption by paper industries. It has been known by media and physical results which can be observed that it is present a water-stressed in our country. Physically it can be seen that some river levels have reduced their capacity over the last years and *MITECO (2022)* provides information to quantify the capacity of the global Spanish water reserve which is about an 39,2%. Therefore, it has been studied the water consumption and wastewater that is removed from a wrapping manufacturing process, presented as a case study.

Hence, the objective of this work is to apply novel technologies to a wrapping paper manufacturing process in order to reduce the water consumption by the implementation of circular economy principals. This objective will be accomplished by four milestones. First a paper manufacturing process will be defined and represented, secondly water consumers, quantities and qualities will be identified, thirdly wastewater streams, quantities and qualities will be identified and finally it will be presented a wastewater treatment and regeneration operations which the goal of reuse this kind of water in the paper process.

The selected wrapping manufacturing process is based on three processes, which are: recycle pulp, wood pulp and papermaking processes. The raw material to produce paper is pulp which will be conform by a 70% or recycled pulp, being the rest from wood. Water is required to different points of the case study presented as demineralized water with high quality, the amount is approximately 12,2m^3 of water per paper tonne produced. The principal characteristic of demineralized water is the lower presence of minerals and salts dissolved thus, TDS and TSS should have reduced levels *(Vogelzang, 2007 % Real Decreto 1960/2700)* in order to have a conductivity less than 1µs/cm *(The Distillate Water Company),* and the principal bacteria *escherichia coli* should not be present (*Real Decreto 1960/2700*. While wastewater represents 10,7m^3 per paper tonne being collected from the papermaking process. This kind of water has high amount of organic matter, being the main source wood fibres and/or lignin derivates and hemicelluloses, which is measured by COD and BOD while it has lower impurities of TDS and TSS *(Dagar, 2022).*

Therefore, wastewater is the major concern in this case study in order to have a circular process. Thus, it has been suggested a process to obtain demineralized water from wastewater for a recirculation system. To reach the mentioned process demineralized water treatment will be studied having as input the raw water, supplied by a river.

Wastewater treatment follows the three phases of a typical WWTP, it will be treated by a clarifier, then by a novel technology, the MBBR system followed by the MF and finally a chlorination. While the demineralized treatment has more operations starting from the chlorination, sand filtration, MF and RO, membrane degasification and finally the mixed bed resin. The RO is a system that can remove a 98% of major contaminants *(Advanced Water Filters)* mentioned while the MBBR system can remove with a high efficiency the biological matter *(Veolia, 2022)*. The MF system is presented in both treatments allowing to optimize the whole water process. This is possible because it has been found that once the treated water leaves the MBBR system will have lower levels of TSS, COD and BOD, being suitable for the MF system of the demineralized treatment. Furthermore, the residual chlorine will remove the biological matter from the wastewater treatment. By the other side, the wastewater produced in the mixed bed resin in the demineralized treatment is introduced into the MBBR system of the wastewater treatment as the TDS and TSS is assumed to be lower at that point, allowing to avoid the clarifier of the wastewater treatment.

Thus, as the main process of paper is based on a recycled one, the wastewater volume will be less than a conventional process. Moreover, the exact number of the reused water has not been possible to know as the information of suppliers and industries is highly confidential. Because of that this work is based on a conceptual manner. Furthermore, the suggested water process can be applied to any paper industry because the main processes are always present, with some variations, and also it can be applied to other non-paper industries that consumes high volumes of water.

2. IDENTIFICATION AND REFLECTION ON THE SUSTAINABLE DEVELOPMENT GOALS

As it has been explained in the summary, the scarcity of our country is really elevated, which contributes to the global warming, which affect to all living beings. Thus, the main objective of this work is to asses new strategies and novel technologies to consume less water in a paper manufacturing process. Hence, this will be done by a case study in order to present a sustainable process, which will be based on a recirculation of the wastewater removed from the paper process. Having this objective defined, it can be identified the main sustainable development goals *(ONU)*, being the Goal 6 the principal one.

- *Goal 6 - Ensure access to water and sanitation for all:* one of the principal targets of this goal is to improve water quality by reducing pollution, eliminating dumping and minimizing release of hazardous chemicals and materials, halving the proportion of untreated wastewater and substantially increasing recycling and safe reuse globally.
- *Goal 9 - Build resilient infrastructure, promote sustainable industrialization and foster innovation*: the target that is related to this work by the state of art is the one that enhance scientific research, upgrade the technological capabilities of industrial sectors in all countries, in particular developing countries.
- *Goal 12 - Ensure sustainable consumption and production patterns*: some included targets are the achievement of sustainable management and the efficient use of natural resources and, reduce waste generation through prevention, reduction, recycling and reuse. If people are aware about these targets, they would buy recycle paper products rather than other materials and at the same time are making thrive the claiming industry.
- *Goal 14 - Conserve and sustainably use the oceans, seas and marine resources*: as this work is focus on paper process it means that the final product will be biodegradable, thus, if it ends into water resources it will not contaminate.

These mentioned goals impact into three broad areas. The goal number 6 is focused on people, the number 9 on prosperity and the numbers 12 and 14 on the planet. Because of that, if this work evolves in the future, it could reduce much of the global water shortage because the paper industry is one of the largest consumers *(Oliveira, 2019)* and therefore reduce global warming which really encompasses all areas.

3. INTRODUCTION

Water consumption is nowadays a significant concern regarding industries, especially for pulp and paper industries. This kind of industry is the third one that most consume water in the European Union *(Oliveira, 2019)*, consuming a 75% of the water provided by rivers *(ASPAPEL, 2000-2009)*. According to an article from *We Are Water Foundation (2022)* Spain is one of the most water-stressed industrialized countries in the world. The country faces the challenge of ensuring long-term water security.

According to *ASPAPEL* foundation in *2021* was found that the consumption of water by paper industries was about 110 million m^3 in our country, being consumed 8,7m^3 by tonne of paper. Another author, *Badar (2011)*, indicates that conventional paper industries consume approximately between 273 and 455m^3/t. While *Boguniewicz-Zabłocka (2019)* indicates that wrapping paper industries, that used recycled paper as raw material, consumes approximately between 6 and 32m^3/t. *ASPAPEL* do not gives information about what kind of paper industry is studied, is a generic data information, but it allows to make the assumption that paper industries are trying to consume less water than conventional paper industries by some circular alternatives and maybe by recycle raw materials. As this last detail, there is a lot of bibliographic description about technologies applied to obtain paper, but the scarcity of new ones is elevated. For this reason, the state of art will be the identification of novel technologies from web by specialized suppliers, books and seminars information.

Thus, the objective of this work is to apply novel technologies to a wrapping paper manufacturing process in order to reduce the water consumption by the implementation of circular economy principals. This objective will be accomplished by four milestones. First a paper manufacturing process will be defined and represented, secondly water consumers, quantities and qualities will be identified, thirdly wastewater streams, quantities and qualities will be identified and finally it will be presented a wastewater treatment and regeneration operations to reuse this kind of water in the paper process.

In general, all pulp and paper industries carry out the same operations but with some differences between them. For this reason, once the desired objective has been achieved, this work can be applied to any industry within the mentioned sector. Maybe the circularity cannot be applied to non-paper industries but, the described water treatment to regenerated it can be used in their installations. It might be of great interest to apply the technologies described in this work to chemical industries because they are the second largest consumers of water *(Oliveira, 2019).*

4. OBJECTIVE AND SCOPE

Nowadays in paper making industry there are a wide range of paper types, ranging from recycled paper, newsprint paper, tissue, packaging paper… Particularly, the base of this project will be the manufacturing of packaging paper, more specifically, the production of wrapping paper.

Paper industries consumes an intensively amount of water from natural resources, being a huge concern because of the scarcity of water due to climate change. Thus, this is the driving factor for the research of new process strategies, which reduce water consumption. Furthermore, wastewater treatment plants used conventional biological methods *(Roy, 2021)* but, this work is intended to apply novel technologies to obtain regenerated wastewater.

Therefore, the objective of this project is to assess new process strategies for the wrapping paper production, which allow to reduce water consumption, within the circular economy principles. This goal has been structured in several milestones:

1. Define a representative type of wrapping paper production process.
2. Identify water consumers in the process, as well as the necessary quantities and qualities.
3. Identify the wastewater streams generated in the process and their quality.
4. Assess the wastewater treatment and regeneration operations required to reuse the wastewater in the different consumption points.

As a methodology, based on bibliographical research, a widespread wrapping paper production process will be defined and specified taking to water consumption and wastewater generation. Through a case-study, the above-described circular economy principles related to water reuse, a novel process scheme will be suggested for the immediate future of the paper industry.

State-of the-art technologies for such purpose have been identified from web by specialized suppliers, books and seminars information.

5. BACKGROUND

5.1. LEGISLATION AND CIRCULAR ECONOMY

In order to achieve the main objective and to ensure that it is accomplished by the legislation and the principles of the circular economy in this country, the description and characteristics of them are described below.

5.1.1. <u>Legislation</u>

The principal regulation on which this work is based, due to the importance of water consumption in paper industries, is the *Real Decreto 1620/2007, of December 7,* which establishes the legal regime for the reuse of treated water. The purpose of this Real Decreto is to establish the legal regime for the reuse of treated water, in accordance with Article 109.1 of the revised text of the Water Law, approved by the Real Decreto Legislativo 1/2001, of July 20.

This regulation states the permitted and non-permitted places and equipment where the regenerated water can be used, among other information. It also provides information on the quality that reclaimed water must have in order to be reused.

Some of the groups that are mentioned within the non-permitted uses and perhaps the most important ones to be emphasized are the human consumption and the hospital facilities, with some restrictions. While the permitted groups where the regenerated water can be are the urban, recreational, agricultural, industrial and environmental. Industrially the regenerated water can be used for processes and cleaning, among other uses. Two frequent uses are mentioned with regards to this last sector, which are cooling towers and evaporative condensers, indicating that the quality of the regenerated water must be of class A, which can be determined as the most demanding or highest quality compared to other industrial uses. Thus, this water must not present the bacteria *escherichia coli* and should have a maximum level of solids in suspension of 5mg/L.

5.1.2. <u>Circular economy</u>

5.1.2.1 Description

According to the *European Parliament (2022)* the traditional linear economic model is based on using a product and then throwing it away. Due to that the materials and energy used in processes with this model are usually unexpensive and very accessible.

Otherwise, the circular economy is a model of production and consumption that involves sharing, renting, reusing, repairing, renewing and recycling materials and products as many times as possible to create added value, in order to extend the life cycle of products. It means reducing waste to minimum and in consequence a minimum global impact. Thus, products that reach the end of its life are kept withing the economy whenever possible. These can be productively used many times, creating additional value.

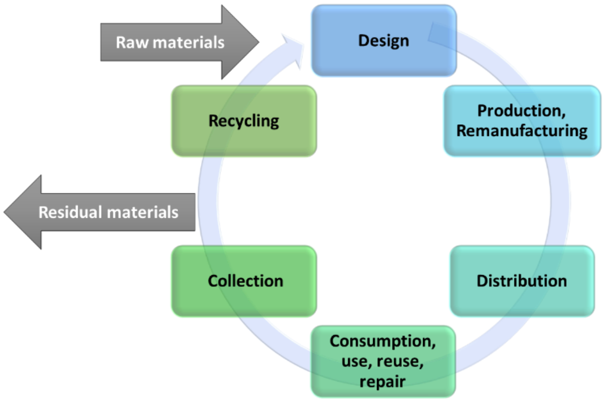

Figure 1: Circular economy diagram

(Figure adapted from European Parliament (2022), ref. 1)

One of the reasons to be focus on the circular economy is based on the increased demand for raw materials and the scarcity of some resources. Several crucial raw materials are finite, and as the population grows, demand also increases. Also, there is one more factor that is crucial for our environment which is the impact created using raw materials. The extraction and use of raw materials have significant consequences to the climate, it increases energy consumption and carbon dioxide emissions, while smarter use of raw materials can reduce polluting emissions.

5.1.2.2 Strategy and Plans

In this country it is followed the Spanish Circular Economy (EEEC) Strategy and Plans *(Ministerio para la Transición Ecológica y el Reto Demográfico).*

The EEEC Strategy, also known as Spain Circular 2030, lays the foundations for promoting a new production and consumption model in which the value of products, materials and resources are maintained in the economy for as long as possible, in which reduce the generation of waste to a minimum and take advantage of those that cannot be avoided to the greatest extent possible. The Strategy thus contributes to Spain's efforts to achieve a sustainable, decarbonized, resource-efficient and competitive economy.

The Strategy has a long-term vision, which will be achieved through successive three-year action plans to be developed, which will make it possible to incorporate the necessary adjustments to complete the transition in 2030. In this context, the Strategy establishes strategic guidelines as a decalogue and sets a series of quantitative objectives to be achieved by 2030, in the case of the water objective is to improve efficiency in water use by 10%.

The action Plans to close the circle of circular economy are five:

- *Line of action "Production"*: promote the design/redesign of processes and products to optimize the use of non-renewable natural resources in production, promoting the incorporation of secondary raw materials and recycled materials and minimizing the incorporation of harmful substances, with a view to obtain products that are more easily recyclable and repairable, redirecting the economy towards more sustainable and efficient ways.
- Line of action "Consumption": reduce the ecological footprint by modifying the guidelines towards more responsible consumption that avoids waste and non-renewable raw materials.
- *Line of action "Waste Management":* effectively apply the principle of waste hierarchy, substantially favouring prevention (reduction), preparation for reuse and recycling of waste.
- *Line of action "Secondary raw materials"*: guarantee the protection of the environment and human health by reducing the use of non-renewable natural resources and reincorporating the materials contained in waste as secondary raw materials into the production cycle.
- *Line of action "Reuse and purification of water":* promote efficient use of the water resource, which makes it possible to reconcile the protection of the quality and quantity of aquatic masses with its sustainable and innovative use.

5.2. PAPER INDUSTRY AND WATER CONCEPTS

There is a wide range of paper manufacturing industries, but this work will be focus on the manufacturing of the wrapping paper product. It has been selected this product because is a great replacement of plastic products whit huge applications. This paper product is made by the process known as Kraft or sulphate process; it manufactures Kraft paper which is the most used material in packaging *(Netramai, 2016)*. There are two main reasons why this kind of paper has been selected:

- *The most globally used*: Kraft technology is used in approximately an 80% of the entire paper production of United States and Europe. Globally manufacturing industries of this kind has been increasing, thus the Kraft paper is one of the most demanding papers. There is a rise in demand for Kraft paper markets in various end-use industries, such as food and beverages, building and construction, cosmetic and personal care automotive and consumer durables, is a key factor that is projected to drive the expansion of this market across the world.
- *Environmentally friendly*: It is usually made of pulp or recycled paper, which allows to apply the circular economy principals. Furthermore, factors such as rapid urbanization and the recyclability feature of the Kraft market are projected to contribute the growth of the global Kraft paper market *(Verified Market Research, 2021)*. Moreover, Kraft process can produce high-quality paper with less pollution than other paper manufacturing processes such as the sulphite process *(Zerbe, 2004)*.

Kraft process can manufacture different types of paper but, it has been selected a mixture between two types of identified Kraft papers, being these two:

- *Virgin Natural Kraft Paper*: this paper is the heavy lifter of the paper world. Its clean and durable fibre content and its low cost make it an ideal option for heavy-duty applications that require a high level of tear resistance. Also, it is perfect for printing as well, being perfect for packaging and protective layering, wrapping, pallet interleaving, carrier sheets, and dunnage *(Jones, 2020)*.
- *Natural Recycled Kraft Paper*: although not as strong and tear resistant as virgin natural Kraft, natural recycled Kraft paper is a more environmentally friendly option, and still carries enough strength to do an excellent job with dunnage and void fill applications, as liners for trays and boxes, interleaves, and bottom wrap for newspapers *(Jones, 2020)*.

As there is no detailed information on the natural recycled Kraft paper process, the generic recycled paper process has been selected instead. Thus, a combination between the virgin natural Kraft process and the recycled paper process will be studied in order to obtain a wrapping paper product, that will be supplied in the form of rolls. It has been chosen the wrapping paper against other types of final product such as storage bags, dunnage bag or paper tape because of the simplicity of the end at the train process. This kind of product will be just cut once the last stage of the manufacturing paper is completed.

In order to understand the case study that will be described in this work, it is necessary to know some basic concepts regarding the wood and the paper manufacturing process in general and based on the Kraft process.

5.2.1. Wood concepts

According to *McDonald (2001)* wood is a cellular lignocellulosic material consisting of cellulose embedded in matrix of hemicellulose and lignin. Wood may also contain significant quantities of extractives (resins and gums) in the form of low molecular weight extracellular compounds and inorganic component typically less than 0.5%. These three main polymer constituents of wood are described below.

- Cellulose: is one of the most abundant organic compounds on earth. It is a linear homopolysaccharide. In general, the cellulose content is approximately 42%. In the native state, adjacent parallel cellulose chains are aggregated together, by inter and intramolecular hydrogen bonds, to form microfibrils approximately of 4nm wide. These microfibrils contain both highly ordered (crystalline) and less ordered (amorphous) regions. The microfibrils are further aggregated with lignin and hemicelluloses to form macrofibrils. Both types of fibrils give strength to the wood.
- Lignin: is a three-dimensional polymer, normally amorphous, built up of phenylpropane units that is laid down within the cell wall. The incorporation of lignin into the cellulose microfibril structures within the cell wall greatly enhances the mechanical strength properties of wood, over pure cellulose, because it acts as a binder or matrix. In general wood can contain between 20 and 35% of lignin.
- Hemicellulose: is a partly crystalline polymer, consisting of various heteropolysaccharides, which are deposited in the cell wall at a level of approximately 30% and are closely associated with cellulose and lignin. The microfibrils of cellulose are recovered by hemicellulose and then by lignin, that is why this component conforms a matrix with the lignin.

5.2.2. Paper manufacturing process

The main processes through which the raw material must pass in every paper manufacturing process are the pulping and the papermaking ones. These two subprocesses will be described below in order to know a little bit about them before the presentation of the case study. In the first subprocess description will be define the two types of pulps that will be treated in this work, the Kraft pulping an the recycle pulping processes.

- Pulping: process where the raw material is introduced and, which could be wood or other lignocellulosic material, is broken down physically and/or chemically such that discrete fibres are liberated and can be dispersed in water and reformed into a web, producing the product called pulp.

  There are four broad categories of pulping processes: chemical, semi-chemical, chemi-mechanical and mechanical. Thus, chemical pulping methods rely on the effect of chemicals to separate fibres, whereas mechanical pulping methods rely completely on physical action (*Biermann, 1996).*

  The most dominant technique employed in wood pulping is the chemical one, being the Kraft process dominant, it is widely used with a production of 70-80% of the total pulp *(Pulp Paper Mill, 2014).* Kraft pulping involves the digesting of wood chips at elevated temperature and pressure in "white liquor". White liquor is a water solution of sodium sulphide and sodium hydroxide. The lignin that binds the cellulose of fresh fibres together in the wood is chemically dissolved by this white liquor *(Cheremisinoff et al, 2010).*

  While the recycle pulping used a mechanical process in order to separate the recycled fibres in this case. Mechanical pulping is a process used for separating fibres without the addition of any chemicals. However, lignin is retained in the pulp resulting in fibres of high lignin content and accordingly they have low strength and brightness *(Biermann, 1993).*

  Moreover, is important to highlight that when it is mentioned a conventional pulp process means any process that is not a recycled one.

- Papermaking: once the pulp has been obtained by the two different types of processes is mixed and then it enters into the papermaking machinery as raw material. The papermaking process is generic for all the different pulping processes. Here a series of operations take place in order to acquire the properties of the final paper product. The main action that takes place is the elimination of the water contained in the pulp, the wastewater.

5.2.3. Water concepts

It has been known by different medias that the world is confronting a water scarcity. Being Spain one of the most water-stressed industrialized countries in the world. It faces the challenge of ensuring long-term water security *(We Are Water Foundation, 2022).* By information published by *MITECO* in august of *2022* it has been possible to know that in that date the capacity of Spanish water reserve was of 39,2% of his total capacity. This capacity indicates the scarcity of water in that moment. In the following figure are presented some percentages of the capacity of the main Spanish rivers.

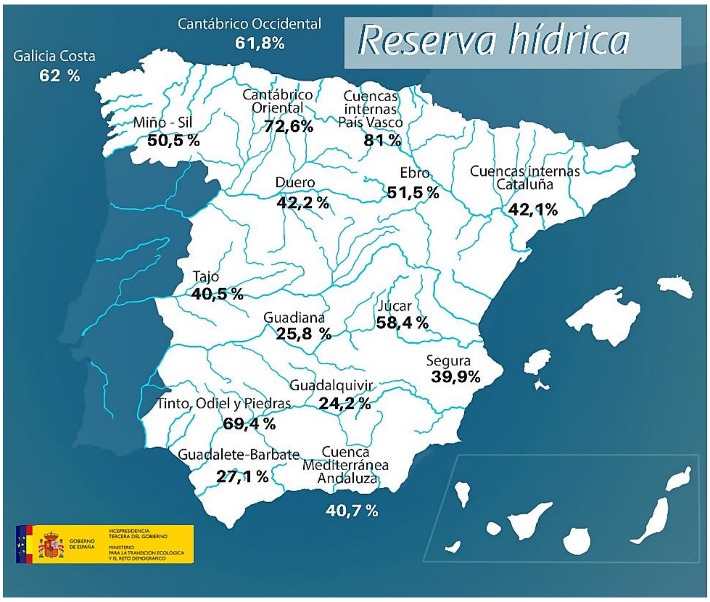

Figure 2: Spain water reserve

(Figure adapted from MITECO (2022), ref. 127)

Moreover, *MITECO (2023)* provides this year's information in outline form, this can be seen in the next figure extracted. It shows that in the last 10 years, the capacity of the Spanish water reserve has been decreasing, following the trend at the beginning of this year.

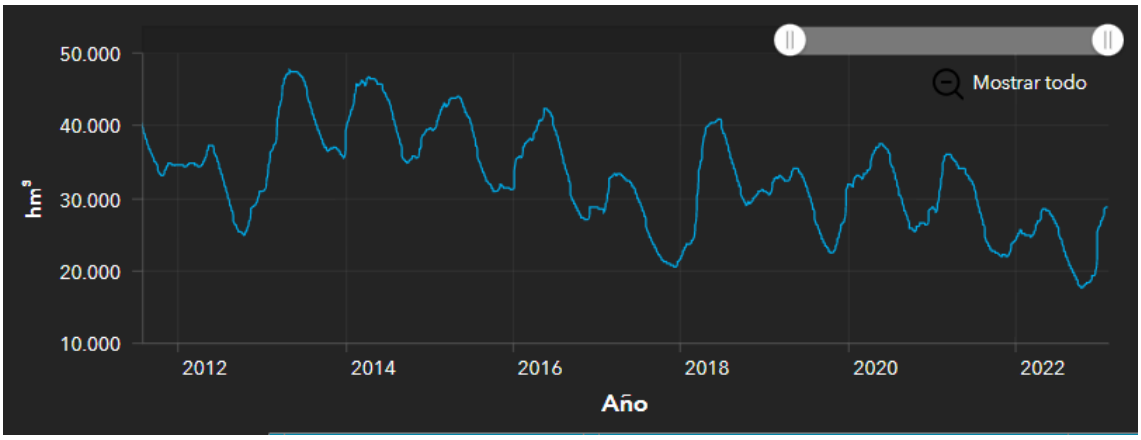

Figure 3: Evolution of Spain's water reserve capacity

(Figure adapted from MITECO (2023), ref. 128)

Therefore, this presented case study will propose an optimal process to ensure that water is not wasted. To understand it, some concepts must be known, which are:

- *Reuse of water*: application, prior to its return to the public water domain and maritime land for a new private use of water that, having been used by whoever derived it, has undergone the purification process or processes established in the corresponding discharge authorization and those necessary to achieve the required quality depending on the uses to which it is to be put.
- *Regenerated water*: treated wastewater that, where appropriate, has been subjected to an additional or complementary treatment process that allows its quality to be adapted to the use to which it is intended *(Real Decreto 1620, 2007).*
- *Demineralized water:* this kind of water refers to water completely free (or almost) of dissolved minerals as a result of a process. This process is usually a combination of different technologies *(Lenntech).*
- *Fresh water*: water that is not salty, for instance water found in lakes, streams, and rivers, but not the ocean. It can be also known as raw water or surface water, contains only minimal quantities of dissolved salts, thus distinguishing it from sea water or brackish water *(GreenFacts).* According to an article from *Osmosys* company *(2019)* the tap water supplied in Barcelona is from two rivers, Ter and El Llobregat. In this work, the water will be supplied from a river and this is why the characteristics that will be presented will refer to surface water.
- *Conventional and advanced wastewater treatments*: One of the claims of his work is to investigate and describe an advance secondary treatment.

Conventional wastewater treatments that are carried out by wastewater treatment plants (WWTP) are based on 3 phases, known as: primary, secondary and tertiary. Then after these phases can be an additional treatment but, those are the base of the operative plants.

As the name says, the primary phase is the first place where wastewater is received, it is based mainly on the removal of suspended solids but, it also removes organic matter. Then the secondary phase operates, there organic matter is the principal pollutant wanted to be removed and, finally there is the tertiary phase where remaining pollutants that had not been removed previously are collected by filters or chemicals and it is optional in some cases *(Dagar, 2022 & Tayal).*

The predominant pollutant in wastewater is the organic matter, that is why the secondary phase is the notable treatment in WWTP. Conventional treatments in this phase are focused on biological processes. The main treatments used in pulp and paper industry are the aerated ponds and activated sludge. The second treatment mentioned is more efficient regarding the pollutant removal. Moreover, the activated sludge can be well controlled and requires less surface, and the microorganisms used are adapted to the receiving wastewater *(Noel, 2017).*

While the *Institute for Sustainability* defines advanced wastewater treatment (AWT) as any process which reduces the level of impurities in a wastewater below that attainable through conventional secondary or biological treatment. Thus, a novel technology will be studied to be applied to the secondary treatment.

6. CASE STUDY

As it was described previously, there are different types of pulping, but as this work is looking for better actions and procedures to have a sustainable process, to produce paper the pulp used will be created by a mixture of two types of pulping, which means, by two types of pulps. These two types of pulp are made by two different raw materials, by fresh fibres from logs and by fibres from recycled paper. Thus, it has been decided that 1t of pulp will be represented by 0,7t provided by the recycle pulp and 0,3t by the fresh pulp.

That is why the process that will be defined has three main subprocesses, which are:

- <u>Recycle pulping</u>: manufacturing of recycled pulp.
- <u>Kraft pulping</u>: manufacturing of fresh pulp.

- Papermaking: manufacturing of the paper by the fresh and recycled pulp.

It has been found that recycle pulp can consume half the water consumption of the fresh pulp *(Ovapcen)* but, fibres are generally shorter, less flexible and less permeable than pulp produced by fresh fibres, consequently, they cannot be used in high quality paper *(Heederik)*. Moreover, a recycled fibre can be used five to seven times in production, not indefinitely. The fibre losses mean that fresh fibres will always be necessary to maintain the fibre balance *(The Biofore Company, 2011)*. An article from *Carranza (2017)* describes that to prepare the pulp paper industries use approximately a 70 – 75% of recycle paper and the rest, 30 – 25%, is conformed with fresh fibres and also additives and fillers. That is why both types of raw material will be used in this work to obtain the final pulp to produce paper. Thus, it has been decided that the final pulp will be composed of 70% recycled pulp and 30% fresh pulp.

Once both types of pulp are mixed a 100% of the water will have been consumed by the two pulping processes, knowing the tons of each type of pulp that will be produced, it is possible to know the amount of water consumed in each case. Water will be consumed by the recycle pulping representing a 70% and the rest by the Kraft pulping.

According to *Badar (2011)* in a conventional manufacturing paper process more than the 80% of the water consumed is removed from the paper production as wastewater and according to *Dagar (2022)* approximately the 85 - 90% of the consumed water is wasted. If the Kraft pulping is considered individually, it will consume a 100% of water and will remove a 90% of wastewater but, in this case, the consume water is a 30% and that is why the wastewater is a 27%.

While, following the article from *Boguniewicz-Zabłocka (2019)* the wrapping paper manufacturing process by recycle pulping removes an 87% of wastewater. As the previous information, this amount refers to a water consumption of 100%. Thus, in this case the consumed water is a 70% and the wastewater is a 61%.

Moreover, with the information given by *Badar* book *(2011)* has been possible to know that approximately a 364m^3 of water are consumed in a conventional manufacturing process by tonne of produced paper. Moving this information to this case study where it is consumed only a 30% of water by the conventional process, means that 11m^3 of water are consumed by tonne of paper produced. While the publication of *Boguniewicz-Zabłocka (2019)* indicates that recycle wrapping paper consumes approximately 19m^3 of water by tonne of paper produced. Thus, this means that in this case study where it is only consumed a 70% of water by the recycled manufacturing paper it will suppose approximately a consumption of 1m^3 of water by tonne of paper. Therefore, both amounts represent a global consumption of approximately 12m^3 by tonne of paper produced.

Knowing that the 88% of water is removed from the whole paper manufacturing process and the global consumption of water it has been determined the quantity of the global wastewater removed, 11m^3/t. Also, with the percentages of the wastewater produced by each pulping process it has been identified the quantities that represents, being 1m^3/t from the recycle pulping and 10m^3/t from the Kraft pulping.

All this information given is summarized in the following table.

		Consumed Water		Wastewater	
Process	**Pulp Quantity [%]**	[%]	[m^3/t]	[%]	[m^3/t]
Recycle pulping	70 [4]	70	1,3[38]	61 [38]	1,1
Kraft pulping	30 [4]	30	10,9[36,37]	27 [36,37]	9,6
TOTAL	100	100	12,2	88	10,7

Table 1: Global consumed and wasted water

(Table adapted from, ref. 36, 37 & 38)

Below is presented a general diagram of the manufacturing process with the main water streams.

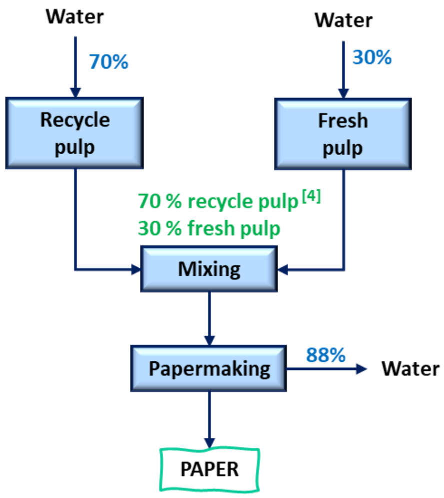

Figure 4: Simplified schematic diagram of the case study paper manufacturing process

It is important to remark that this case study is a basic manufacturing process, there is a wide range of processes involved but, they all follow the same model that will be described.

6.1. PAPER MANUFACTURING PROCESS

6.1.1. Recycle pulping

This pulping process has five differentiated sections which can be seen in the next diagram and are explain below of it.

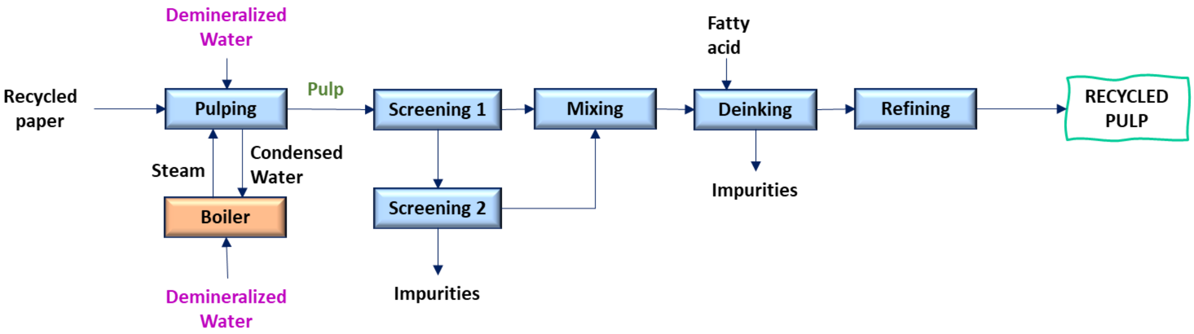

Figure 5: Schematic diagram of the recycled pulping process

(Diagram adapted from ref. 5,6 & 7)

- *Pulping*: also called secondary breaking or recycle pulp, where the recycle paper is introduced into a vessel called hydro-pulper with water and is agitated by rotating blades while is heated by a steam from a boiler *(Vogler et al., 1815)*. There the resultant pulp will contain a 99.5% of water *(Northwest Pulp & Paper Association)* and the rest will be the main material, the recycled paper. Once, the mixture is done, the pulp is collected.
- *Screening*: the pulp is forced through a pressure screen, which are design to remove contaminants, such as stickies and shives, which reduce the strength, smoothness and optical properties of the paper *(Olson)*. Also, removing that contaminants process equipment will be protected from potential damage.

  This equipment has one inlet where the pulp is introduced and two outlets, one for the accepted pulp and another for the rejected part. Once the pulp is introduced into the equipment is falls into the middle of a rotating basket where the centrifugal force pushes the pulp towards the walls removing contaminants through the basket holes and leaving the pulp clean. *(Valmet Forward)*.

  It will be used two screen systems. First the pulp will be introduced into the primary screen and when the rejected part falls it will go to a secondary screen to ensure that the fibres that fell down with contaminants are recovered (*Biermann 1996)*. Finally, it will be a mixing vessel, which will collect the pulp from both screeners.

- *Deinking*: this procedure can be done with a washing vessel but as the objective of this work is to reduce the wastewater, this method will not be contemplated as it requires a lot of water *(The Biofore Company, 2011)*. Instead, it will be done the flotation method.

  Recycled paper usually has printing ink and glue or adhesives, to ensure that the pulp does not contain any of the mentioned materials it is introduced into the flotation equipment. The pulp is fed into a large vessel called flotation cell, at the top of the cell compressed air will be injected while surfactant will be sprayed.

  Once air is introduced, air bubbles are created and float to the surface of the cell. These bubbles will contain the impurities that are wanted to be removed. Then, the sprayed surfactant creates a surfactant layer on the top of the cell. When an air bubble passes through this layer, the surfactant adsorbs onto the bubble surface. As a result, the bubble can be stabilized, and a foam layer can be generated. Thereby, the ink particle can be transferred to the foam layer as in a conventional flotation process leaving a treated pulp *(Deng, 1999)*. The most used surfactant is fatty acid, that is why this component will be introduced into the flotation vessel *(Venditti)*.

- *Refining*: the pulp obtained is beaten to make the recycled fibres swell, making them ideal for papermaking. If the pulp contains any large bundles of fibres, refining separates them into individual fibres *(Northwest Pulp & Paper Association)*, making them more flexible ready to form bonds in paper *(Norman, 2009)*. It means that this mechanical treatment and modification of fibres is important to produce high quality papers.

As the paper that is wanted to produce is brown, no bleaching treatment will be done after finishing the refining treatment.

6.1.2. Kraft pulping

At the same time, while the recycled pulp is being produced the recycle pulping is being done by the Kraft process. This process is carried out through the following steps.

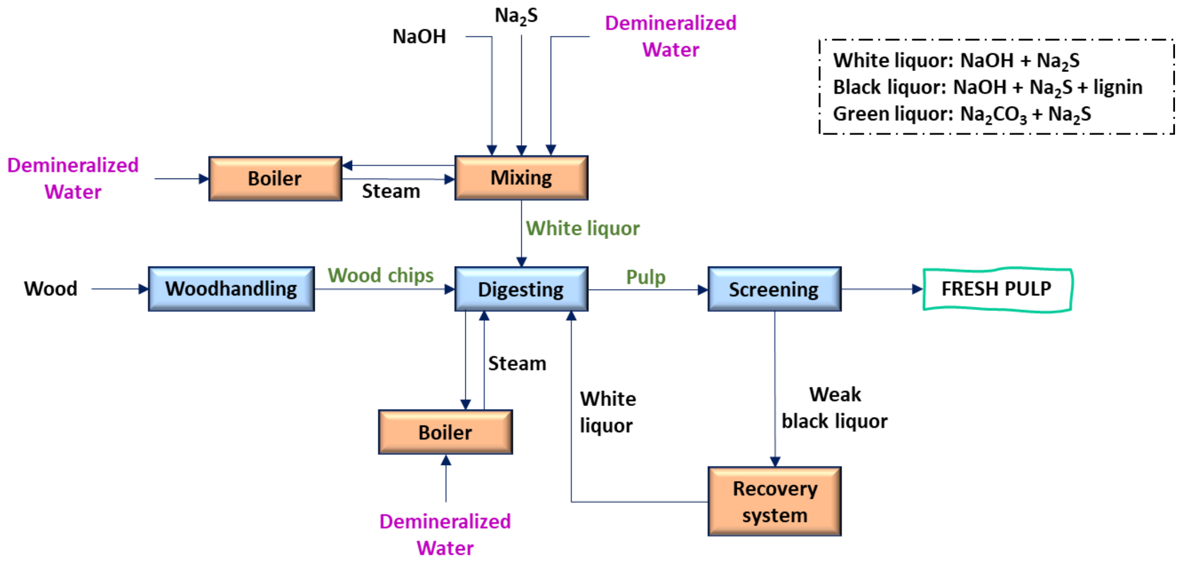

Figure 6: Schematic diagram of the Kraft pulping process

(Diagram adapted from ref. 20,21 & 22)

- *Woodhandling*: In this section the wood is introduced into a line of three equipment in order to achieve clean wood chips for the digester equipment. The involve parts are debarking, chipping and cleaning. These treatments would not be inside this case study.
- *Mixing*: in this step the "white liquor" is produced in a mixer with heat supplied by a steam line coming from a boiler. The white liquor is a water solution of sodium sulphide (Na_2S) and sodium hydroxide (NaOH). Both components are diluted previously separately in tanks with demineralized water. The 80% of the lignin, which binds the cellulose fibres together in the wood, is chemically dissolved by this white liquor into the digesting treatment *Cheremisinoff (2010)*. Moreover, the 50% of hemicelluloses and the 10% of celluloses are at the same time dissolved.
- *Digesting:* the main purpose of digesting also known as cooking is to remove lignin and other impurities from the chips fibres and to keep cellulose and hemicellulose as far as possible, so that the fibres can be separated into pulp by this chemical method *(CNBM Group Corporation)*. The raw materials introduced are the chips and the white liquor, the digester starts to agitate the components and then the temperature and pressure are elevated by a heat exchanger incorporated inside the equipment which works by a steam line from a boiler.

The reactions done in the digester equipment are produced when the Na_2S hydrolized in presence of water and gives hydroxide (NaOH) and hydrosulphide (NaSH). The reaction that takes place is:

$$Na_2S + H_2O \leftrightarrow NaOH + NaSH \qquad (1)$$

Thus, the main chemical reaction in the Kraft cooking process can be expressed as:

$$NaOH + NaSH + Wood \rightarrow Na - compound + S - compound \qquad (2)$$

Where the wood represents various organic compounds as like cellulose, hemicellulose, lignin, fats and resins.

Finally, what comes out of the equipment is the pulp formed together with the white liquor, which has changed its colour to black by the high percentage of Na_2S *(Pulp Paper Mill, 2015).*

- *Blow tank*: it is an atmospheric tank that receives the pulp and recover tremendous amounts of gases and heat which are released during the blow. To discharge the digester a blow valve at the bottom of it opens and the pressure in the equipment pushes the pulp into the pipeline through the blown tank *(Vector Solutions).* In this section the chips that did not break down releasing the fibres in the cooking step are realised in this one *(Aalto University).* While the gases released during this operation will go to an incineration system, which is out of the scope of this case study, because it contains non condensable gases (NCG) which cause environmental damage. These gases are conformed by sulphur compounds *(Lin).*
- *Screening*: in this section the pulp is separated from the cooking liquor *Cheremisinoff (2010),* using the same equipment as it was explained in the cleaning section of the recycled paper, which is the screening equipment. With this final step the pulp is ready to be used in the mixing equipment with the recycled pulp into the papermaking process.

As the recycle pulping, no bleaching treatment will be described because this is not the major problem of this work, that is why oxygen delignification and bleaching would not be presented after the screening section.

Then there is a circuit to recover the chemicals introduced into the digester and at the same time to reduce the water consumption because as it was explained the white liquor is made of chemicals and water. After this circuit, the white liquor can be re-used as raw material for the digester. Here below, are described the steps of this recovery circuit.

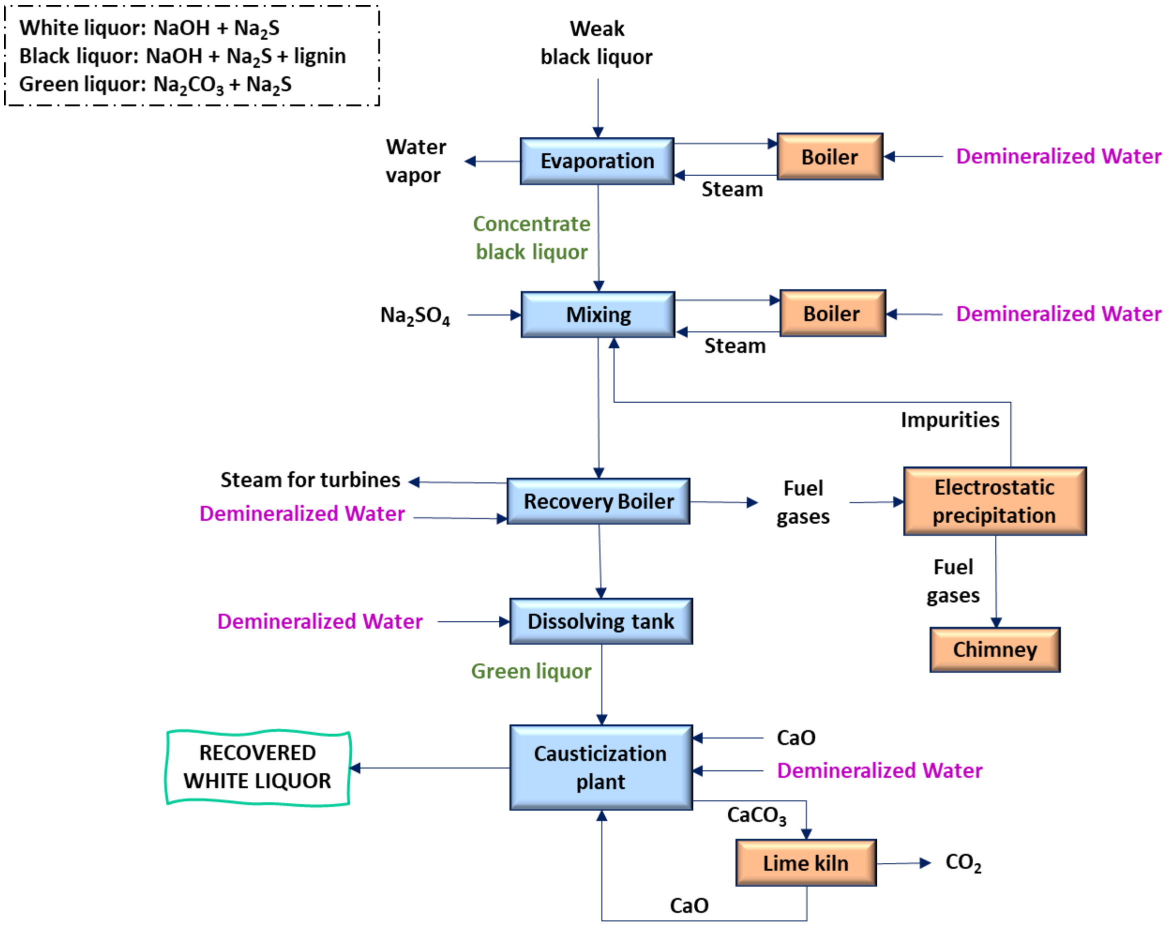

Figure 7: Schematic diagram of the white liquor recovery system

(Diagram adapted from ref. 20,21 & 22)

- *Evaporation*: once the used black liquor, called also weak black liquor, is separated from the pulp in the screening section it is introduced into an evaporator equipment in order to obtain condensed black liquor by removing the water.

  The evaporator has 2 main parts: the vapor body and the heat body. The heat part is composed by a tube heat exchanger where steam provided by a boiler is introduced into the tubes and transfer heat to the weak black liquor which is introduced at the bottom of the equipment and circulates outside the tubes. The steam condensates and is collected into a water storage to be transform again in the boiler. While the weak black liquor circulates to the top of the evaporator, to the vapor section. There, the liquor is separated into water vapor, which is released to the atmosphere, and concentrated black liquor *(Vector Solutions)*.

- *Recovery Boiler*: the concentrated black liquor is burnt in the recovery boiler, a large steam boiler to achieve the objective of this operation, the obtention of green liquor.

First, the black concentrated liquor and sodium sulphate (Na_2SO_4) are mixed in a jacketed vessel and heated by a steam from a water boiler. This step is necessary because the concentrated black liquor is quite viscous and once it is heated it can be pumped and sprayed. The recovery boiler is composed by a furnace, preheated section and economizer. Boiler water circulates in the economizer section inside tubes, there is heated and then it flows to the super preheated part which is at the top of the furnace where the liquor melts. Thus, the mixture is introduced and sprayed into the furnace section where the droplets dry to the point of burning and finally fall to the bottom into a char bed, then, the char burns and the smelt is collected *(Vector Solutions).* The char bed is a mixture of incombustible residue and solid chemicals *(Vakkilainen, 2022)* while the smelt produced is a mixture of Na_2S and Na_2CO_3. Finally, the smelt is collected in the dissolving tank, which is the fourth part of the recovery boiler section, where is diluted with water to dissolve the molten salts, producing the called green liquor *(Local Wiki).*

In this equipment the Na_2SO_4 is reduced to Na_2S while small particles are entrained by flue gases. The flue gases are organic matter of the black liquor, which produces heat and steam *(Järvinen, 2012-17).* These flue gases are directed out of the recovery boiler and circulated into an electrostatic precipitator where the particles are collected and returned to the mixer while the gases are burned in a chimney *(Vector Solutions).*

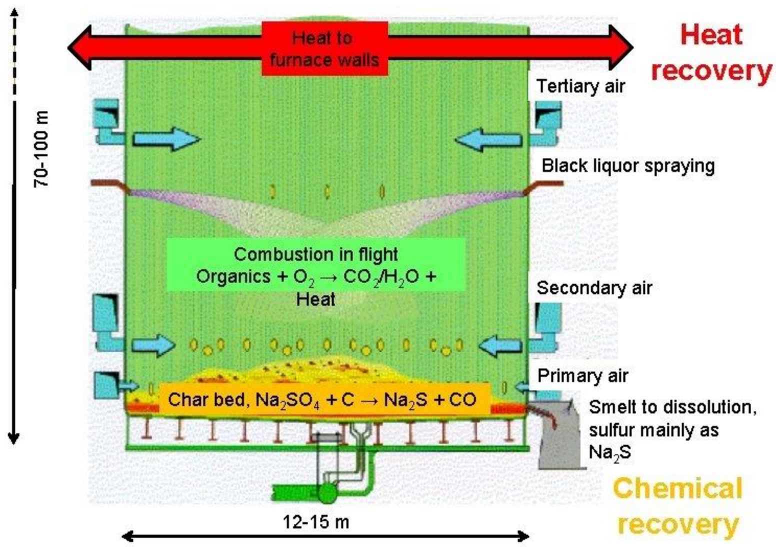

Figure 8: Schematic representation of the processes inside the recovery boiler (Figure extracted from Järvinen (2012-17), ref. 28)

Thus, the reactions that take place in the recovery boiler are:

$$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O \quad (3)$$

$$Na_2SO_4 + 4C \rightarrow Na_2S + 4CO \quad (4)$$

- *Causticization plant*: the green liquor enters into the slaking system which is composed by a conjunction of a mixture vessel and a classifier. The system is full fill with water to ensure the mixture, then lime (CaO) is added to it and at the same the green liquor is also introduced. The mixture is agitated in the vessel section and is introduced to the classifier system where grits, impurities of the lime, are separated, circulating the mixture of green liquor towards the causticization process *(Vector Solutions).* Finally, the reaction is then completed in a causticizer, which is an agitated tank.

In this process, when is introduced the CaO the following reaction occurs:

$$CaO_{(s)} + H_2O \rightarrow Ca(OH)_{2\,(s)} \quad (5)$$

Then, when the slaking reaction that produces $Ca(OH)_2$ is done, this component reacts with the Na_2CO_3 from the green liquor producing $CaCO_3$, called lime mud, and NaOH, which is a component of the white liquor.

$$Ca(OH)_{2\,(s)} + Na_2CO_3 \leftrightarrow CaCO_{3\,(s)} + 2NaOH \quad (6)$$

These two reactions (5 and 6) can be expressed as:

$$Na_2CO_3 + H_2O + CaO \leftrightarrow CaCO_{3\,(s)} + 2NaOH \quad (7)$$

At this point, the mixture is circulated into a screening pressure where the lime mud is separated from the white liquor *(Figueiredo, 2012)*. The white liquor obtained is then incorporated into the digester equipment.

- *Lime kiln*: this process is done to recover CaO from the $CaCO_3$ separated in the screening system, by a rotatory equipment. The equipment is cylindrical, rotatory, and inclined to ensure that the solid goes through the kiln. The $CaCO_3$ is introduced at the high end of the cylinder and heated by a burner which is in the opposite end. As it approaches it is calcined and it is generated the CaO, which is collected in an air-cooling chamber *(Vector Solution)*. The reaction that is done is the below one, where the CO_2 is liberated to the atmosphere and the CaO is collected and recirculated.

$$CaCO_{3\,(s)} + Heat \rightarrow CaO_{(s)} + CO_2 \qquad (8)$$

6.1.3. Papermaking

Following the line of the paper manufacturing process, the next procedure is done in the papermaking machine and in the finishing section. This papermaking machine has differentiated sections to prepare the final product, where showers are implemented in three sections, head box, wire section and press section in order to clean the processed pulp. These sections are the following ones:

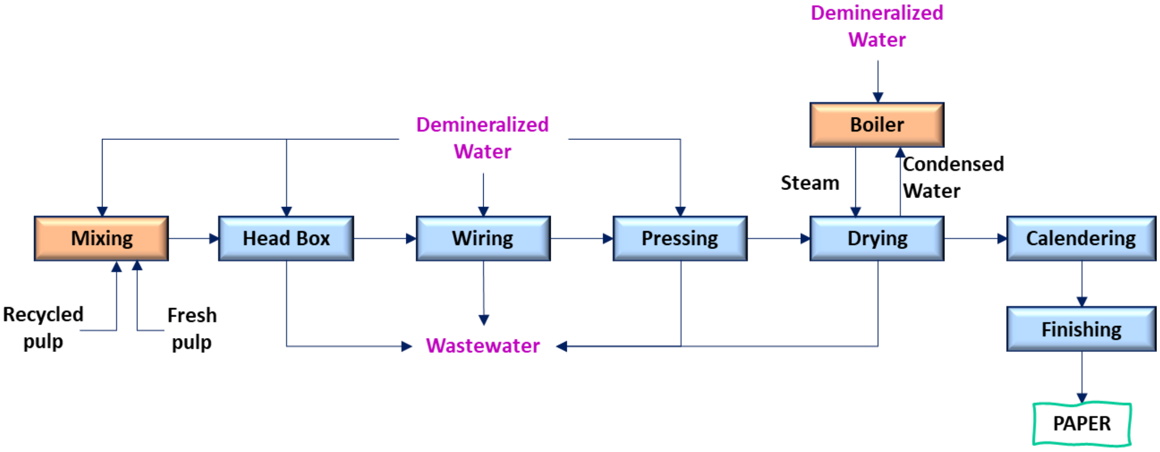

Figure 9: Schematic diagram of the papermaking process
(Diagram adapted from ref. 5,6 & 7)

- Mixing: once, the refining treatment from the recycle pulp is finished, the pulp will be blended with the fresh pulp into a vessel with water *(Northwest Pulp & Paper Association)*. Moreover, this mixer vessel will be used as the pulp storage before being introduced into the papermaking procedure.

- *Head Box*: this is a chamber that distributes the watery pulp stock to the wire section, regulating the thickness and weight of the paper being formed. Thus, the head box continually agitates the watery pulp to prevent the fibres from stay together *(Global Paper Machinery % Engg. Co.)*. Then it is injected between two fast moving wires. The stock in this section is 99% of water, while there is just 1% of pulp and additives. The wire section forms the stock watery pulp into a net *(The Biofore Company, 2011).*
- Wire: the water drains away, and fibres are screened on the top of the wire, on a finely woven plastic mesh which is an uniform layer *(The Biofore Company, 2011).* The fibres quickly begin to bond together to form a watery sheet *(Northwest Pulp & Paper Association).* The wires are specially designed to keep the bond fibres lying down, letting the water drain. Water is collected throughout the papermaking process for reuse *(The Biofore Company, 2011).*
- *Press*: at the beginning of this section the stock is an 80% of water. The pulp is taken into the wet presses by the press felt. The wet presses squeeze the water content down to 50% and the felts absorb water from the paper web. Pressing improves fibre bonding by bringing the fibres closer together *(The Biofore Company, 2011).*
- *Drying*: the paper web continues into the drying section, which consists of steam heated cylinders that evaporate more water. The operation of these cylinders, or also known as drums, is done by steam inside of them. The steam which, when in contact with the treated pulp condenses and is collected through pipes that direct the condensate to the centre of the drum *(Vector Solutions).* Then, in order to recover the condensate, there is a circuit. This circuit consists of a water storage tank, through which water also enters to start the entire circuit, following a boiler that generates the necessary steam for the drums. After going through this series of cylinders, paper has a moisture content of 3 – 8% *(The Biofore Company, 2011).*
- *Calendering*: a calendar is a device with two or more rollers through which the paper is run. The compression from the rollers and the heat received by de paper gives it a smooth and glossy properties.
- *Finishing:* finally, there is the last part of the process that has the task of obtaining the desired look. Here the calendaring paper is introduced into the cutting machine to produce a specific size roll and are packed *(Cepi).*

6.1.4. Main materials

In this section will be described the main raw materials that are involved in the whole paper manufacturing process.

- *Recycled paper*: as it has been described at the beginning in the objective section, the main claimed is to incorporate or increase the circular economy reducing the wastewater, thus the recycled paper has been chosen as the principal component of the pulp. The paper used as recovered must be Kraft paper, such as industrial bags, grocery bags, inner plies of multiwall sacks or plain wrapping paper. More ideally wrapping paper. To obtain a high-quality product it is important to not mix this paper with other kinds.
- *Fresh fibres:* which means that the fibres used will come from forest plants, these fibres will be obtained from industries that carry out the appropriate treatment of the tree logs to obtain the fibres. The source of the supplied logs will be a mixture between softwood and hardwood, thus the advantages of both types will be contemplated in the final paper products.

  Some advantages of softwood are that the fibres are longer and thin, it has a higher percentage of lignin and is more flexible, while the main advantage of hardwood is elevated density, it gives opacity and durability to the final product *(CNBM International Pulp & Paper & Nissha Metallizing Solutions).* Moreover, both types are environmentally friendly, but softwood is better, this is because these trees grow faster than their counterparts, meaning that they can replenish faster *(Dufier Timber, 2021).* Thus, it has been chosen the softwood as the preferred one, the percentage of raw material to be mixed will be higher than the hardwood.
- *Water*: the water used in paper manufacturing process will have already passed through a demineralized water process as it is the required one. This process will have raw water as input, being from a river. The necessary treatment and quality will be explained in the following sections.

6.2. WATER PROCESS

6.2.1. Contaminants

The major contaminants found in water will be explained, differentiating the raw water and the wastewater contaminants, because both types of water will need a process to obtain the required quality water. All the contaminants from the whole work will be manage by an external entity.

6.2.1.1 Wastewater contaminants

Effluent wastewater from pulp and paper industry contains contaminants, mainly from the recycled pulp process. Some of them have been previously identified during the description of some treatments. These compounds cause problems throughout such as reduction in optical and strength properties, this is why there are treatments focused on removing them. Here below is presented a table with some identification parameters of the wastewater stream from the previous paper process.

Subprocess	*Stream number*	*Equipment involved*	*Main Contaminants*[37,18,81]	*Water quantity* [m^3/t]
Papermaking	30	Head box Wire section Press section	*COD* *BOD* *TDS & TSS*	*10,7*

Table 2: Characteristics of wastewater

(Table adapted from ref. 37, 18 & 81)

In this table it can be seen that the wastewater of the whole process is removed by the papermaking process, more specifically by the head box, wire, pressing and drying sections. Some of the main contaminants are presented to show that this kind of water have some pollutants, and this is why it must be treated.

The higher contaminants found are those that constitutes the <u>chemical oxygen demand (COD)</u>, which is the measure of the capacity of water to consume oxygen during the decomposition of organic matter in the water. In other words, it is the amount of oxygen that is needed to oxidise the organic matter present in a quantity of water *(SciMed).*

Moreover, there is another big concern about pollutants which is measured by the biodegradability of the organic matter contained in the wastewater, this indicator is known as the <u>biochemical organic demand (BOD)</u>. It is reflected by the consumption of oxygen needed by aerobic microorganism to oxidize the organic matter present in the wastewater at a specific period *(Dagar, 2022).*

Thus, the principal concerns about contaminants are the organic matter measured mainly by COD and BOD. According to *Dagar (2022)* the pollution represented by COD is approximately 5 times bigger than the BOD. Furthermore, wastewater present a lower number of total solids, being a part of them the suspended solids which include organic and inorganic solids, in comparison of the others two pollutants mentioned. Identifying the materials introduced in the whole process the principal organic solid which could be presented in wastewater is the fatty acid while the main inorganic solids could be sediments and metals coming from the storage or from recovered paper. Hence, wastewater values have been known by *Dagar (2022)* and are presented in the following table.

Parameter	Wastewater Value[37]
pH	8,5-9,5
Conductivity	1,4-3mS/cm[132]
COD [ppm]	716
BOD [ppm]	155
TDS [ppm]	4.410
TSS [ppm]	3.300
DO (20ºC) [ppm]	No data

Table 3: Characteristics of wastewater from large plants

(Table adapted from, ref. 37, 116, 118, 119, 131 & 132)

Some identified detrimental components that can be found in wastewater pulp are:

- *Stickies*: These substances are adhesives with tacky substances coming from recycled paper. They mainly consist of organic materials, such as styrene butadiene, styrene acrylic latex binders, rubber, vinyl acrylates, polyisoprene, polybutadiene etc. *(Gribble,. 2010).*
- *Volatile fatty acids (VFA)*: suspended solid from bacteria or recovered paper contaminated during its usage, storage and recovery. Also, it can be from the deinking treatment.
- *Wood fibres and/or lignin derivates and hemicelluloses*: conforming the organic matter from both types of pulping processes.
- *Sulphide*: this inorganic anion is the main contaminant coming from the Kraft process, from the production of black liquor in the recovery system *(Han, 2021 & Jung, 2011).*

All these compounds, except for wood fibres and/or lignin derivates and hemicelluloses, will conform a small percentage of contaminants due that there are some specific treatments to removed them as the screeners and the deinking sections.

6.2.1.2 Raw water contaminants

On the other side, it is important to know that raw water have contaminants, and this is why it will be explained later the demineralized water process. According to *Cifuentes* article these are the principal pollutants of surface water in comparison of ground water.

- High total dissolved solids (TDS): are the amount of organic solids, fats, metals, minerals, salts and ions, dissolved in a particular volume of water. Particles that are under 2µm size. Common examples are: calcium, chloride, magnesium, potassium, zinc, aluminium, copper, lead… *(Woodard, 2021 & Campbell, 2021).* According to the data published by *CAT (2022)* the river Ebre reached in 2021 628ppm of TDS.
- Low total suspended solids (TSS): such as microplastics, oxidized material and organic matter (living or non-living). Particles that exceed 2µm size. Examples are clay, gravel, sand, silt… *(Campbell, 2021).*
- High biological activity: such as virus, bacteria and protozoa. Following the inform of *CAT (2021)* in a Spanish river, Campredó, it was found that the highest number of biological matter was achieved by the *escherichia coli*, being 372NMP/mL. Also, this, would impact into the level of organic matter that would be presented in raw water.

As this work is taking place in Spain, the values mentioned by the *CAT* inform will be integrated into this case study to have a number of references. Hence the pH and conductivity have been selected from the inform of January, from the river Ampolla *CAT (2023)* while the other references are annual information of the year 2021 from Tarragona rivers. Moreover, the TSS value has been known by the data of Cantabria rivers from *Prego (2006).*

Parameter	Value
pH	8,3[119]
Conductivity [µs/cm]	1517[119]
TDS [ppm]	628[117]
TSS [ppm]	130[133]
Escherichia coli [NMP/mL]	372[117]
DO(25ºC) [ppm]	7,0[123]

Table 4: Characteristics of raw water

(Table adapted from ref. 117, 119, 123 & 133)

Furthermore, it can be seen in the presented table the content of dissolved oxygen (DO), which is highly important in this type of water because it can cause huge problems to the operation and machinery of boilers equipment. DO is a measure of the amount of oxygen, usually thought of as a gas, that is dissolved in water. Oxygen is essential to life and is also the most common element found taking part in corrosion reactions. Thus, can be thought of as the fuel needed for the corrosion process to proceed *(IC Controls).* According to *Miller (2005)* water can be used for boilers equipment with an amount of 5ppb DO.

6.2.2. Required water quality

In this section it will be differentiated two types of water, the demineralized water which is one of the raw materials of the presented paper process having raw water as input and, the regenerated water obtained by the wastewater that has been removed from the paper process.

6.2.2.1 Demineralized water

According to an article of *Cifuentes*, it will be described the quality of the water depending on the requirement and its main characteristics. Water is needed in the whole process, that is why it has been identified the consumers, quality and quantity required. As it has been seen in the schematic diagrams and PFD's the type of the needed water is the demineralized one. The first table in this section shows the characteristics of this required water.

Demineralized water required				
Water quality required	Stream number	Water use	Equipment involved	Water quantity [m^3/t]
High	36 44 45 86	Dilution water	- Pulper - Mixer - NaOH tank - Na_2S tank - Dissolving tank	12,2
High	22 28 29	Shower water	- Head box - Wire section - Press section	
High	88 89	Chemical water	- Causticization (slaking) - CaO tank	
Very high	23 47 80 84	Steam and Power Generation	- Boilers	

Table 5: Characteristics of required demineralized water
(Table adapted from Cifuentes, ref. 33)

This last table shows that demineralized water is required in a high or very high quality. Thus, this work will study some strategy through novel technologies in order to obtain a very high demineralized water, which is more restrictive than the other, by treating raw water. As it can be identified the major quality is required by boilers equipment, this is because as the impurities enter the boiler water, it fouls the steam boiler and leads to damaging the boiler system, reducing its lifespan and efficiency. The main contaminants that cause problems are the dissolved salts and oxygen content. Dissolved salts can accumulate on the surfaces with the evaporation of water as part of the regular operation of the steam boiler. The salt hinders the transfer of heat from the steam boiler, which reduces the overall efficiency of the steam boiler system. If the TDS value is greater than 500ppm can cause scaling narrows in boiler tubes, resulting in reduced flow and contributing to pressure drops. Tubes accumulated with scaling require replacement that costs both time and money. While oxygen will eventually lead to the thinning and pitting of the metal pipes. It can hinder the heat transferring abilities of the steam boiler and causes contamination of the boiler water *(Rakhoh Industries, 2021 & Carbotecnia, 2021).*

Demineralized water has been defined by that kind of water with minimum amount of dissolved minerals, thus, the principal characteristics to define the quality of it are the conductivity and the quantity of dissolved solids contained. In the next table are shown some characteristics that must have the demineralized water in order to be classified as high quality. The major parameters have been known by an analysis of a high-quality demineralized water from *The Distilled Water Company* and, the value of the parameter TDS has been known by the publication of *Vogelzang (2007).* Moreover, the parameters of TSS and the bacteria *escherichia colia* have been known by the *Real Decreto 1620/2007* that details this information in order to obtain regenerated water by wastewater, it is not indicated as demineralized water parameters but, it allows to know that the final values must be lower than the values presented. Furthermore, the *Real Decreto* indicated values refer to water used by boilers, which required higher levels of quality than generic industrial uses.

Parameter	Value	Some components	Value [ppm][114]
pH	5-8[114]	Chloride	<1
Conductivity [µs/cm]	<1[114]	Heavy metals	<0,1
TDS [ppm]	0,028-0,05[115]	Nitrate	<2
TSS [ppm]	<5[35]	Calcium	<0,01
Escherichia Coli [NMP/mL]	Absence[35]	Sodium	<0,02

Table 6: Characteristics of high-quality demineralized water

(Table adapted from ref. 35,114 & 115)

6.2.2.2 Regenerated water

The wastewater will be introduced into a process in order to obtain regenerated water allowing the reuse of it. Here, will be presented a table with some parameters found by different authors. As there is no sufficient information about regenerated water, the parameters shown are a combination between regenerated water, raw water and discharged water parameters, being more suitable the regenerated values. Parameters from discharged water refer to water that can be discharged to the environment which means that will be more in line with the raw water levels. Thus, values refer to both of this kind of waters will work as maximum values that must not be exceed. This is because the wastewater treatment is based on a WWTP process, which use raw water as an input obtaining regenerated water.

The Real Decreto has provided information about the maximum values of the TSS and *escherichia coli* parameters to reused the water, while the DO value has been extracted from the *Nasr (2014)* publication which indicates the quantity of this parameter once water has been regenerated in a WWTP. The pH value has been known by the *Environmental Conservation Rule of Bangladesh (1997)* for effluent discharge, being used by *Mominur (2010)* in his publication about pulp wastewater. The conductivity is from the river Spanish Ampolla extracted from *CAT (2023)*, the organic data information, COD and BOD, from the *European Commission (2021)* for water discharged and. Even though the pH is from another country rule it gives numbers of references.

Parameter	Regenerated water Value
pH	6-8[116] ≤ x
Conductivity	1517µs/cm[119] ≤ x
COD [ppm]	125[118] ≤ x
BOD [ppm]	25[118] ≤ x
TDS [ppm]	No data
TSS [ppm]	<5[35]
Escherichia Coli [NMP/mL]	Absence[35]
DO (20°C) [ppm]	1,47-7,03[131]

Table 7: Characteristics of regenerated water

(Table adapted from, ref. 35, 116,118,119 & 131)

6.2.3. Demineralized water treatment

Once consume process streams had been identified, the following step in order to achieve the goal of this work is to research about the process of the consumed water, the demineralized one. Thus, the process of this kind of water will be explained.

The selected process follows the circuit presented by the *City Water Purifier* company but with an advance water pre-treatment filtration. The main advantages of the advance filtration selected are that it produces small amounts of waste than the conventional one, which also has a bigger design because it need to be composed by more than one equipment to achieve the operation *(Pure Aqua, Inc.)*. Thus, this means the quality of the water once it has been passed through the filtration treatment will be higher. The process is shown in the next diagram:

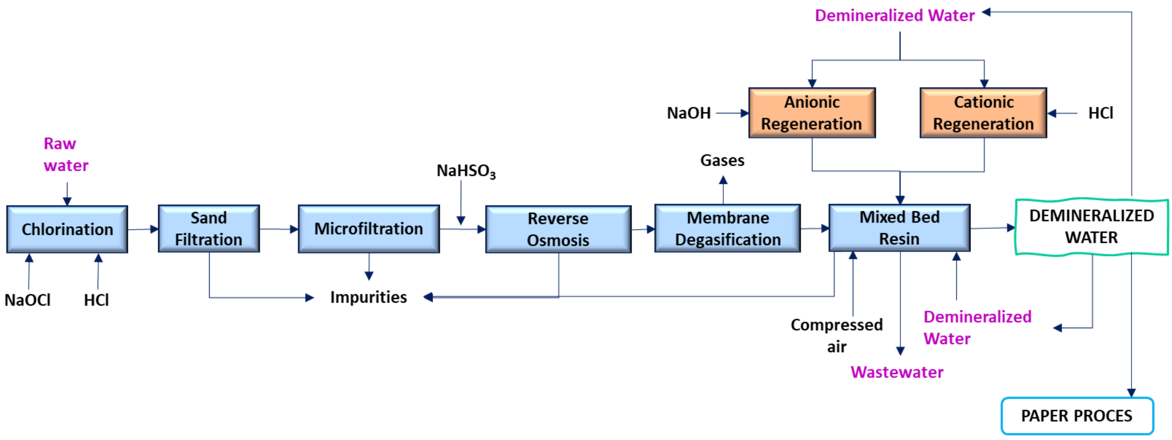

Figure 10: Schematic diagram of the demineralized water process

(Diagram adapted from City Water Purifier company, ref. 41)

As it can be seen in the last schema the process is based on six units, which are the chlorination, two pre-treatment filtrations to prevent the tightness contamination in pipelines and to ensure the efficiency of the following treatments, the reverse osmosis, the degasification and the mixed bed resin. The main contaminants removed by each treatment will be summarized in a final table from this section.

6.2.3.1 Chlorination

Biological contamination is a common issue of water caused by the presence of living organisms, such as parasites, bacteria, protozoan, viruses or other pathogens like algae. Chlorine inactivates microorganism by damaging its cell membrane. Once the cell membrane is weakened, the chlorine can enter the cell and disrupt cell respiration and DNA activity. This compound is highly effective against bacteria compared to the rest of contaminants.

Thus, chlorine will be added to a tank of raw water to oxidize the biological contamination and prevent the growth of it at the following treatments. Chlorination can prevent biological grow in the RO membrane surface, because of that this treatment will be the first one in this water process. Once the raw water arrives to the RO system fouling will not occur in the membrane system, because there will not be as much biological matter content allowing an efficiently removal of the last biological matter that could exist at that point *(Care Water)*.

Once the chlorine has been added to the raw water it will form hypochlorous acid (HOCl) and hypochlorite ions (OCl^-), which are the main oxidating and disinfecting compounds, being the HOCl the most effective. The amount of each compound present in the water is dependent on the pH level of the water.

$$Chlorine\ form + H_2O \rightarrow HOCl + OCl^- \qquad (9)$$

The combination of HOCl and OCl^- ions makes up what is called "free chorine", which have a high oxidation potential that means many different compounds are able to react with it.

First, when chlorine is added to the raw water it is called "chlorine demand" which is the amount of chlorine that is required to satisfy all the impurities presented, which can be also described as the amount of chlorine needed before free chlorine can be produced. Once the chlorine demand has been met, breakpoint chlorination (the addition of chlorine to water until the chlorine demand has been satisfied) has occurred. After the breakpoint, any additional chlorine added will result in a free chlorine residual proportional to the amount of chlorine added. Residual chlorine is the difference between the amount of chlorine added and the chlorine demand. This residual chlorine helps to prevent recontamination of the treated water. This explanation is shown in the next diagram.

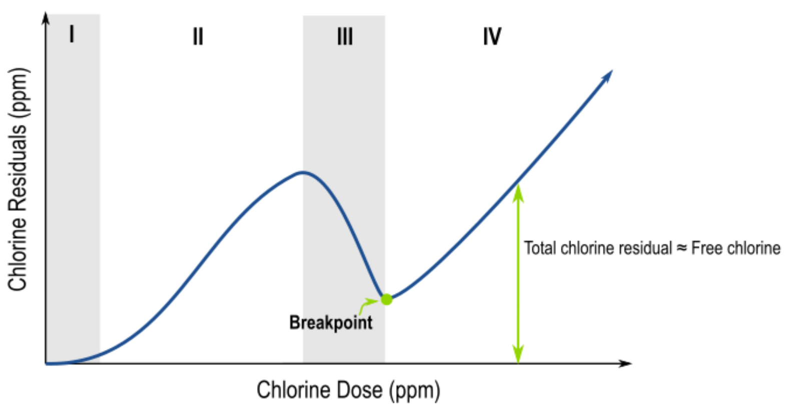

Figure 11: Diagram of the chlorine breakpoint curve
(Figure extracted from Kuntze, ref. 101)

There are three different forms of chlorine to be used, but the selected one has been the liquid sodium hypochlorite (NaOCl) because is the easiest to handle and required less amount of the compound than the other forms to treat the same volume of water. Once NaOCl is in contact with water it will dissociate into its ions, sodium (Na^+) and hypochlorite ions (OCl^-) forming the HOCl and NaOH. Then the HOCl will be dissociated in water as OCl^- and H^+.

$$NaOCl + H_2O \leftrightarrow HOCl + NaOH \qquad (10)$$

$$HOCl \leftrightarrow OCl^- + H^+ \qquad (11)$$

The NaOCl will increase the pH of the water through the formation of NaOH. To obtain HOCl, which is more effective removing contaminants, the pH of water should be decreased *(SDWF & Akvopedia)*. Hence, at the same time NaOCl is introduced to the raw water tank the HCl is also entered to lower the pH and increase the disinfecting power *(Rowe, 2013)*. These reactions will be done in a contact tank, with an agitator. The NaOCl and the HCl will be introduced by gravitation into the contact vessel. The HCl will have a loop conform by a pH analyser and a controller to ensure that the pH of the water is acidic, thus, the HCl discharged will be controlled.

According to a publication of *NetSol Water* company the major difficulty with the polyamide thin-film RO membranes is the removal or annihilation of any chlorine or other potentially oxidative chemicals. Free chlorine has a low tolerance for this membrane. Thus, when the water has been discharged from the chlorination system it will be treated then into the microfiltration one, which would not suffer any damage as the membrane is made of other material, then it will be directed to a storage vessel that is previous of the reverse osmosis. There in the storage vessel a reducing agent, liquid sodium bisulphite ($NaHSO_3$) will be added. Reducing agent injection is one of the most prevalent ways for breaking down chlorine.

The $NaHSO_3$ has been selected because is the most common reducing agent and has a higher efficiency than other, as it preferentially interacts with free chlorine to convert it to the harmless chloride ion and obtaining at the same time sodium sulphate ($NaHSO_4$). The reaction that is carried out is the following one:

$$NaHSO_3 + HOCl \rightarrow NaHSO_4 + HCl \qquad (12)$$

In order to ensure that the water has been treated in this step surely it will be added to the outline stream an oxidation reduction potential (ORP) sensor. This sensor measure determines the oxidizing or reducing potential of a water sample. It indicates possible contamination, especially by industrial wastewater. It does not measure the quantity of chlorine, but it is a real-time reading of chlorine's performance. To ensure that the RO membranes will be protected ORP measurement should be less than 200mV *(Garcia, 2020 & Ysi)*. Thus, the combination of this sensor with a controller, the $NaHSO_3$ will be added in less or more amount to the water treated.

6.2.3.2 Sand filtration

Sand filtration is the first filtration done in this process to reduce the number of dissolved ions and big particles through granular material *(PB International)*, thus, the main objective of this treatment is to ensure that the following systems, which are the microfiltration first and then the reverse osmosis, are not damaged and at the same time to prevent the fouling phenomena.

Raw water is introduced into the filtration vessel and is discharged by troughs that are as water canals, then when those are full filled water flows vertically from the top of the vessel to the bottom of it, passing through a filtered bed made of mixed media. After being treated by the filtered mixed media it will be collected by a drainage system at the bottom of the vessel, composed by strainers.

The filter will show a high pressure drop across the bed, which will indicate that the mixed media have to be clean from impurities in order to continue removing them *(WaterProfessionals)*. According to *Halfor (2020)* rapid sand filtration is indicated for process industry and water works, operating in discontinuous method. This method means that the treatment must be stopped, and a backwash takes place in the opposite direction of the input water to be treated, thus, water enters from the bottom of the vessel. First, compressed air is introduced and then water to clean the bed. The drained water will follow a circuit to a second sand filter to separate impurities from this untreated water.

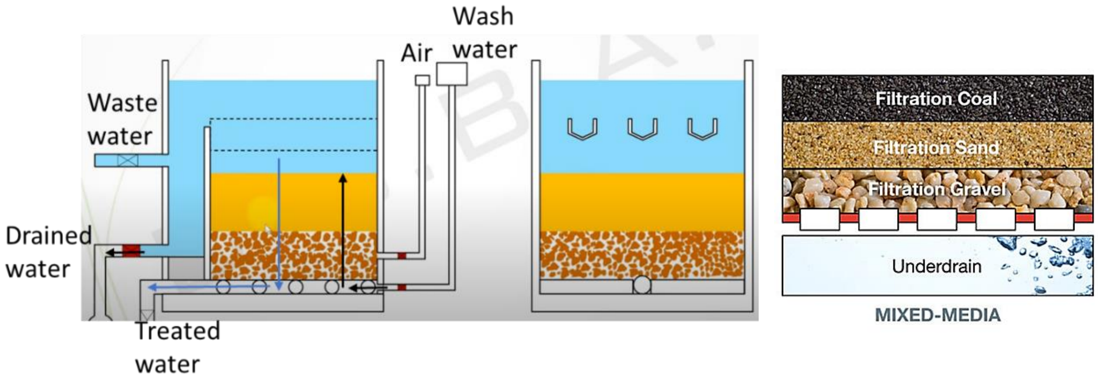

Figure 12: Schematic representation of sand filter and media bed
(Figures adapted extracted from DCBA online (2021) & River Sands (2020), ref. 98, 62)

This mixed-media will be made by three layers of particles with different densities, by anthracite or also known as coal being the lightest at the top, followed by sand and then gravel, deposited in this configuration after backwashing. This filter set up assists in trapping the largest contaminants in the anthracite layer at the top, with the smaller debris being caught in the lower layers.

The anthracite has a larger, more angular particle size to trap particulate matter before it gets to the sand layer and clogs it. It also allows a lower backwash velocity to give the same bed expansion than if sand alone were used. The occupied depth can be from 31 to 46cm, being the grain size from 0,4 to 0,8mm.

Then, in the middle of the mixed bed there is the silica which can occupy 25-40cm of depth with a grain size from 0,4 to 0,8mm. Finally, there is the gravel which occupies 8-15cm and the grain size ranges from 0.3 to 8mm. The gravel maintains a consistent and diffused flow during filtration and prevents disruption of the sand layer during backwash, keeping the sand out of any filtration pipe. Trapping the contaminants in this manner ensures efficiency and longer run times between backwash cycles. Some benefits that explain the selection of this configuration are:

- Due to their design, they can trap and retain larger amounts of contaminants before requiring backwashing.
- Use of the entire bed for filtering out contaminants ensures longer periods of running time.
- Produces high quality, filtered water *(River Sands, 2020 & Schmitt).*

6.2.3.3 Microfiltration

This is the second advance filtration treatment, similar to reverse osmosis, it uses hydrostatic pressure between 0.5 – 5 bars *(Cox)*, produced by the action of a high-pressure pump to increase it on the side of the fresh water, thus, fresh water is forced through a semi-permeable membrane *(Puretec Industrial Water).*

The semi-permeable membrane is microporous *(García, 2009-2010)* and the pore size is ranged approximately between ~ 0.1 – 0.01µm *(Aqua-chem).* This kind of membrane allows to retain suspended solids and solutes of high molecular that have a higher size than the pore sizes, while fresh water and low molecular weight solutes pass through it *(Crystal Quest).*

This treatment follows the cross-flow filtration, which allows water to sweep away contaminant build up and also allow enough turbulence to keep the membrane surface clean, while the standard filtration collects contaminants within the filters systems. There is one input where the water enters and two outlets, one where the permeate is obtained, which is the treated water and the other is the concentrated flow, which has the components retained to be reject *(Puretec Industrial Water).*

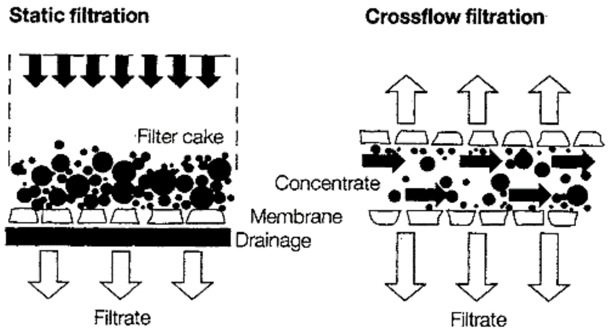

Figure 13: Schematic representation of water flow

(Figure extracted from Scott (1995), ref. 54)

The selected material of the membrane has been done by a publication of *Wang (2013)*, where it is indicated the use of the polyvinylidene fluoride (PVDF) for this treatment when the following system is the reverse osmosis. Also, an article from *Febriasari (2020)* compares this material with polysulfone (PSf) and the result shown is that the PVDF is more hydrophilic and has smallest pores. Thus, PVDF is the selected membrane material.

Moreover, the chosen membrane configuration has been the hollow fibre because it has the highest packing density compare with other types, including the easiest to clean, being one of the most used *(Wenten, 2008 & Wang, 2013).* While it will work with a module of inside-out because of the higher water permeability than the outside-in, even though it is more apt to foul but, the probability of this phenomenon occurring is lower due to the fact that there is a previous filtration treatment. This type of configuration utilizes thousands of long, porous flexible filaments ranging from 1 to 3.5mm wide, that are potted in place in a PVC shell. Water is introduced into the shell and then it passes through the membrane filaments to the outside of them while the contaminants continue the circuit until exit the system *(Synder Filtration).*

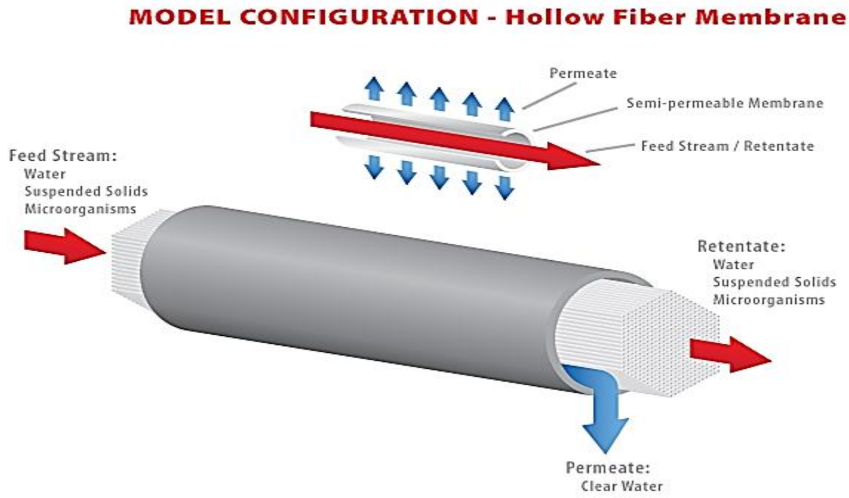

Figure 14: Schematic representation of the hollow fibre inside-out MF

(Figure extracted from Synder Filtration, ref. 60)

6.2.3.4 Reverse osmosis system

This treatment has the same process principal explain previously in the microfiltration system, but here the membrane permeates only the solvent and, which is the water, retain the solute and dissolved solids *(Hisham, 2002)*. Also, the differential pressure must be greater than the osmotic pressure, higher than >20 bars *(Cox).*

The type of membrane used in reverse osmosis are the dens ones. Those membranes are semi-permeable and pore-free structures where the passage of substances through the membrane follows a solution diffusion model, in which the solution components dissolve in the membrane and subsequently diffuse through it. Thus, the different solubility and diffusivity of the solution components in the membrane allow the separation of molecule-sized substance and ions. *(Condorchem Envitech).*

It has been selected the aromatic polyamide as membrane material, which is one of the most used materials for reverse osmosis. This material is stable against chemical and biological agents *(Martín, 2016),* thus, it is less susceptible to biological fouling. This kind of membrane rejects 98% of contaminants *(Advanced Water Filters),* specially indicated for COD and BOD as it can remove a 99% of them *(Keshav, 2012)*. The semi-permeable membrane is used with two more different layers. First it will be the dens polyamide membrane, secondly a porous PSf introduced that provides mechanical strength to withstand high pressure during the treatment, good stability in a wide pH range and it has a pore size lower than 1µnm. Then thirdly, it will be a polyester layer which main function is to provide mechanical stability to the layer system.

Moreover, the selected configuration is the spiral one, because it is used when a high throughput is required, and it have less fouling problems than others configurations *(Butt, 1996).* The spiral configuration is built by surrounding a collector pipe with the layers system, where the PSf layer will be the feed spacer for the water that is wanted to be treated and the polyester layer will be the permeate carrier *(Martín, 2016).* Polyamide membranes edges will be glued together having between a permeate spacer. The purpose of the feed spacer is to provide space for water to flow between the membrane surfaces allowing a uniform flow between the membrane leaves *(Synder Filtration).*

Then, once feed water is introduced into the reverse osmosis cylindrical vessel it will flow, by the cross-flow method, through the feed spacers tangentially across the length of the element. Water will pass across the membrane surface into the permeate spacer, retaining contaminants, where it will follow a circuit towards the central permeate tube and will be collected at the end of the edge. Contaminants of the feed then become concentrated at the end of the element body *(Synder Filtration).*

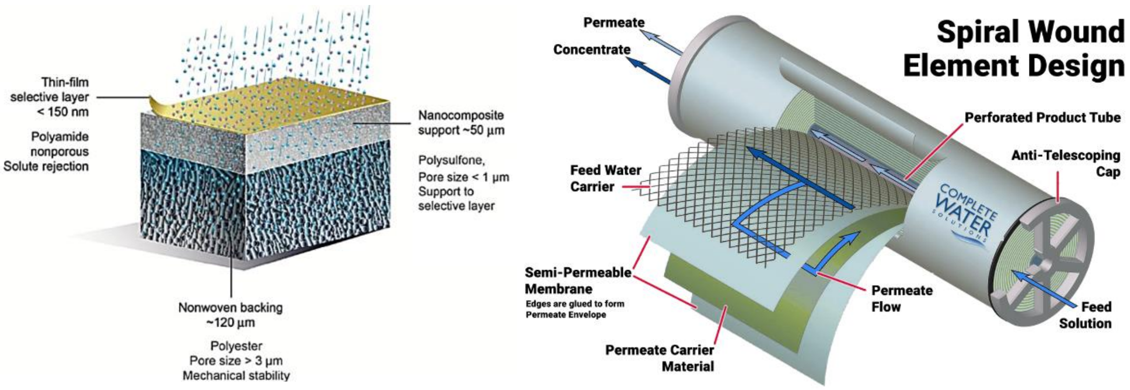

Figure 15: Schematic representation of the reverse osmosis configuration

(Figure extracted from Idarraga (2018) & Olszak (2020), ref. 52 & 140)

6.2.3.5 Membrane degasification

Previously it was mentioned that the higher quality is needed for boilers equipment, this is why here it will be explained a treatment to purifier more the demineralized water to be used in the paper process. As it has been identified the TDS and TSS will have been removed once this treatment is reached. Hence, the other contaminant which is detrimental for boilers is the content of DO in water. According to *HydroGroup* company this treatment should be a polishing step for the osmotic water.

After seen the different degasification processes, it has been selected the membrane degassifier system, because of the higher efficiency that presents. This process allows to achieve an oxygen content called residual because is less than 1µg/L and a residual content of carbon dioxide of less than 1ppm. Some advantages that have are that is smaller than the other degasification equipment, it operates at a pressure less than 5bar and the use of chemicals is optional. To make the treatment more sustainable no chemical will be used in this case study, only a vacuum will be introduced into the system. Moreover, when chemicals are introduced the levels of TDS are increased, being are detrimental for the boiler equipment.

Hence, the treatment is based on a membrane that allows the pass of gases within it, it is characterized by being microporous, hydrophobic and made of hollow fibres contained in a large area promoting mass transfer. Thus, water will not pass through the membrane because of the first two characteristics mentioned.

Water to be treated is introduced at one end of the tubular vessel that contains the membrane, it flows outside the membrane, there the gases pass through it and circulate in the middle of the vessel until being removed by a pipe. There is a baffle in the centre, which forces liquid to flow radially over the fibres to maximize membrane surface area contact. Vacuum will be introduced inside the membrane in a counter-current flow direction by the operation of a pump, it offsets the equilibrium between the liquid phase and gas phase by the driving force created to transfer mass. Then, the gases removed will be liberated to the atmosphere *(Maintech Systems & Miller, 2005).*

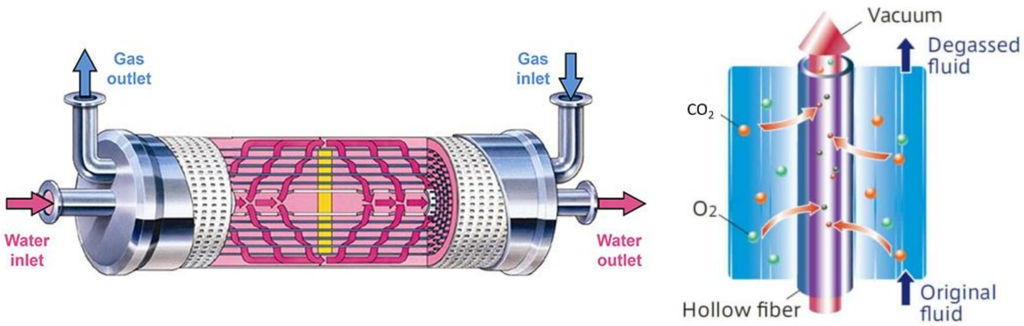

Figure 16: Schematic representation of the membrane degasification configuration and operation. (Figure extracted from Membratec & Agape Water Solutions, ref. 130 & 129)

6.2.3.6 Mixed bed resin system

This system is an ion exchange method used to obtain high purity demineralized water and it is often the last treatment step in the water treatment process train, as polishing. It is normally used as post-treatment after reverse osmosis systems *(Hidro Water & American Water Chemicals).* Thus, this system is used to remove TDS when is low, which is suitable for the kind of water that will be treat at this point *(Lian Tadbir Water Reuse Technology).*

It is composed by a column where strong acid cationic resin (SAC) and strong basic anionic resin (SAB) are mixed evenly *(Hidro Water & American Water Chemicals).* These two resins are blended in a ratio of roughly 40% SAC and 60% SAB bed shells *(NetSol Water).*

According to *Dardel (2021)* SAB resins are always regenerated with caustic soda (NaOH) in the demineralisation process with a concentration of 4% and the SAC resin is commonly regenerated by hydrochloric acid (HCl) with a concentration of 5%, thus, these two types will be used. Moreover, the selected path that the regenerant solution will follow is the reverse flow regeneration (RFR), which is also known as counterflow regeneration. This configuration is based on the downflow loading and up flow regeneration. The RFR has been selected because the treated water has much higher purity than the other existing configuration and it requires less regenerant and results in less contaminant leakage.

Thus, the whole treatment can be divided into 6 steps to be explained, which are:

- Service: Once both types of resin have been mixed in the vessel water is introduced from the top of the vessel. There the cations dissolved in the water are exchanged for hydrogen ions (H^+) by a cationic resin, while anions dissolved in water are exchanged for hydroxide ions (OH^-) by an anionic resin. Hydrogen ions and hydroxide ions react to water. With increasing service life, the ion exchange resins deplete *(Lian Tadbir Water Reuse Technology).*

  The reactions that will be carried out will have the following form:

$$NaOH + X^{n-} \rightarrow Na_nX + OH^- \qquad (13)$$

$$HCl + X^{n+} \rightarrow XCl_n + H^+ \qquad (14)$$

$$H^+ + OH^- \rightarrow H_2O \qquad (15)$$

- Simultaneous Regeneration: then when the resins are depleted, they are left to settle, which means that there will be a differentiation between both types, being the anionic resin on the top and the cationic resin below of the first one.

Then, the NaOH is diluted in demineralized water and is introduced into the distributor, which is composed by a nozzle plate. The NaOH will be sprayed into the anionic resin. Simultaneously, HCl is also diluted with demineralized water and injected at the bottom of the vessel, place where it is the cationic resin, then there it is distributed by another nozzle plate. Both dilutions are injected with low flow rate to allow adequate contact time with the resin.

- Drain Down: the regenerants are flushed out gradually by the slow introduction of dilution water from the top of the vessel, typically at the same flow rate as the regenerants solution and are drained by the regenerant collector which is between both types of resins.
- Mixing: compressed air is introduced at the bottom of the vessel in order to mix again the resins.
- Final Rinse: finally, the mixed resins are rinse with feed water at service flow rate until a target water quality level is reached *(Dardel, 2021 & Samco Technologies, 2017).*

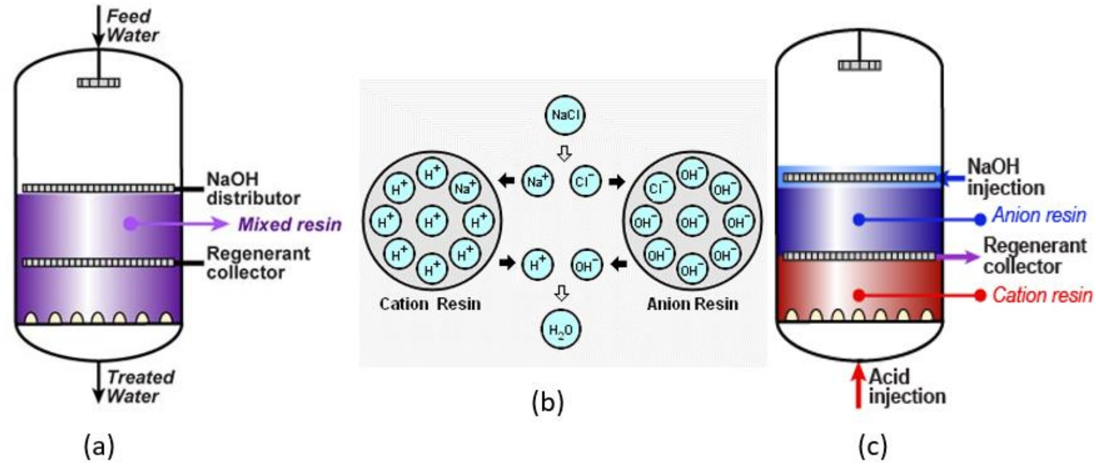

Figure 17: Schematic representation of the mixed bed resin system in progress from figure A to figure B (Figure extracted from Dardel (2021) & Choudhary, ref. 70 & 69)

As it was described previously, it is needed demineralized water to produce the regenerants dilutions, thus, from an external company it will be filled a tank storage with this kind of water. Once, demineralized water is produced by this process, one stream will be recirculated to the dilution vessels and the other will be distributed to the required points of the manufacturing paper process.

			Contaminants removed [44, 46]					
Treatment	Surface of separation	Size (µm)	Solids in suspension	Bacteria	Virus	Multivalent ions	Monovalent ions	Organic molecules
Sand filtration	Granular particles	>5	✓	×	×	×	×	×
MF	Membrane	~ 0.1 - 10	✓	✓	×	×	×	×
RO	Membrane	~ 0.001	✓	✓	✓	✓	✓	✓
Mixed Bed Resin[65, 73]	Ion exchanger	<<2	×	×	×	✓	✓	✓

Table 8: Components removed by the different water treatments

(Table adapted from ref 44, 46, 65 & 73)

As it was described the main contaminants in raw water are TDS, TSS and biological matter. Previously it was mentioned that the reverse osmosis efficiency is about an 98%, thus, this means that all the principal contaminants will be removed to this point as this system is suitable for all the contaminants except gasses, which will be remove in the degasification operation. In the following table are summarized the contaminants that could be presented and their removal treatments.

	Contaminants		
Treatment	COD & BOD	TDS & TSS	Biological matter
Chlorination [134]	×	×	✓
Sand filtration [61]	✓	✓	×
MF [44,46]	✓	✓	✓
RO [44,46]	✓	✓	✓
Mixed bed resin [65,73]	✓	×	×

Table 9: Main contaminants removed by demineralized treatment

(Table adapted from ref. 44, 46, 61, 62, 75 & 134)

According to the information identified in the past sections, the process would achieve the following data information.

Parameter	Raw water Value	Demineralized water Value
pH	8,3[119]	5-8[114]
Conductivity[μs/cm]	1517[119]	<1[114]
TDS [ppm]	628[117]	0,028-0,05[115]
TSS [ppm]	130[133]	<5[35]
Escherichia coli [NMP/mL]	372[117]	Absence[35]
DO (25ºC)	7,0ppm[123]	5,0ppb[125]

Table 10: Characteristics of raw water and demineralized water

(Table adapted from ref. 35,114, 115, 125 & 133)

6.2.4. Wastewater treatment

Previously the contaminants that could be presented in wastewater stream have been described in the contaminants section. Thus, the process to ensure the regeneration of the wastewater from the papermaking process will have three phases to remove that contaminants and then some treatments to obtain the required water. Here below is presented a diagram of the wastewater treatment that will be explained.

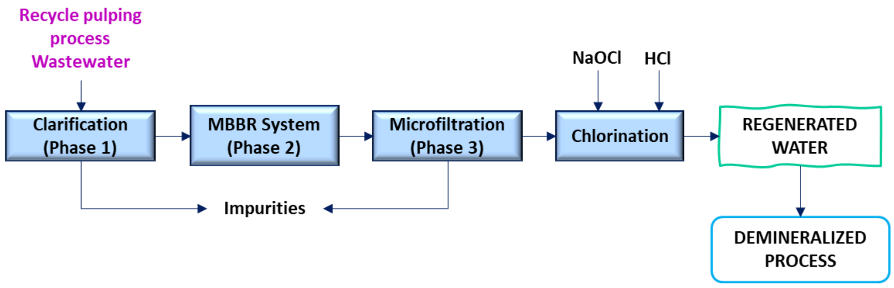

Figure 18: Schematic diagram of the wastewater process

(Diagram adapted from saVRee company (2020) & Veolia (2022), ref. 96 & 91)

As it has identified, the main kind of water required is the demineralized one. Then, once the wastewater treatment process has removed the contaminants presented, the effluent, which is the treated water or called regenerated water, will be introduced into other treatments to obtain demineralized water. Thus, the process will be achieving circular objectives. The needed treatments will be explained below.

6.2.4.1 Primary phase

Previously it has been mentioned that in wastewater there is a low amount of suspended solids, thus, in order to remover them a sedimentation treatment will be done by the equipment called primary clarifier. Approximately 60 to 65% of the suspended solids, as scum, will be removed by gravitational force *(Carmel Area Wastewater District)* and, those suspended solids that sediment, will be removed in a 90 – 95% *(saVRee, 2020).* The wastewater is allowed to settle for a period, in a setting tank and it produces a clarified water effluent in one stream and a liquid-solid sludge (called primary sludge) in a second stream.

The operation begins once the wastewater is introduced into the clarifier bottom, there is circulated by a pipe through the centre of the vessel. There, wastewater is discharged inside the vessel, and a cylindrical energy dissipating inlet will reduce the velocity of it. Thus, having a slower discharged velocity a better sedimentation will be done. Following the water circuit is interrupt by feed well, which helps reducing the velocity and therefore allowing a better settle at the bottom of the suspended solids.

Inside the tank mechanical rake arm with scrapers rotate slowly to move the solids, also referred to as primary or raw sludge, along the bottom. Moreover, scrappers collect any materials that float on the surface of the water. Then finally, a hopper collects the sludge from the pit sludge at the bottom of the vessel. At the same time, suspended solids which have lower density than the water remain floatable with bubbles created by the action of the scrappers. These compounds form a froth or scum, which raises to the top of the wastewater surface. The scum is collected by a large baffle that directs it to the cylindrical wall.

Meanwhile the wastewater is collected in the outlet section, which is at the wall vessel. In order to prevent the circulation of scum to this section, there is a scum baffle at the top surface of the wastewater around the wall to separate it from the outlet section. Then, behind the scum baffle there is the V-notched wire, which allows the pass of the wastewater as a thin film of clean water through the edges forms by the “V” wire structure. Finally, the treated water is introduced to the effluent launder where is collected *(Frankel, 2022 & saVRee, 2020).*

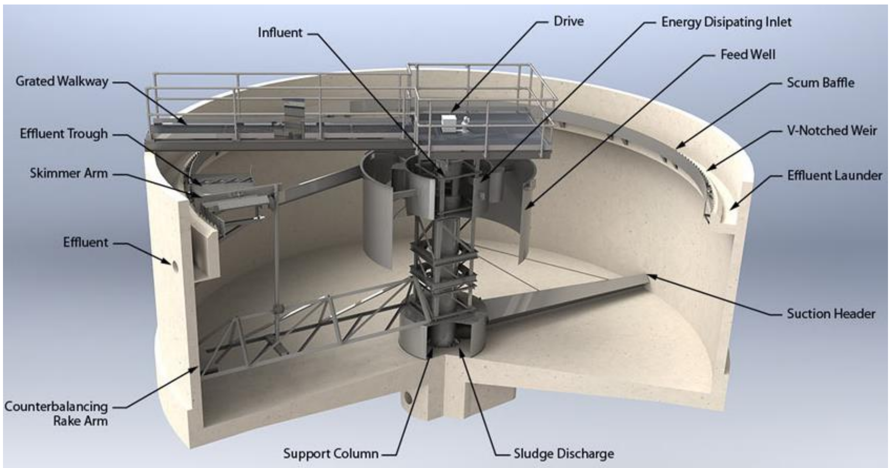

Figure 19: Schematic representation of the clarifier

(Figure extracted from Monroe Environmental, ref. 91)

6.2.4.2 Secondary phase

This phase is essential for removing organic matter which is measured mainly by COD. A seminar of *Veolia* company has provided the opportunity to learn about an innovative treatment compared to the conventional ones, this is the moving bed biofilm reactor (MBBR) treatment, which can achieve higher removal rates in biofilm. Moreover, it is considered suitable for pulp and paper industries. Thus, this phase will be done by the MBBR technology.

Some advantage of the selected treatment is that the concentration of microorganisms is higher than the activated sludge conventional treatment, consequently the biological reactor fills less surface space. Moreover, there is no need for sludge recirculation because of the low TSS concentration in the effluent.

The MBBR treatment is based on biological activity using biofilms that grow attached to the surface of a bio-media, continuously moving within the wastewater medium. Biofilms are composed by a microbial association, microorganisms and their extracellular products, while the bio-media is made of polyethylene or polypropylene with a density close to that of water and are designed to provide a large, protected surface for bacterial growth. The system of the biofilm and the bio-media is called carrier. The reactor is filled with carriers up to a maximum value of 67%. Due to their density being close to that of water and the fact that only part of the reactor volume is filled with them, carriers are allowed to move freely inside the reactor. Moreover, air is injected at the bottom of the vessel, producing air bubbles to agitate the carriers in the medium and allows good conditions for oxygen transfer to the liquid phase *(CESP Africa)*. This type of agitation has been selected because it is commonly used in full-scale reactors top achieve the last characteristics mentioned.

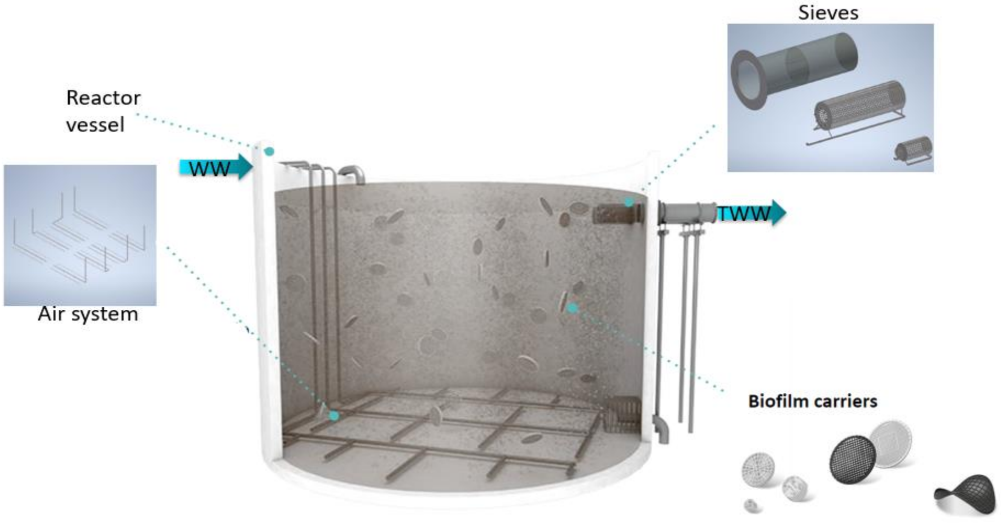

Figure 20: Schematic representation of the MBBR
(Figure adapted extracted from Veolia seminar, ref. 91)

Thus, the organic matter and other wastewater components are diffused into the biofilm where the treatment occurs, mostly by biodegradation. There are 4 phases in the biofilm process. First, there is the liquid phase form by the wastewater in the reactor, then there is the stationary liquid phase, following there is the biofilm and the bio-media surface. Being the biofilm conformed by aerobic and anaerobic microbial layers.

The process begins when the sludge, or also named as particulate matter is attached to the biofilm surface, then, the diffusion of the matter to the inside of this phase, there the hydrolysis happens. The organic chemical in contact with water form two or more new substances. Then, the main reaction is carried out removing the organic matter and producing CO_2 and H_2O. Finally, a contrary diffusion is done, to the biofilm surface where the detachment occurs *(Wikipedia & Veolia).* The excess sludge and the treated water enter to sieves where the mesh material allows the pass of them through it but keeps the carriers inside the vessel *(Lanyu Gustawater, 2020).*

The main reaction occurred in the biofilm carriers is:

$$A + B + O_2 \rightarrow CO_2 + H_2O + C + D \qquad (16)$$

Being:

A = Organic matter

B = Biofilm microorganisms

C = Biofilm biomass

D = Oxidized products (sludge)

The selected biofilm carrier following the indications of *Veolia* is the AnoxkTM F3, which is cylindrical and indicated to the pulp and paper industry.

Figure 21: Schematic representation of the biofilm carrier and process (Figure extracted from Veolia, ref 91)

6.2.4.3 *Tertiary phase*:

Finally, there is the third phase which is not always necessary but, it will be done in this process as this wastewater is wanted to be treated in order to recirculated. Moreover, the water is required with high quality or deeply high. Thus, this phase will be carried out to remove any contaminant that has pass through the other phases without being removed.

According to the information given by the *Veolia* seminar, the MBBR can be complemented with compact technologies such as a second clarification, microfiltration, actiflo…but, in this process it has been selected de microfiltration treatment because this one is part of the demineralized water process. Thus, this information allows to optimize this wastewater process, connecting the secondary phase to the demineralized microfiltration treatment.

6.2.4.1 *Chlorination*

As it has been mentioned previously in the demineralized water treatment, chlorination is important to avoid microorganism growing and to remove the remain ones. Thus, this step is necessary in order to have a low or absence of biological matter. As in this water process there is not an RO system presented this chlorination treatment will be added to the end of the process, without the addition of $NaHSO_3$. Also, it has been decided to use this system at this point according to information based on wastewater processes published by *Suprya* and *Curtis (1998).*

The main contaminants that were mentioned about the wastewater were mainly the COD and BOD, followed by the TDS and TSS. Below, is contaminants and their treatments to be removed are summarized.

	Contaminants		
Treatment	COD & BOD	TDS & TSS	Biological matter
1ry phase[94, 96]	✓	✓	×
2ry phase[91]	✓	×	×
3ry phase[44,46]	✓	✓	✓
Chlorination[134]	×	×	✓

Table 11: Main contaminants removed by wastewater treatment

(Table adapted from ref. 44, 46, 91, 94, 96 & 134)

6.2.5. Proposed water treatment

Once both water treatments have been identified, studied and described, a proposed global water treatment has been possible to reach. Thus, as it has been explained in the wastewater treatment, the tertiary phase of it is based on the microfiltration treatment. This allows to create a circular water circuit that is shown in the following diagram, where the microfiltration and chlorination from the wastewater treatment are removed.

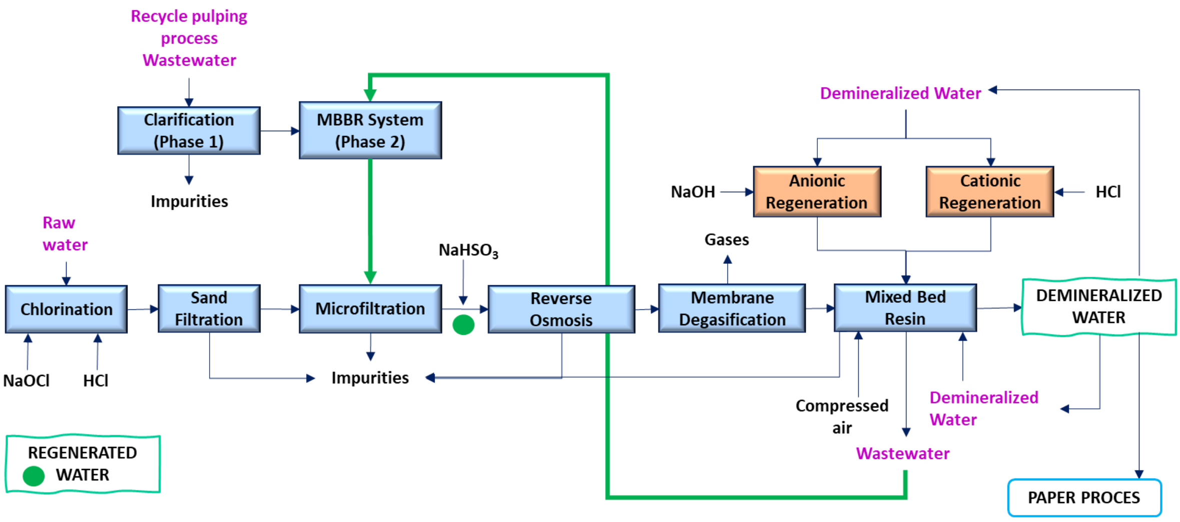

Figure 22: Schematic diagram of the proposed water treatment

As it can be seen in the diagram the microfiltration system from the demineralized treatment is used by both types of water treatments. Thus, once water has been treated by this equipment it will be at this point regenerated. Moreover, as the mixed bed resin system removes wastewater, this will be introduced into the MBBR system of the wastewater treatment. This stream is not introduced into the primary phase because this wastewater has been obtained only by the mixed bed resin system, which means that the quality of this wastewater is higher than the one that comes from the recycle pulping process. Hence, as it has higher quality this stream can be introduced into the secondary phase.

Below is presented a summarized table that shows the main characteristics of the principal types of water that are considered in this case study.

Parameter	Wastewater Value[37]	Regenerated water Value	Raw water Value	Demineralized water Value
pH	8,5-9,5	6-8[116] ≤ x	8,3[119]	5-8[114]
Conductivity	1,4-3mS/cm[132]	1517μs/cm[119] ≤ x	1517 μs/cm [119]	<1μs/cm[114]
COD [ppm]	716	125[118] ≤ x	No data	No data
BOD [ppm]	155	25[118] ≤ x	No data	No data
TDS [ppm]	4.410	No data	628[117]	0,028-0,05[115]
TSS [ppm]	3.300	<5[35]	130[133]	<5[35]
Escherichia coli [NMP/mL]	No data	Absence[35]	372[117]	Absence
DO(25°C)	No data	1,47-7,03[131]	7,0ppm[123]	5,0ppb[125]

Table 12: Characteristics of the main kinds of water

(Table adapted from, ref. 35, 37, 114, 115, 116, 117, 118, 119, 123, 125, 131, 132 & 133)

The TSS and TDS in the wastewater treatment are principally removed in the primary phase and the level of them are polished at the MF, as this last system is mainly indicated for the removal of organic matter, COD and BOD in this treatment. Hence, this means that once the MBBR system is finished the level of the mentioned parameters will be more in line with the values of the regenerated water, which are lower than the raw water values. Thus, the outgoing MBBR stream could be connected to the demineralized treatment avoiding the sand filtration of it. Also, this information could be discussed by the conductivity but, the regenerated water has an assumed value from the raw water.

Moreover, there is no data about the amount of organic matter from the raw water but, it is assumed that raw water will have greater levels of COD and BOD than the outgoing MBBR stream because it has not been treated as the regenerated one when it is at the chlorination point. As it has been indicated, the COD and BOD are removed by the MBBR and MF system, but mainly by the principal one, in the wastewater treatment while in the demineralized treatment are remover by the MF, RO and mixed bed resin. The point where the stream from the wastewater treatment is connected is previous from the demineralized treatments, thus, this stream will have lower levels of organic matter.

Furthermore, the regenerated water does not present the *escherichia coli* bacteria once the wastewater treatment has been finished but, as the chlorination step from this water treatment has been removed it could present a percentage of biological matter including the mentioned bacteria. This does not present a problem because an assumption can be made, wastewater has a lower content of biological organisms as it only requires a chlorination system whereas the demineralized water treatment requires a chlorination and a RO system. Moreover, once both treatments are connected, the residual chlorine from the demineralized chlorination will remove the remain biological matter. This means that this last compound will protect the raw water that has been treated in the chlorination system from the wastewater and at the same time will help this last kind of water to remove the biological matter that could contain. Also, the DO parameter in the regenerated water ranges lower than the raw water corroborating the mentioned above.

7. CONCLUSIONS

- This work does not have precise quantified values, but it will waste less water than the conventional ones, as it is based mainly on a recycle process and at the same time it has a circular water process in order to reuse the wastewater.
- The final proposed process of the water can be applied to any type of pulp and paper industries. This is because industries of this kind follow the same procedures in order to obtain the final product, pulp or paper.
- Moreover, it could be applied also to other kind of industries, such as chemical industries, that are the second ones that consume more water. Some variations will be presented as the products manufacturer in this industry can be really differentiated but the basis of the treatments studied will be the same.
- It has been done this work on a conceptual manner, by bibliographic information due to the limited data shared publicly by companies. Thus, the few data presented are assumptions done extracted by different authors.

8. NOTATION

Abbreviations

AWT	Advanced wastewater treatment
BOD	Biochemical organic demand, mg O_2/L (ppm)
COD	Chemical oxygen demand, mg O_2/L (ppm)
FRC	Free residual chlorine
RFR	Reverse flow regeneration
MBBR	Moving bed biofilm reactor
MF	Microfiltration
NCG	Non condensable gases
PSf	Polysulfone
PVC	Polyvinyl chloride
PVDF	Polyvinylidene fluoride
RO	Reverse osmosis
SAB	Strong base anionic resin
SAC	Strong acid cationic resin
TDS	Total dissolved solids
TSS	Total suspended solids
VFA	Volatile fatty acids
WWTP	Wastewater treatment plant

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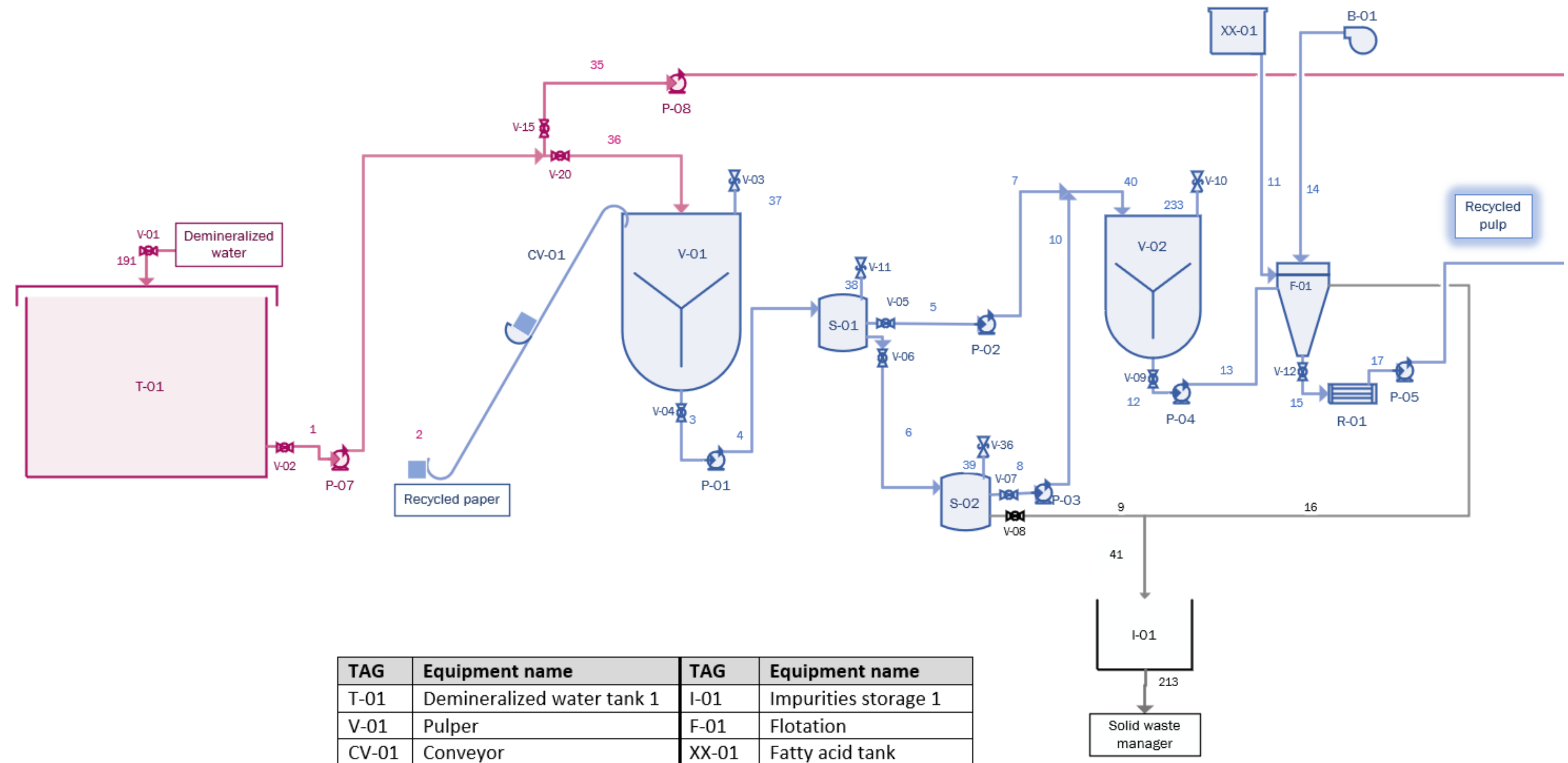
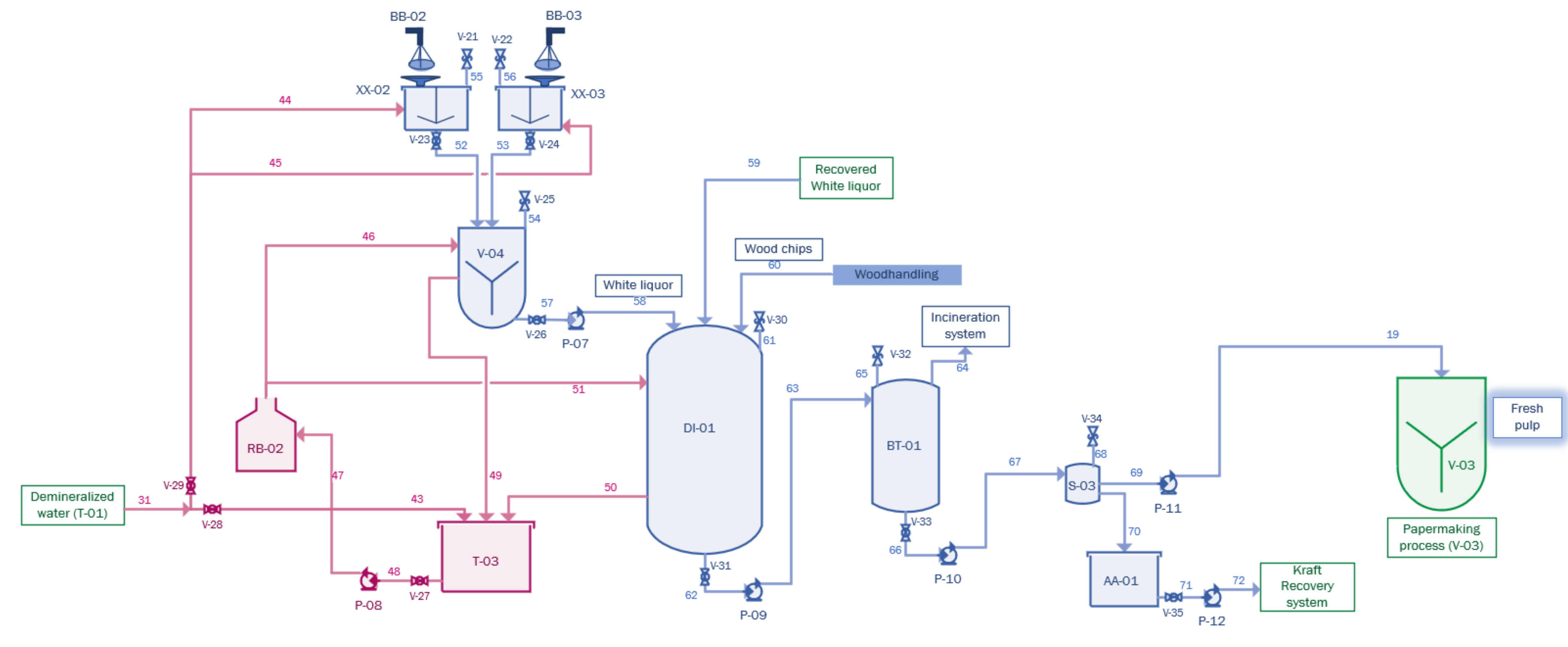
APPENDIXS

APPENDIX I: PFD's



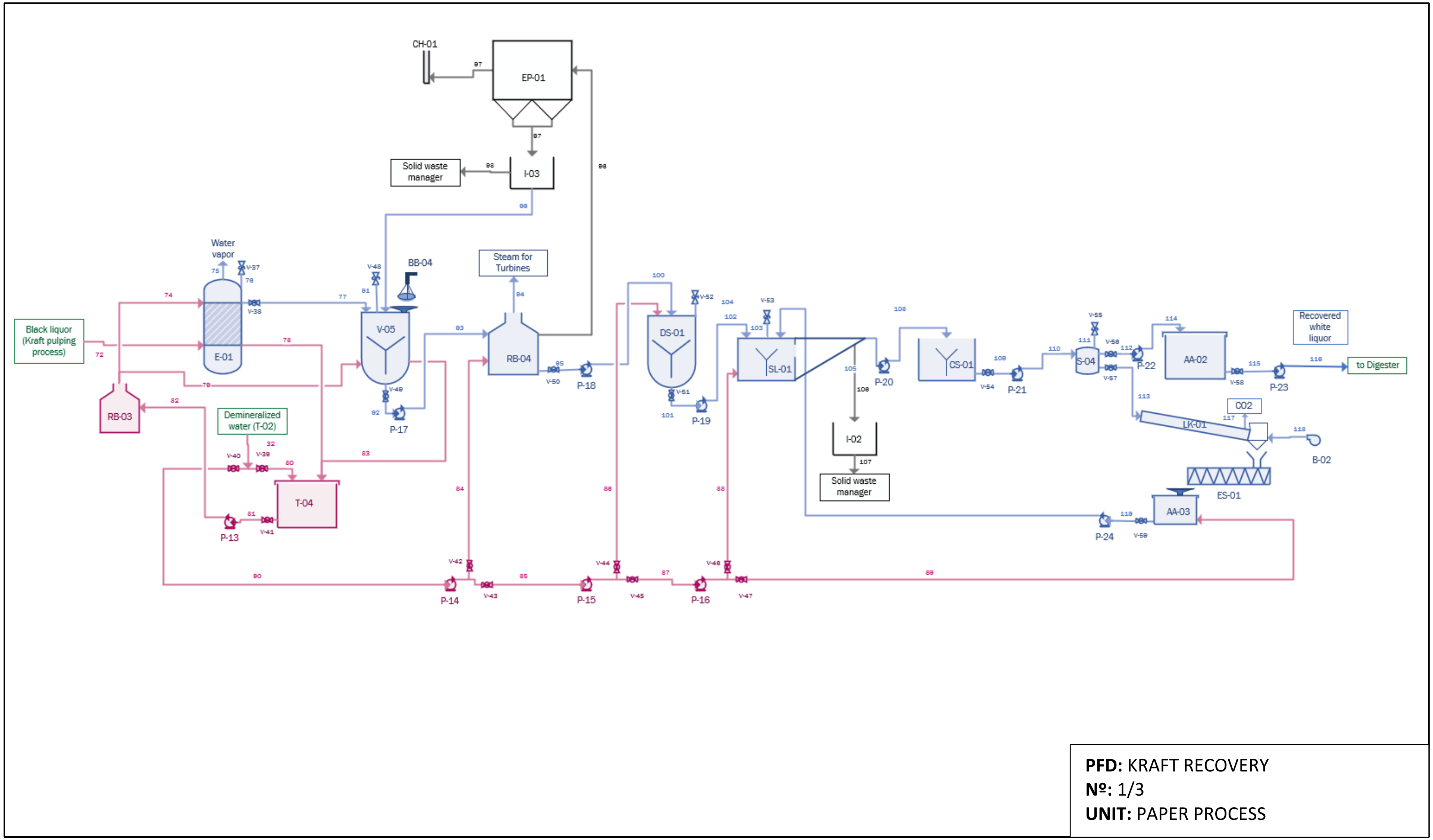
TAG	Equipment name	TAG	Equipment name
T-01	Demineralized water tank 1	I-01	Impurities storage 1
V-01	Pulper	F-01	Flotation
CV-01	Conveyor	XX-01	Fatty acid tank
S-01	Screener 1	B-01	Blower 1
S-02	Screener 2	R-01	Refiner
V-02	Mixer		

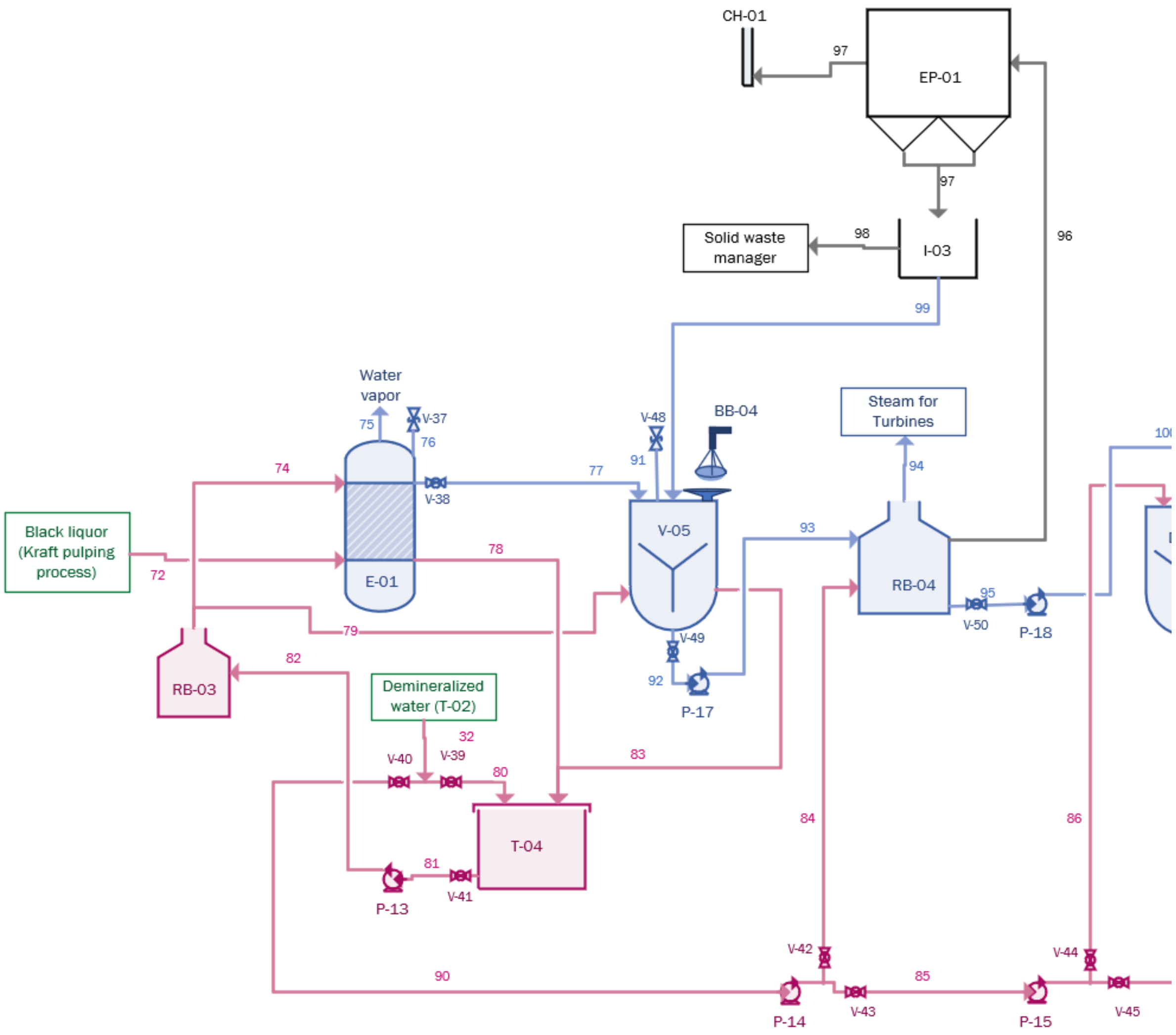
PFD: RECYCLE PULPING
Nº: 1/1
UNIT: PAPER PROCESS



TAG	Equipment name	TAG	Equipment name
XX-02	NaOH dilution mixer	T-03	Demineralized water tank 3
XX-03	Na_2S dilution mixer	DI-01	Digester
BB-02	NaOH bag dispenser	BT-01	Blow tank
BB-03	Na_2S bag dispenser	S-03	Screener 3
V-04	Black liquor mixer	AA-01	Black liquor storage tank
RB-02	Recovery boiler 2		

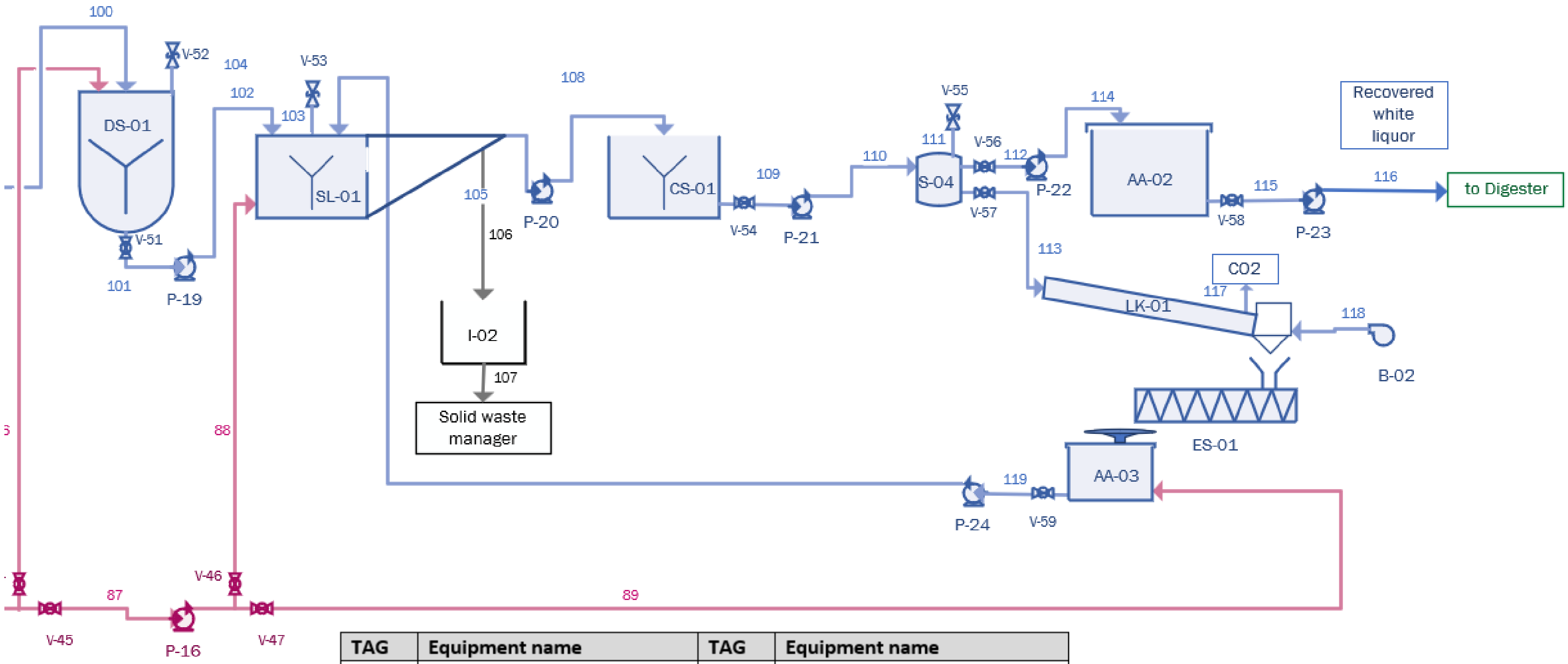
PFD: KRAFT PULPING
Nº: 1/1
UNIT: PAPER PROCESS


CH-01
EP-01
Solid waste manager
I-03
Water vapor
BB-04
Steam for Turbines
Black liquor (Kraft pulping process)
E-01
V-05
RB-04
DS-01
SL-01
CS-01
S-04
AA-02
Recovered white liquor
to Digester
RB-03
Demineralized water (T-02)
T-04
P-17
P-18
P-19
P-20
P-21
P-22
P-23
I-02
Solid waste manager
CO2
LK-01
B-02
ES-01
AA-03
P-24
P-13
P-14
P-15
P-16
PFD: KRAFT RECOVERY
Nº: 1/3
UNIT: PAPER PROCESS



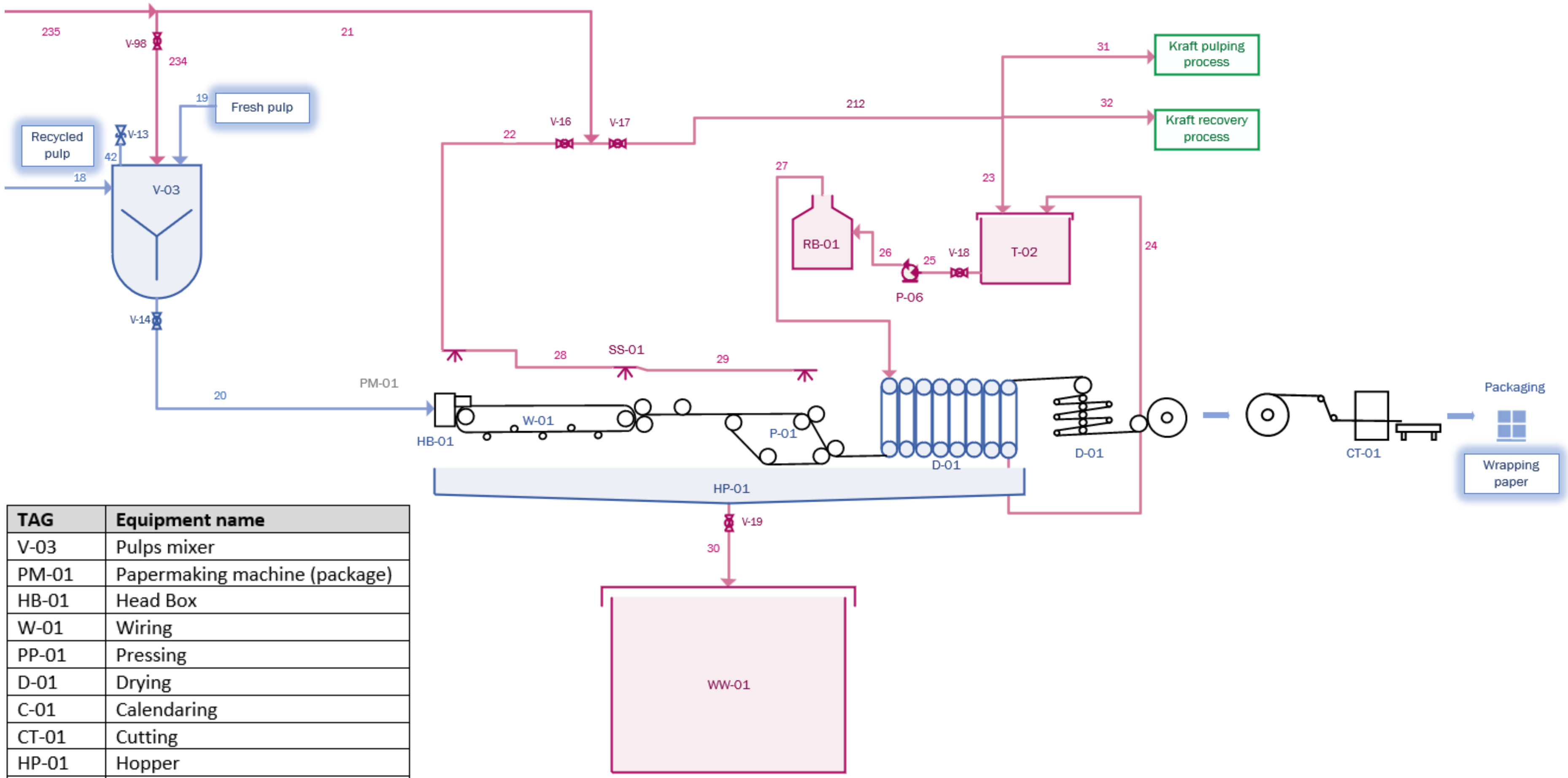
TAG	Equipment name
RB-03	Recovery boiler 3
T-04	Demineralized water tank 4
E-01	Evaporator
BB-04	Na_2SO_4 bag dispenser
V-05	Recovery black liquor mixer
RB-04	Recovery boiler black liquor
DS-01	Dissolving tank
SL-01	Slaking system
CS-01	Causticization tank
S-04	Screener 4
AA-02	Recovery black liquor storage
LK-01	Lime kiln system
ES-01	Endless screw
B-02	Blower 2
AA-03	CaO storage tank
I-02	Impurities storage 2
I-03	Impurities storage 3
EP-01	Electrostatic precipitator
CH-01	Chimney

PFD: KRAFT RECOVERY
Nº: 2/3
UNIT: PAPER PROCESS



TAG	Equipment name	TAG	Equipment name
RB-03	Recovery boiler 3	AA-02	Recovery black liquor storage
T-04	Demineralized water tank 4	LK-01	Lime kiln system
E-01	Evaporator	ES-01	Endless screw
BB-04	Na_2SO_4 bag dispenser	B-02	Blower 2
V-05	Recovery black liquor mixer	AA-03	CaO storage tank
RB-04	Recovery boiler black liquor	I-02	Impurities storage 2
DS-01	Dissolving tank	I-03	Impurities storage 3
SL-01	Slaking system	EP-01	Electrostatic precipitator
CS-01	Causticization tank	CH-01	Chimney
S-04	Screener 4		

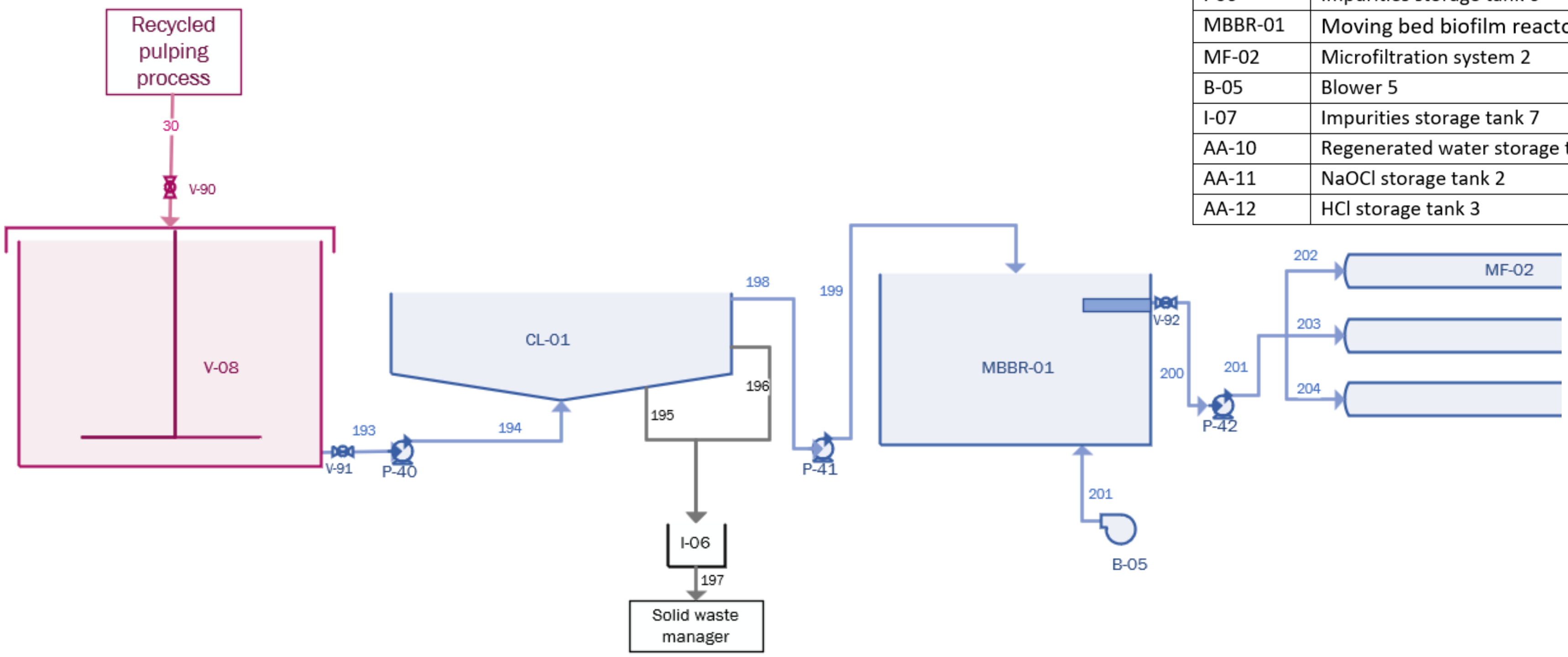
PFD: KRAFT RECOVERY
Nº: 3/3
UNIT: PAPER PROCESS



TAG	Equipment name
V-03	Pulps mixer
PM-01	Papermaking machine (package)
HB-01	Head Box
W-01	Wiring
PP-01	Pressing
D-01	Drying
C-01	Calendaring
CT-01	Cutting
HP-01	Hopper
T-02	Demineralized water tank 2
RB-01	Recovery boiler 1
SS-01	Shower System
WW-01	Wastewater tank 1

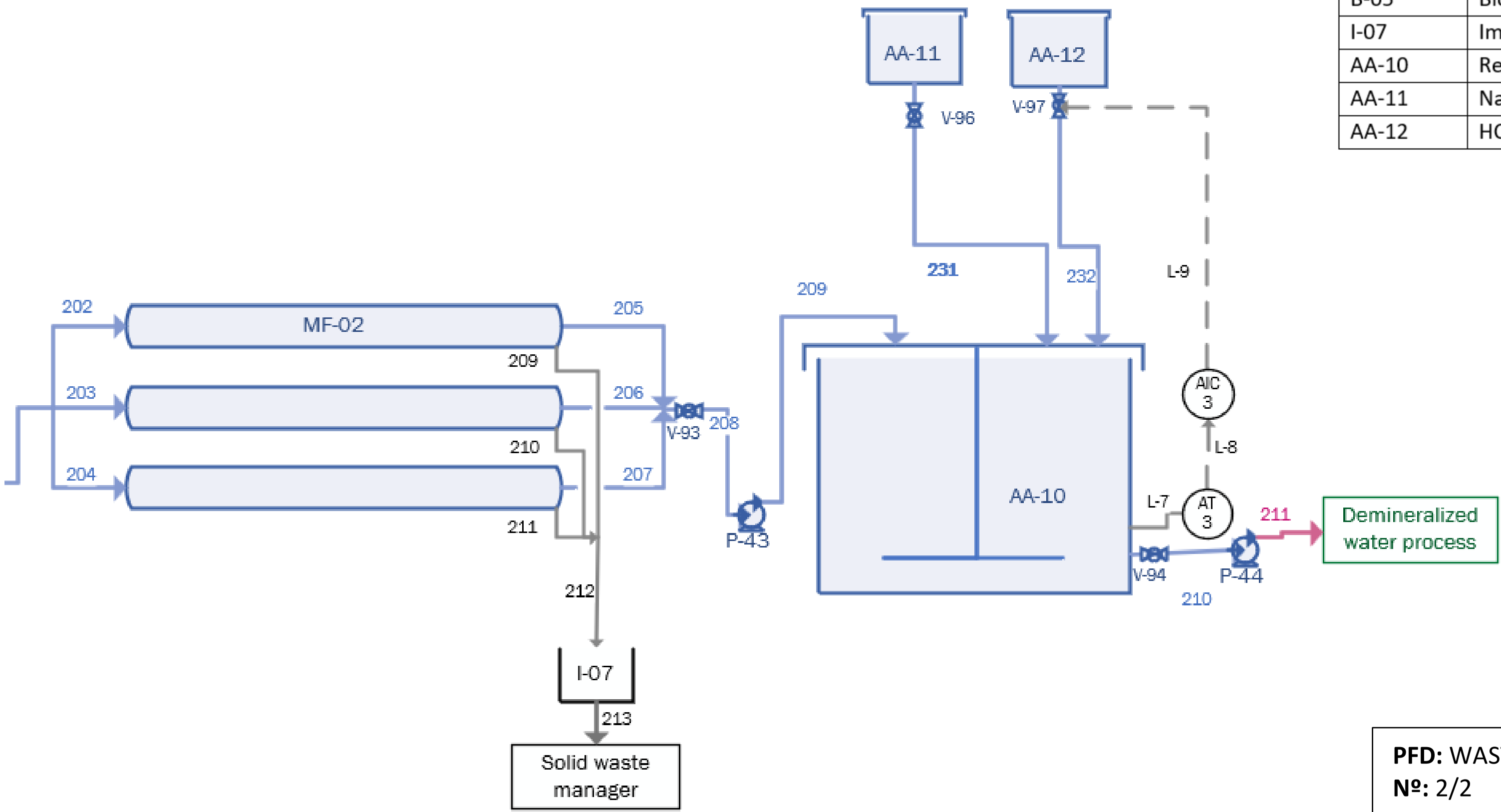
PFD: PAPERMAKING PROCESS
Nº: 1/1
UNIT: PAPER PROCESS

TAG	Equipment name
V-08	Water tank mixer
CL-01	Clarification tank
I-06	Impurities storage tank 6
MBBR-01	Moving bed biofilm reactor
MF-02	Microfiltration system 2
B-05	Blower 5
I-07	Impurities storage tank 7
AA-10	Regenerated water storage tank
AA-11	NaOCl storage tank 2
AA-12	HCl storage tank 3

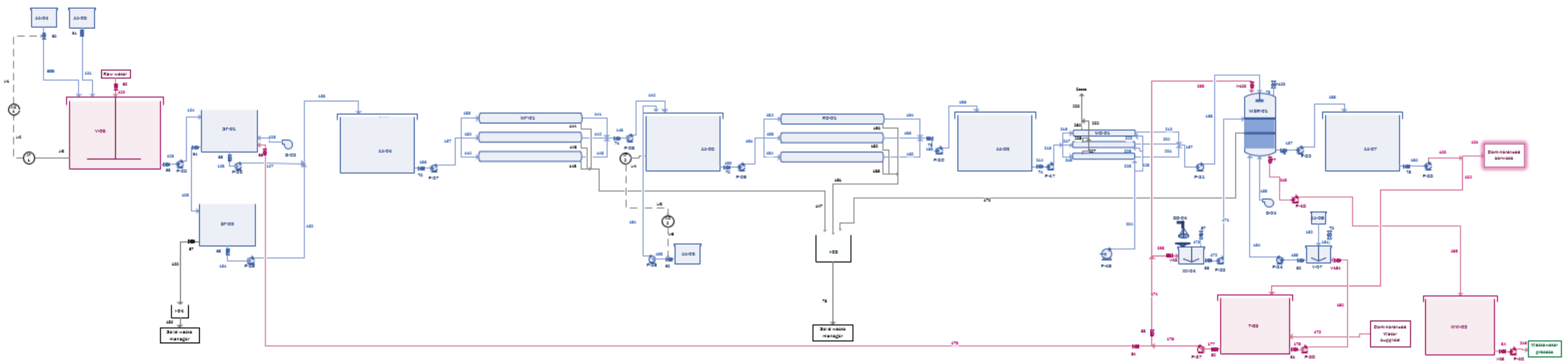


PFD: WASTEWATER TREATMENT
Nº: 1/2
UNIT: WATER PROCESS

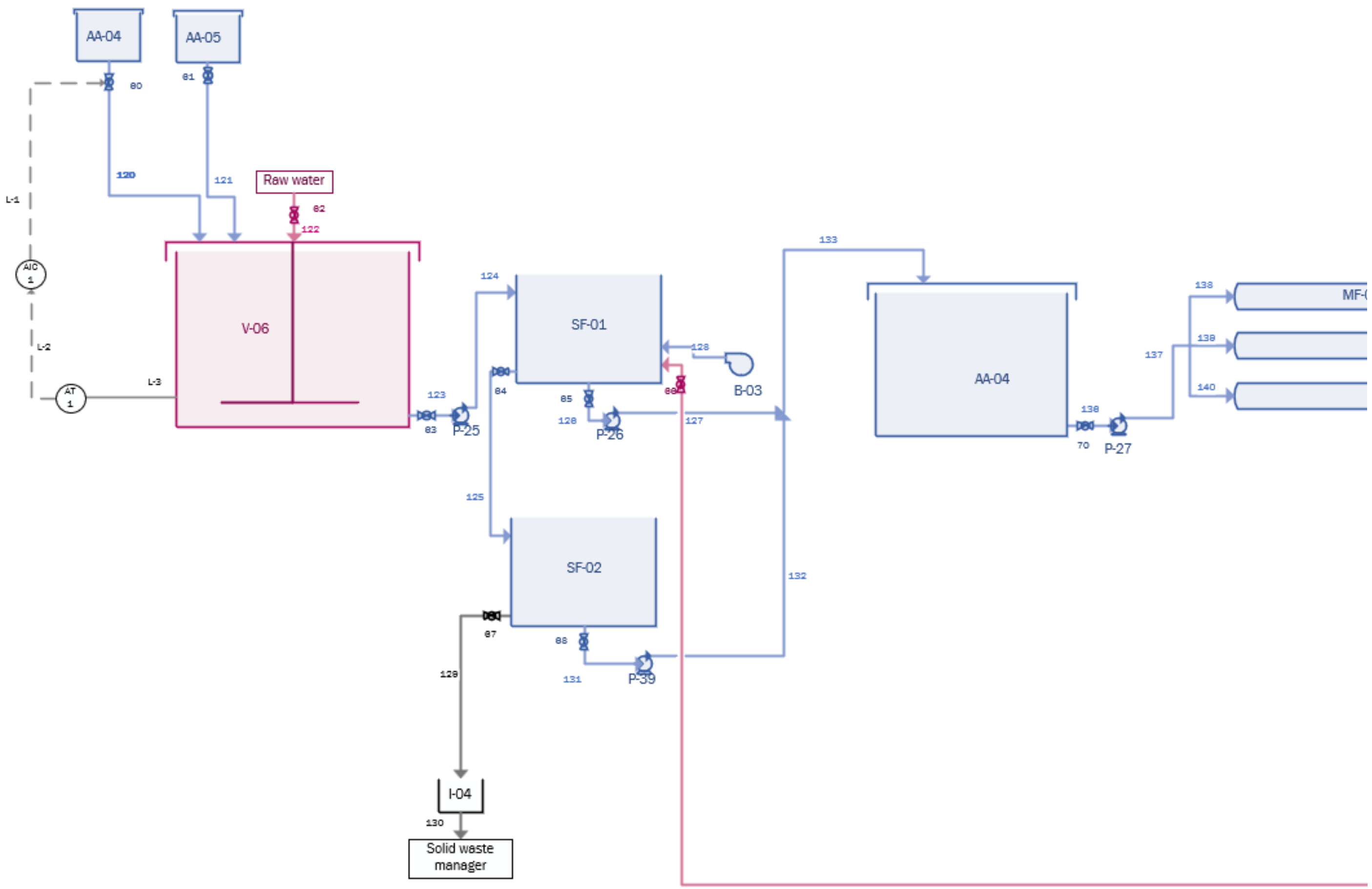
TAG	Equipment name
V-08	Water tank mixer
CL-01	Clarification tank
I-06	Impurities storage tank 6
MBBR-01	Moving bed biofilm reactor
MF-02	Microfiltration system 2
B-05	Blower 5
I-07	Impurities storage tank 7
AA-10	Regenerated water storage tank
AA-11	NaOCl storage tank 2
AA-12	HCl storage tank 3



PFD: WASTEWATER TREATMENT
Nº: 2/2
UNIT: WATER PROCESS

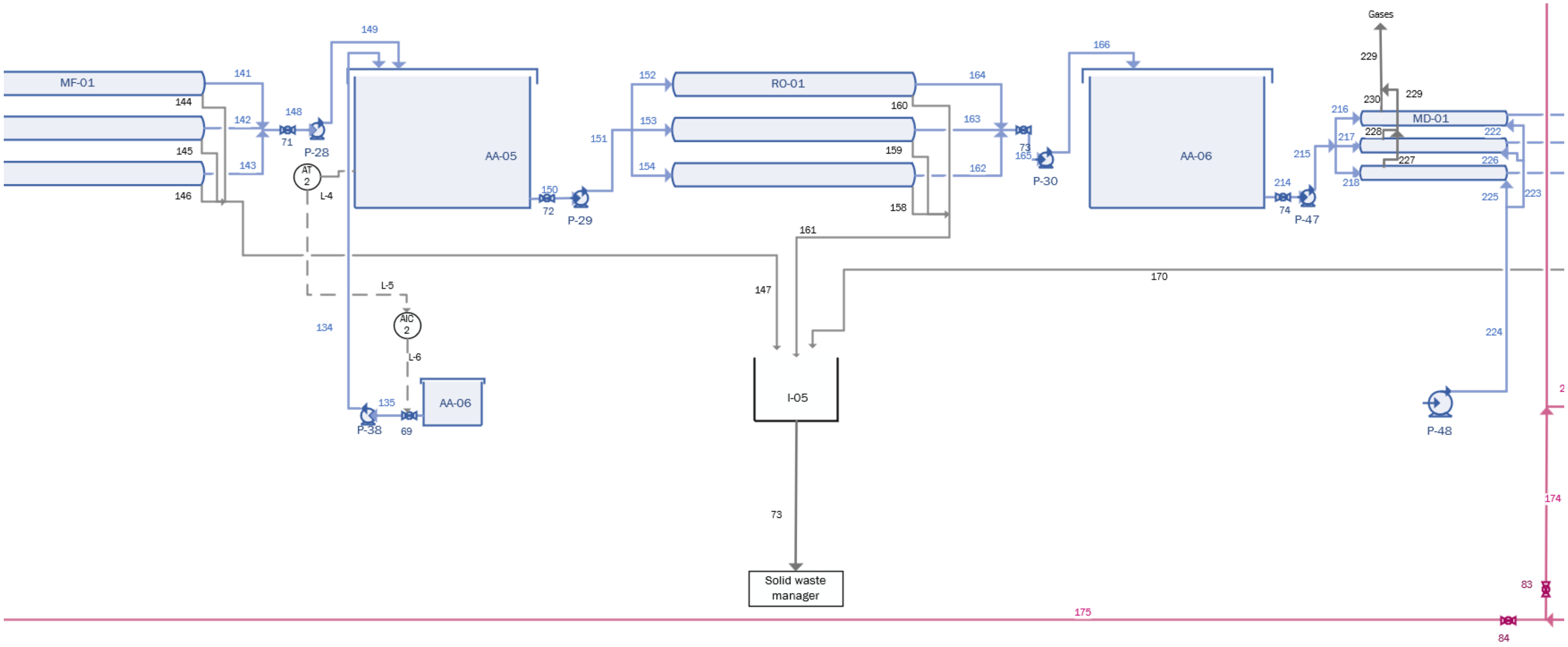


PFD: DEMINERALIZED WATER TREATMENT
Nº: 1/4
UNIT: WATER PROCESS



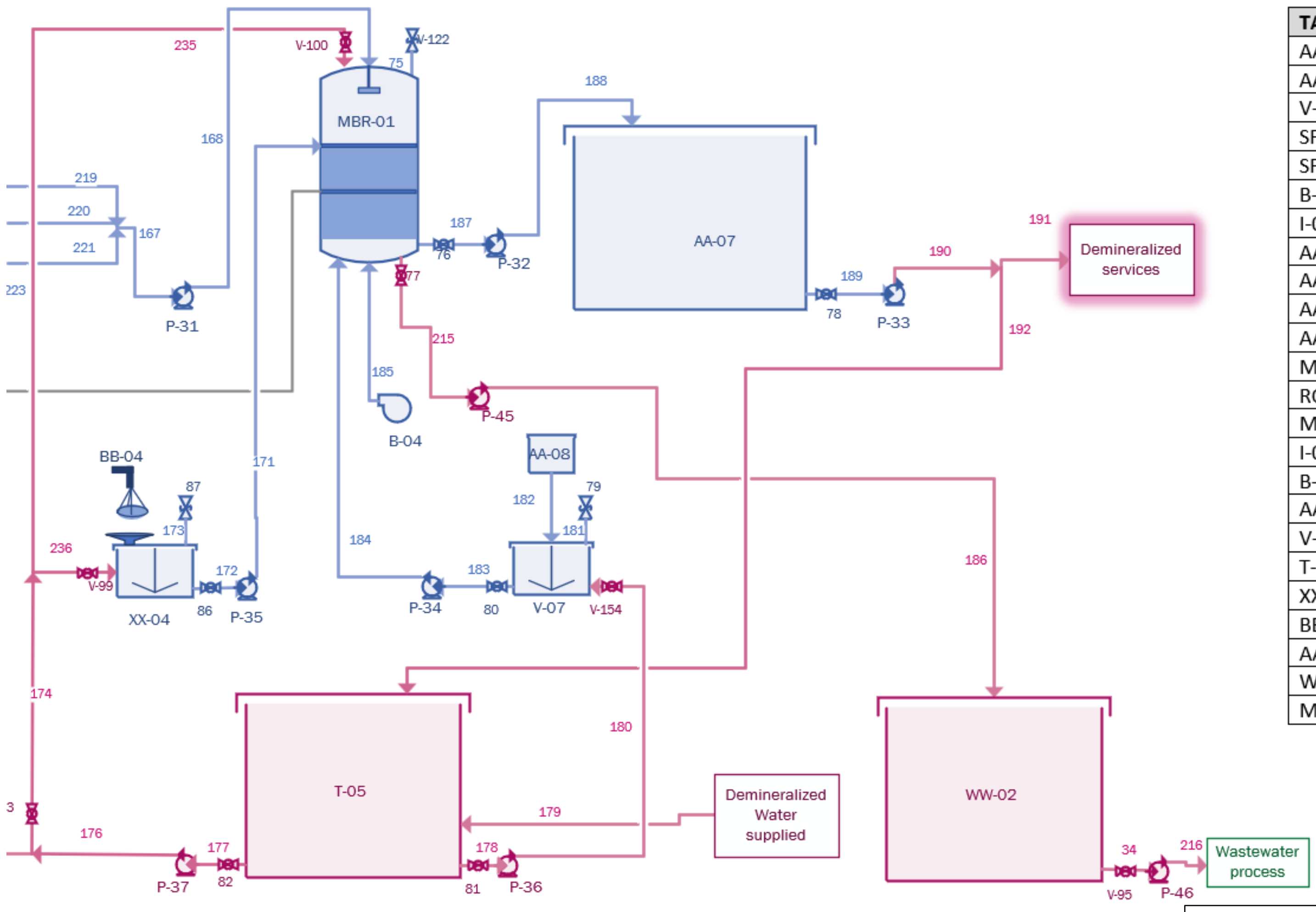
TAG	Equipment name
AA-04	HCl storage tank 1
AA-05	NaOCl storage tank
V-06	Water tank mixer
SF-01	Sand filtration tank 1
SF-02	Sand filtration tank 2
B-03	Blower 3
I-04	Impurities storage tank 4
AA-04	Water storage tank 1
AA-05	Water storage tank 2
AA-06	Water storage tank 3
AA-07	Water storage tank 4
MF-01	Microfiltration system 1
RO-01	Reverse osmosis system 1
MBR-01	Mixed bed resin 1
I-05	Impurities storage tank 5
B-04	Blower 4
AA-08	HCl storage tank 2
V-07	HCl dilution tank
T-05	Demineralized water tank 5
XX-04	NaOH dilution tank
BB-04	NaOH bag dispenser
AA-09	$NaHSO_3$ storage tank
WW-02	Wastewater tank 2
MD-01	Membrane degasification system

PFD: DEMINERALIZED WATER TREATMENT
Nº: 2/4
UNIT: WATER PROCESS



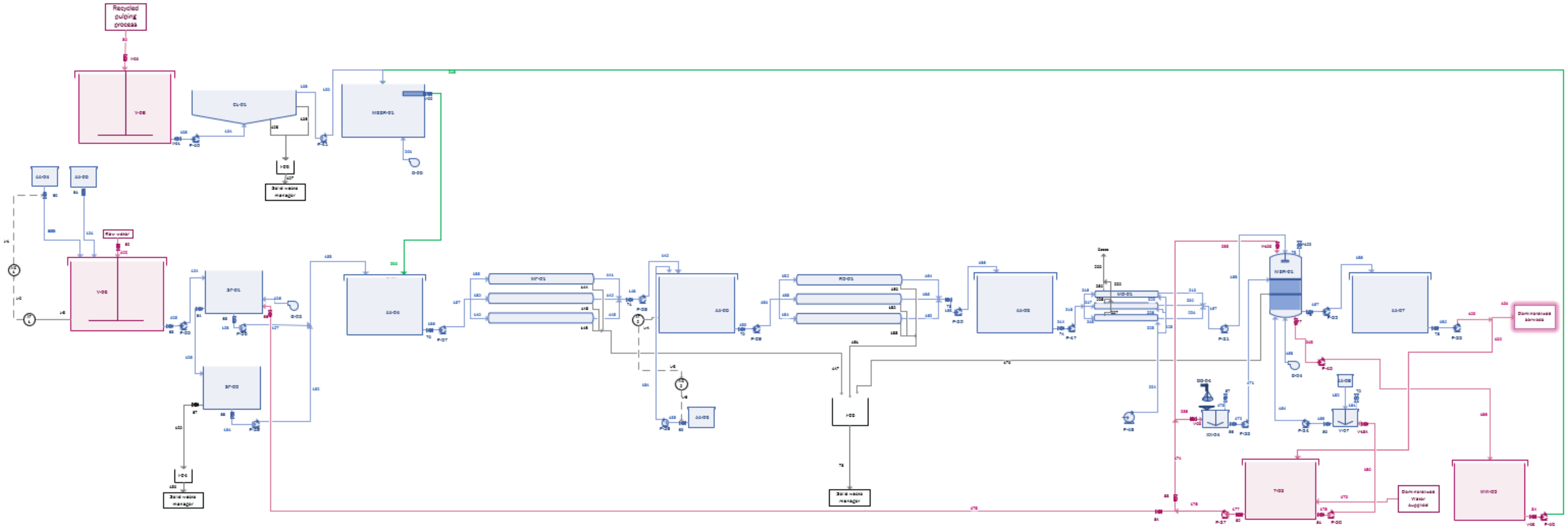
TAG	Equipment name	TAG	Equipment name	TAG	Equipment name
AA-04	HCl storage tank 1	AA-05	Water storage tank 2	AA-08	HCl storage tank 2
AA-05	NaOCl storage tank	AA-06	Water storage tank 3	V-07	HCl dilution tank
V-06	Water tank mixer	AA-07	Water storage tank 4	T-05	Demineralized water tank 5
SF-01	Sand filtration tank 1	MF-01	Microfiltration system 1	XX-04	NaOH dilution tank
SF-02	Sand filtration tank 2	RO-01	Reverse osmosis system 1	BB-04	NaOH bag dispenser
B-03	Blower 3	MBR-01	Mixed bed resin 1	AA-09	$NaHSO_3$ storage tank
I-04	Impurities storage tank 4	I-05	Impurities storage tank 5	WW-02	Wastewater tank 2
AA-04	Water storage tank 1	B-04	Blower 4	MD-01	Membrane degasification system

PFD: DEMINERALIZED WATER TREATMENT
Nº: 3/4
UNIT: WATER PROCESS

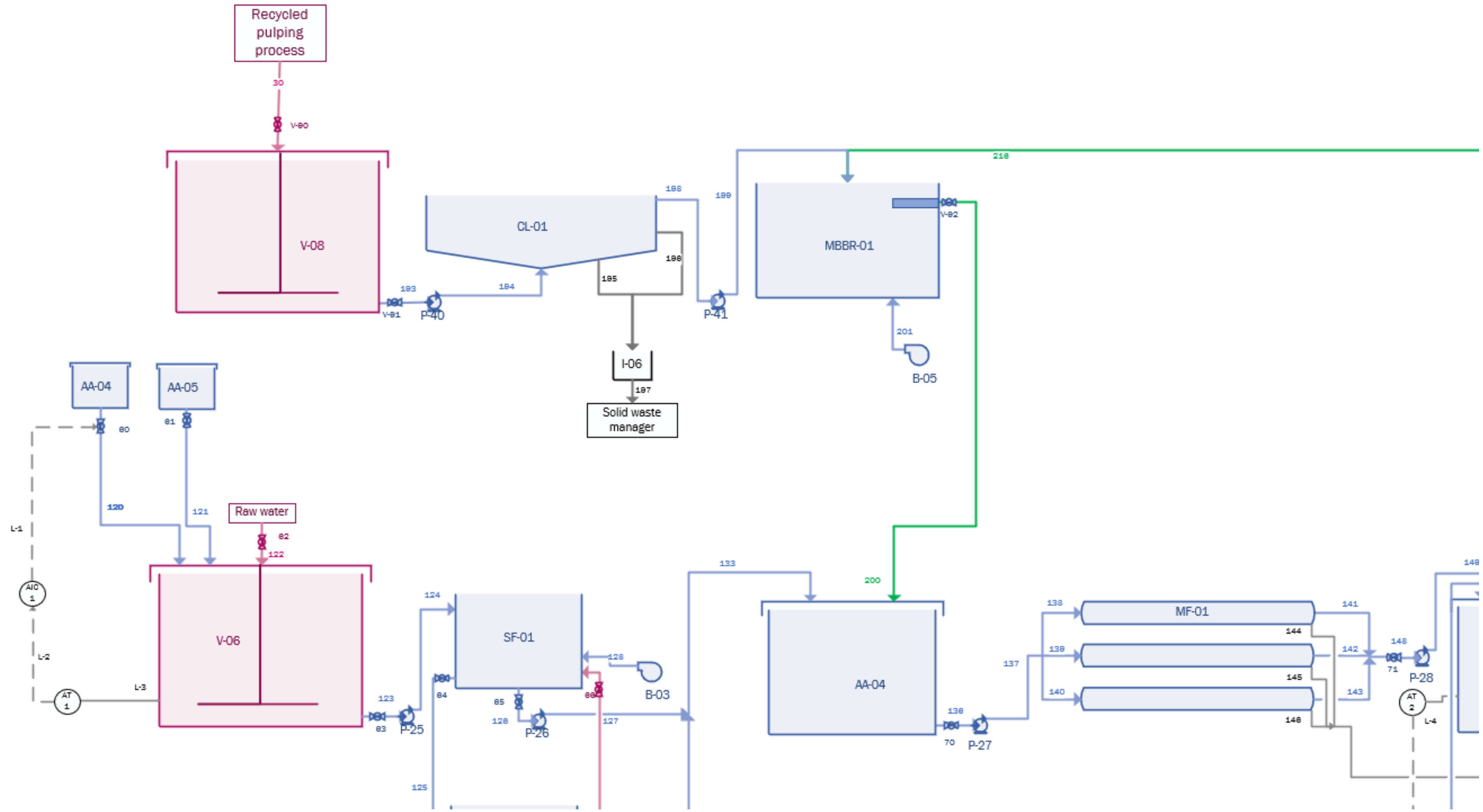


TAG	Equipment name
AA-04	HCl storage tank 1
AA-05	NaOCl storage tank
V-06	Water tank mixer
SF-01	Sand filtration tank 1
SF-02	Sand filtration tank 2
B-03	Blower 3
I-04	Impurities storage tank 4
AA-04	Water storage tank 1
AA-05	Water storage tank 2
AA-06	Water storage tank 3
AA-07	Water storage tank 4
MF-01	Microfiltration system 1
RO-01	Reverse osmosis system 1
MBR-01	Mixed bed resin 1
I-05	Impurities storage tank 5
B-04	Blower 4
AA-08	HCl storage tank 2
V-07	HCl dilution tank
T-05	Demineralized water tank 5
XX-04	NaOH dilution tank
BB-04	NaOH bag dispenser
AA-09	$NaHSO_3$ storage tank
WW-02	Wastewater tank 2
MD-01	Membrane degasification system

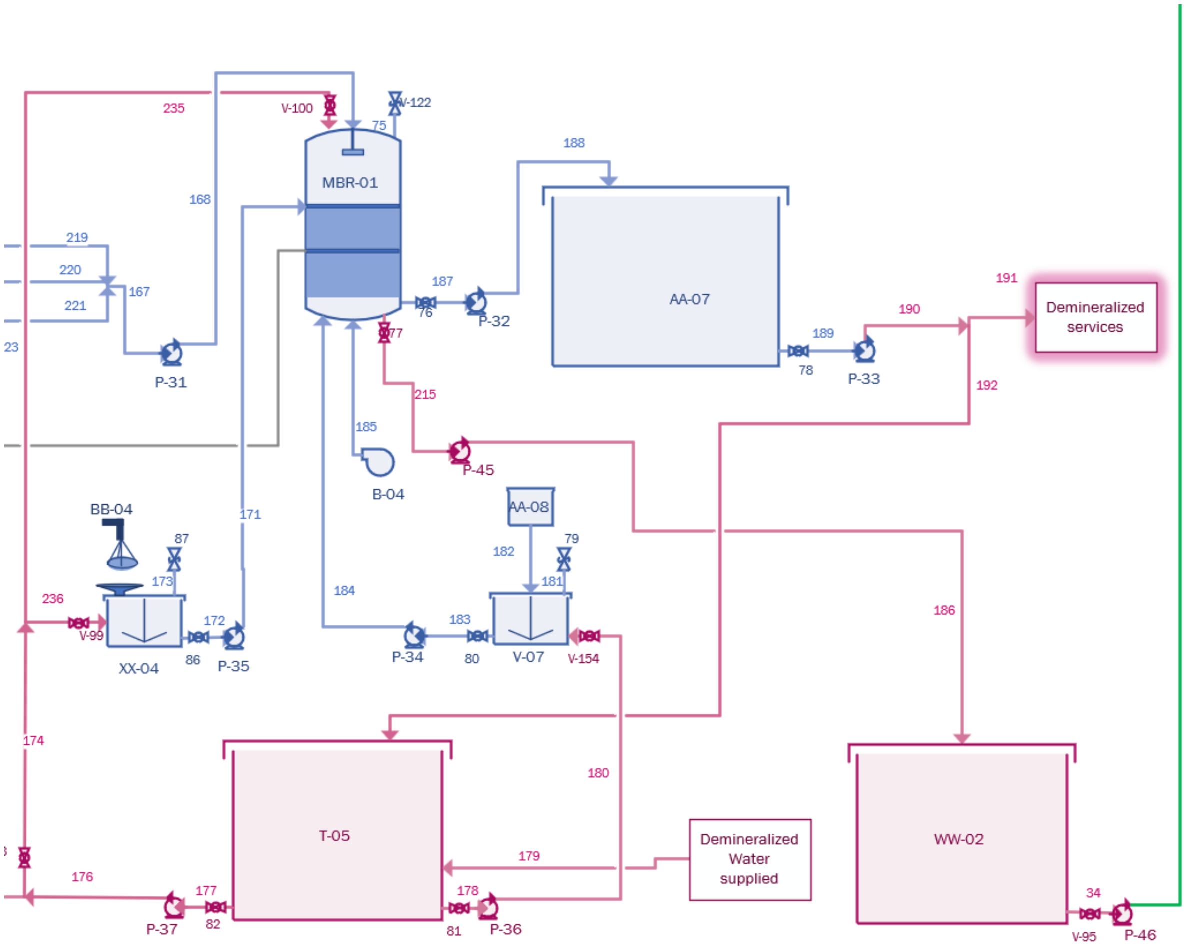
PFD: DEMINERALIZED WATER TREATMENT
Nº: 4/4
UNIT: WATER PROCESS



PFD: SUGGESTED WATER PROCESS
Nº: 1/3
UNIT: WATER PROCESS



PFD: SUGGESTED WATER PROCESS
Nº: 2/3
UNIT: WATER PROCESS



PFD: SUGGESTED WATER PROCESS
Nº: 3/3
UNIT: WATER PROCESS