



Treball Final de Grau

Coagulants for water purification: selection of products and design of the manufacturing process.

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Agrair al meu tutor, el doctor José María Gutiérrez González tota la ajuda facilitada al llarg del present treball. Els seus consells, així com les seves observacions, han sigut per mi d'un elevat valor, ja que m'han permès descobrir nous punts de vista i solucionar problemes sorgits al llarg de tot el treball. Crec que la seva àmplia experiència m'ha servit per aprendre molt al llarg del treball i aprofundir sobres certs conceptes que durant la carrera no havia pogut observar.

A més, i davant de la difícil situació viscuda durant els últims mesos, també voldria agrair-li el seguiment realitzat. Durant tot aquest temps ha estat pendent de mi en tot moment sense excepció, responent de forma ràpida tots es dubtes que li he fet arribar.

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SUMMARY

Coagulants are inorganic salts, mainly based on iron or aluminium, widely used in water treatment as colloids destabilizers in order to clarify and remove organics and suspended solids. Current work seeks to develop a production process for a new type of coagulant for water purification consisting on a polymerized blend of iron and aluminium chloride (PAFC).

Initially the study focuses on the characterization of a new third-generation coagulant, a blend salt of aluminium and iron, its mechanism of action as well as a comparison with the different current market options.

Later, the development of a new production process is detailed, including the determination of the necessary stages. At this point, among other aspects, the different alternatives available in reference to the process are studied, such as the raw material to be used or the needed unit operations. The new manufacturing process will mainly consist in 3 stages. The first stage will be the chemical reaction for dissolving iron and aluminium from bauxite tailings. The second stage will be the clarification of solved salts and separation of mud and suspended particles. Finally, the last stage will consist on the polymerization of the simple mixture of aluminium and ferric chloride in order to achieve a more complete product with better performance.

By last, following the guidelines established in the previous points, the sizing of the equipment for the new production plant has been carried out. This point includes both the sizing of the different equipment involved in the process and other aspects as design of the plant with the different control loops, the plant piping and the lay out.

The main characteristics of the designed plant are the implementation of vitrified reactors in order to cope with the high corrosion resulting from free HCl. Therefore, it will be necessary to consider this limitation throughout the manufacturing process, both in the design of the main equipment and the secondary elements such as pipes or pumps.

It has been also considered designing a continuous operation of the plant. As will be seen in detail both reactors and polymerizers will necessarily work by batch. Therefore, considering the

reaction times and the loading and unloading times, the plant has been designed as similar as possible to a continuous plant through the implementation of reactors in parallel.

The final product resulting from the new plant would be competitive within the current coagulants market and would provide better performance than classic coagulants.

Keywords: Coagulants, flocculants, Polyaluminum ferric chloride, PAFC, bauxite tailings, water purification.

RESUM

Els coagulants són sals inorgàniques, principalment a base de ferro o alumini, àmpliament utilitzades en el tractament de l'aigua com a desestabilitzadors de col·loides per tal d'aclarir i eliminar els orgànics i els sòlids en suspensió. Aquest treball pretén desenvolupar un procés de producció d'un nou tipus de coagulant per a la depuració d'aigües residuals que incorpori una barreja polimeritzada de clorurs de ferro i d'alumini (PAFC).

En una primera fase l'estudi es centra en la caracterització d'un nou coagulant de tercera generació, una sal mixta d'alumini i ferro, el seu mecanisme d'acció i una comparació amb les diferents opcions actuals del mercat.

Posteriorment, es detalla el desenvolupament del procés de producció del nou producte, incloent la determinació de les etapes necessàries. Arribats a aquest punt, entre altres aspectes, s'estudien les diferents alternatives disponibles en referència al procés, com la matèria primera a utilitzar o les operacions unitàries necessàries. El nou procés de fabricació constarà principalment de 3 etapes. La primera fase serà la reacció química per a la dissolució de ferro i alumini a partir dels residus de bauxita. La segona etapa serà la purificació del producte i la separació dels fangs i partícules en suspensió. Finalment, l'última etapa consistirà en la polimerització de la barreja d'alumini i clorur fèrric per aconseguir un producte més complet amb un millor rendiment.

Per últim, seguint les directrius establertes en els punts anteriors, s'ha realitzat el dimensionament dels equips per a la nova planta de producció. Aquest punt inclou tant la mida dels diferents equips implicats en el procés com altres aspectes com el disseny de la planta amb els diferents llaços de control, la canalització de la planta i la distribució i implementació dels equips.

Una de les principals característiques de la nova planta dissenyada serà l'ús de reactors vitrificats per tal d'afrontar l'alta corrosió que resulta del HCl lliure. Per tant, ha calgut tenir en

compte aquesta limitació durant tot el procés de fabricació, tant en el disseny dels equips principals com dels elements secundaris, com ara les canonades o les bombes.

També s'ha considerat dissenyar la planta per un funcionament en continu. Com es veurà, tant els reactors com els polimeritzadors funcionen necessàriament per càrregues. Per tant, tenint en compte els temps de reacció i els temps de càrrega i descàrrega, la planta s'ha dissenyat el més semblant possible a una planta en continu mitjançant la implementació de reactors en paral·lel.

Així doncs, com a resultat s'ha obtingut un producte final competitiu dins del mercat actual de coagulants, el qual proporcionaria un millor rendiment que els coagulants clàssics utilitzats actualment.

Paraules clau: Coagulants, floculants, Policlorur d'alumini i ferro, PAFC, bauxite tailings, tractament d'aigües.

1. INTRODUCTION

One of the first steps in water purification, both in drinking water production or sewage water treatment, is removal of colloidal organic matter and suspended solids through a physical-chemical treatment. Here, the use of coagulants is most useful and widely used as colloid destabilizers, allowing particles and organic colloids settling in a faster way and so reducing settling tanks required dimensions. This treatment has great advantages such as less sensitivity to variations in flow and composition, as well as great flexibility in the design of the plant and its possible adaptation according to the characteristics of the water. It can be applied to different effluents, studying in each case the product that provides the best results.

Due to the constant need to improve the quality of industrial wastewater over the last decades, the study of coagulation-flocculation has been deepened, as well as the use of new coagulants that allow improving the performance of the already existing. Therefore, new organic and inorganic coagulants have been studied to achieve a good removal of organic matter and solids in suspension in the purification processes of municipal and industrial wastewater.

The present study presents the manufacturing process of a new coagulant that leads to improve the performance of the products currently available on the market. In addition, it pretends to optimize costs and carbon footprint using a by-product from the alumina production process as a raw material.

Initially, current types of coagulants are described, as well as their mechanism of action. Subsequently, a new developed product is presented, pointing up its advantages and determining the production process it will require. Finally, the detailed design of a plant capable of producing the new compound in a viable way is carried out, which allows the production of a new competitive coagulant within the sector.

2. OBJECTIVES

Water purification uses a variety of coagulating agents (usually named as coagulants) that facilitate the separation of colloid and suspended solids and additionally phosphorous removal. These coagulants are usually inorganic salts, often combined with polymeric compounds as flocculants.

The objective of this work is the design of a coagulants manufacturing plant. Coagulant(s) will be selected through a bibliographic study. This study will be organized in different tasks in order to develop the most suitable design. Those tasks will be:

- Selection of coagulants to be manufactured through a bibliographic study. A wide range of products will be considered, evaluating raw material availability, performance and applications. This study should also allow select the production scale.
- Outline of the production process. Including selection of raw materials, and unitary processes.
- Basic design of the manufacturing plant. Through process synthesis techniques, the set of operations to be performed will be selected and the main equipment of the plant will be sized.
- Detailed design of the plant. Control systems and auxiliary equipment will be selected, and a spatial distribution of them will be established.

3. COAGULANTS AND COAGULATION PROCESS

The aim of chemical coagulation is destabilizing the colloids present in the water in order to aggregate them before sedimentation. Such colloids are difficult to separate in a natural settling process without the use of external coagulants and flocculants, due to their dimensions and the stable system that they form with the water molecules near them. This system produces a repulsion effect between the different colloids because of the potential that appears between them and the nearby water molecules, leading to a stable situation without any tendency to precipitate. (Lindquist, 2003)

The flocculation process is the mechanism by which small flocs aggregate in order to form larger flocs easy to precipitate in settling tanks. Industry usually uses polymeric flocculants to speed up the process of aggregating colloids and thus forming the precipitate.

This process involves organic flocculants. These compounds are synthetic structures of water-soluble polymers manufactured by free-radical polymerization of unsaturated monomers or by step-growth condensation reactions. In general, the most important are copolymers derived from acrylamide and a charged comonomer". (Kirk-Othmer, 2005)

Organic flocculants have a wider application field compared with inorganic ones due to their high specificity. In fact, we often use organic flocculants in such different processes as the purification of radioactive products such as uranium, the paper industry, oil and gas extraction or the coal industry. (Ullmann's, 2011)

On the other hand, in the treatment of most industrial and urban wastewater, as well as in drinking water treatment, polymeric flocculants need the previous action of classical coagulants to destabilize the colloids. Thus, classical coagulants still mean a key factor in water treatment and in the current inorganic chemical industry. Moreover, the consumption of aluminum sulfate (the oldest classic coagulant) is currently increasing its demand by 2-3% per year only in the field of water treatment. (Kirk-Othmer, 2005)

Additionally, inorganic coagulants are the main used tool to decrease the amount of dissolved phosphorous in wastewater, combined with biological treatment. As the European legislation in terms of phosphorous release to the natural water is becoming stronger, more and more quantities of inorganic coagulants are being used in sewage water treatment.

Therefore, due to the high existing demand for classic coagulants, the present TFG is focused on the manufacturing process of inorganic compounds instead of the manufacturing process of organic flocculants.

Finally, adding that much of the literature uses the generic term flocculants to cover the entire process when talking about coagulation and flocculation in water treatment. This expression specially appears in English literature, because English-speaking countries usually use this term in a generic way. (Lindquist, 2003)

3.1. ZETA POTENTIAL AND CHEMICAL COAGULATION

The colloid stability theory is explained by the double electrical layer theory, which is based on neutralization of the system because of opposite charged ions attraction. This theory says that the colloid-water system is divided into different zones with specific characteristics.

Colloids consist basically on proteins, starches and other carbohydrates, as well as on humic acid compounds. All these particles are charged negatively on this surface, defined by the Nernst potential. These charges attract ions of the opposite charge, some of them strongly bounded to the colloidal particle, forming a stable layer known as Stern layer (with a Stern potential). The rest of counter-ions are in a mobile layer, the diffuse layer or Gouy-Chapman layer. Inside this layer, we find a limit, below which all molecules remain attached to the colloid in their movement. That layer is known as limit layer, and it has the Zeta potential. (Company, 2000)

The existence of this Zeta potential and the double layer existing around the colloid prevents the approximation between the colloidal particles, avoiding that Brownian movement could allow the aggregation by the effect of the attractive forces of Van der Waals. Therefore, the chemical coagulation process consists in destabilizing the colloids system by the effect of highly-charged metal ions over the double layer, allowing the aggregation of small flocs in higher ones that can precipitate by themselves.

The chemical coagulation is usually carried out by the addition of aluminium or iron salts. Because of their high charge, the hydroxide complexes formed by metal ions in the water reduce the Zeta potential of the system and they can be adsorbed on the surface of the particles. (Company, 2000)

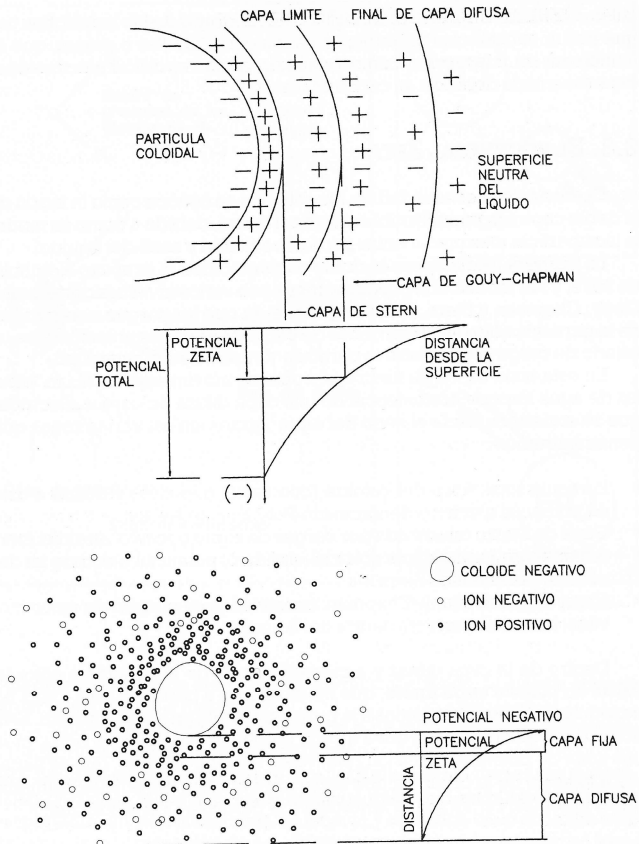
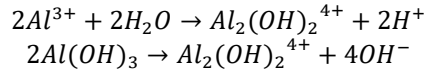


Figure 1. Zeta potential scheme.

(Figure from Company, 2000)

Aluminium and iron salts tend to hydrolyse in water forming the corresponding hydroxide. This is not a direct reaction, but intermediate hydrolysed forms and metal elements can be found simultaneously. In addition, all these compositions tend to form hexa-aquo complexes with short life but with high charge density that can enhance the coagulation process ($\text{Me}(\text{H}_2\text{O})_6^{3+}$). This complexation follows the next scheme:



This example shows a simple complexation reaction, but the resulting polymeric structures can have much more complicated formulas such as $Al_{13}O_4(OH)_{24}(H_2O)_{12}^{7+}$ or $Al_{13}(OH)_{24}(H_2O)_{24}^{15+}$. (Company, 2000)

These aquo complexes are stable under acidic pH conditions. Specifically, the resulting forms of Al^{3+} are stable at pH between 3-4 while the resulting forms of Fe^{3+} are stable at pH 1-2. By the time that hydroxo-aquo complexes are formed, the pH increases gradually and finally forms the hydroxides compounds. These are produced at pH greater than 5.5 / 6 in case of Al^{3+} while in for Fe^{3+} it will be at pH between 4.5 / 5. (Ullmann's, 2011).

Aluminium and iron salts usually use two different mechanisms for destabilizing colloid:

- Neutralisation of charge.

Adsorption of counter-ions on the surface of the particle, acting directly over the Zeta potential of the particles. If the added product is able to form positively charged species with charge density enough, the colloid may attract it and may even adsorb it on its surface. That process reduces considerably the colloid Zeta potential. (Patent, CN104973669A, 2015)

Trivalent salts such as aluminium or iron can achieve the charge density necessary to produce such effect. In fact, the critical coagulation concentration decreases approximately 30 times for each extra negative charge of the metal. Thus, the coagulation power of Al^{3+} is about 11 times greater than Ca^{2+} , and 730 times greater than Na^+ . Moreover, the relative ion size also affects, since smaller ones can be more easily adsorbed. (Company, 2000)

On the other hand, you should control not to overdose the salt. If so, the situation could reverse due to a reversal of the charges that would restore the stability of the system. (Ullmann's, 2011)

- Sweep coagulation.

This process occurs as a result of the flocs formation. When a polyaluminium ion is adsorbed to the surface of a particle or dissolved substance, additional hydroxides may start to grow from this point. A kind of mesh is so created, capable of trapping other colloidal particles

present in the solution and dragging them to the bottom of the tank. Therefore, the colloid precipitation process is maximized.

3.2. COAGULANT TYPES

As said, there are two main groups of coagulants, iron salts and aluminium salts. Following, a description of the most important (more commonly used) examples of these salts is reviewed.

3.2.1. Aluminium sulphate

This compound is commonly known by the name of Alum. When we add this compound to water it produces a hydrolysis reaction. The Al^{3+} forms an octahedral sp^3d^2 hybridization structure, where electrons from the water occupy free orbitals. After that, the complexation reactions are produced, forming polynuclear compounds through olation and oxolation reactions. Finally, electrostatic interaction and adsorption form the precipitated metal hydroxide and complete the coagulation process.

As explained, different species resulting from hydrolysis are generated in parallel. Specifically, the main hydrolysed species resulting from aluminium sulphate will be: $\text{Al}(\text{OH})_4^+$, $\text{Al}(\text{OH})_2^+$, Al^{3+} and $\text{Al}(\text{OH})_4^-$.

Aluminium sulphate is used both in urban wastewater treatment and in drinking water purification. This product, although it is the most widespread and oldest of all known coagulants, generates some problems that limit its range of applicability. First, it is difficult to treat certain wastewater due to their pH and the difficulty that this causes for the formation of the associated polymeric forms. The pH range of applicability of aluminium sulphate is between 6 and 8.5, which, as will be seen in the development of the other coagulants, is lower than others. (Company, 2000)

Moreover, the use of this coagulant decreases the pH of the system and some solved aluminium can remain in the water once the coagulation process finishes. That's because the solubility product of its hydroxide is 410g/kg, being more soluble than other inorganic coagulants. Therefore, their precipitation is more difficult. (Ullmann's, 2011)

On the other hand, aluminium sulphate gives a low residual coloration to the treated water. That's highly important when we are dealing with drinking water or water that will be disinfected by UV lamps before discharge or use (Patent, CN108675418A, 2018).

The residual dissolved aluminium amount in the treated water is controlled by the current legislation, especially in water for public consumption. In that case, the R.D. 140/2003 of February 7 establishes that the Maximum Admissible Concentration of the residual aluminium present in drinking water is 0,2 ppm. Moreover, it sets an operational recommendation level of 0.05 ppm. (Company, 2000)

3.2.2. Aluminium polychloride

Aluminium polyhydroxychlorides are aqueous solutions of aluminium hydroxychloride (polychlorinated aluminium hydrate) in polymeric form whose general formula is $(Al_n(OH)_mCl_{3n-m})_x$, where n represents the number of aluminium atoms in the polymer chain while x is the polymerization index (around 15). This type of compound is commonly known as PAC.

Aluminium polychloride is classified as a second-generation coagulant. This is because they are a prepolymerized form, containing hydroxylated polymer chains, which gives better performance compared to first-generation coagulants such as aluminium sulphate or iron salts. This means that these high charged chains should not be generated within the coagulation process. (Ullmann's, 2011).

The most important hydroxylated forms in water are:

- Monomers: Al^{3+} , $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al(OH)_3$, $Al(OH)_4^-$
- Polymers: $Al_2(OH)_2^{4+}$, $Al_3(HO)_4^{5+}$, $Al_{13}O_4(OH)_{24}^{7+}$

There are mainly two compound types that we call aluminium polychloride: aluminium polyhydroxychlorides (with an Al_2O_3 content between 13.5% and 18%) and aluminium polyhydroxychlorosulphate (with an Al_2O_3 content of 10%) that includes sulphates in the molecule. (Company, 2000)

These polymeric molecules undergo a process of hydrolysis in aqueous medium through a slower kinetics than aluminium sulphate. That effect produces new insoluble species that are also less sensitive to possible changes in the environment (pH, alkalinity, temperature...). In this way, the fact of having a coagulation rate higher than aluminium sulphate and other inorganic products causes compact and easily settling flocs at low temperatures. (Ullmann's, 2011)

Aluminium polyhydroxychlorosulphate is usually used in the treatment of drinking water while in wastewater or heavily polluted water we rather use aluminium polyhydroxychlorides.

The relative independence of the pH where the hydrolysis reaction occurs gives to PAC a wide range of use. Specifically, the pH values at which said reaction occurs can range from 5 to 10, the field of applicability being considerably wider than for aluminium sulphate. In addition, these products has a higher performance at lower dosage, leading to a lower reduction in the pH of the treated water. Moreover, it's possible to use higher doses of the product, necessary in the treatment of heavily polluted waters, without residual turbidity due to overdosing. (Company, 2000)

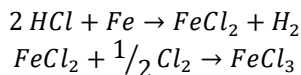
Thus, unlike aluminium sulphate, PAC exhibits excellent behaviour in the treatment of both urban and industrial wastewater, becoming a clear alternative to iron salts for such cases. In addition, this product also achieves good results in mixed plants, where physicochemical treatment is combined with a biological treatment, since it protects the biological treatment.

3.2.3. Ferric chloride

Ferric chloride is a widely used inorganic coagulant in the treatment of both urban and industrial wastewater. The demand for iron chloride is increasing in the United States with values between 3% and 4% per year, since it has been shown that in certain fields it obtains better results than aluminium sulphate (the oldest and most used classical coagulant), while it's cheaper than PAC. (Kirk-Othmer, 2005).

The product is usually obtained as a result of the oxidation of ferrous chloride ($FeCl_2$) by the action of liquid chlorine (Cl_2) or another strong oxidant. On an industrial level, the raw material used may have different origins: ferrous chloride can be extracted from oxidized iron or from the treatment of ilmenite, but its most common origin is as a byproduct of the steel pickling process. (Ullmann's, 2011)

Thus, the reactions that take place in the production of ferric chloride are the following:



In the coagulation process, ferric chloride is hydrolysed in water to form precipitated ferric hydroxide through successive hydrolysis reactions, originating different ionic forms: Fe^{3+} , $Fe(OH)^{2+}$, $Fe(OH)_2^+$, $Fe_2(OH)_2^{4+}$, $Fe(OH)_3$, $Fe(OH)_4$, $Fe(OH)_6^{3-}$. (Company, 2000)

Coagulation with ferric chloride occurs from pH 5.5 on, without danger of redissolution of the precipitated hydroxide at high pH, as in the case of aluminium. The formation of the precipitate is finally reached after the generation of the successive previous ferroxylated

hydroxocomplexes, which polymerize through olation (dehydration) and oxolation reactions (loss of protons that causes pH decrease). These reactions are reversible, but the generated complexes are of relatively stability depending on the pH in which they are found. (Company, 2000)

So ferric chloride has a fairly wide field of applicability, which gives it an advantage over other inorganic coagulants. Optimal coagulation pH goes from 5 to 10, this range being greater than the case of aluminium sulphate. In addition, the solubility product of the associated hydroxide is greater, so that the final product will be less soluble in water, preventing iron residues from persisting in the treated water. (Company, 2000)

Additionally, since iron has a higher molecular weight, the formed floc will be denser than those obtained with aluminium salts. That leads to a faster decantation and greater sludge compactness. (Lindquist, 2003)

Finally, ferric chloride, since it's usually produced from by-products, has a lower cost than most aluminium salts.

3.2.4. Ferric sulphate

Ferric sulphate is another of the most commonly used inorganic coagulants in today's industry. Like ferric chloride, it is often used in wastewater treatment, although in certain countries (e.g. Great Britain) it's widely used as a coagulating agent in drinking water. (Kirk-Othmer, 2005)

The product is obtained by oxidation of the ferrous sulphate heptahydrate, coming in most cases as a by-product from the production process of TiO_2 (titanium dioxide) pigments. In this process, ilmenite, a mineral rich in iron and titanium, is attacked with acid (mostly sulfuric acid). The iron is then removed from the liquor obtained by crystallization as ferrous sulphate. Ferrous sulphate attacked with sulfuric acid oxidizes to ferric sulphate. (Company, 2000)

Ferric sulphate has a very similar use to ferric chloride, since both are coagulants with iron III as a metallic element. Additional benefit of ferric sulphate comes from a wider pH range (between pH 4.5 and 12), than ferric chloride or aluminium sulphate. As a result, it is very useful in cases where it is necessary to work at very high pH such as the treatment of water with high manganese or heavy metals contents. (Company, 2000)

Moreover, like ferric chloride, ferric sulphate forms flocs of great density and compactness, so it is very useful in the treatment of water with a high content of suspended particles.

3.2.5. New coagulants

So far, the four most frequent coagulants have been described, consisting of two main metallic elements, aluminium or iron. Currently, however, a new type of coagulants known as mixed salts is being developed, including both metallic elements into its formula. These products are able of taking advantage of the synergies between them, leading to a higher performance in most of the cases. (Patent, CN103864187A, 2014)

The current trend is the introduction of this type of coagulants in the replacement of the classic coagulants that allow improving the performance and the observed results. There are currently two types of product groups with these characteristics, polychlorides and polysulphates with different characteristics as well as with a different range of applicability.

Specifically, this document will focus on the development of a production process for polyaluminum ferric chloride (PAFC). The choice of a polychloride above a polysulphate product is based on different observed characteristics:

- Applicability limitations. Polysulphates, as with simple sulphates, have a lower applicability range than chlorides. Specifically, polysulphates generate problems in the treatment of wastewater due to the appearance of hydrogen sulfide in the anoxic phases of the process, as digestion or sludge dewatering. (Company, 2000)

On the opposite, PAFC can be applied in a wide variety of purification processes, mainly due to the possibility of acting with a very wide pH range (4-11). Additionally, it doesn't promote the generation of H_2S , on the contrary, it removes sulphur from the water system in form of ferric sulphur, controlling the high corrosion effect of H_2S over facilities and workers health. (Patent, CN106395917A, 2017)

- Complex application process. The production process of polymeric aluminium ferric sulphate is a very complex process due to the problems that the mixture between both sulphates presents. The process is difficult to reproduce on an industrial scale, so it is currently only applied on a small scale and in very specific purification processes. (Patent, CN108675418A, 2018)

On the contrary, the production process of mixed polychloride is simpler, which allows production on a large scale to generate viable product quantities at an economical profitability level. In this case, the synthesis of the mixed salts is similar to that of each of the salts individually, so the main technical challenge lies in the polymerization of the final product. (Patent, CN102774889A, 2012)

- Difficult polymerization. Unlike chloride, where a lot of aluminum polychlorides are developed and well known, sulphates present significant difficulties for polymerization. That's very important, since a polymerized final product provides advantages to the rate of formation of the floc as well as the pH working range. (Patent, CN104973669A, 2015)

All these reasons lead to develop the manufacturing process of a mixed iron and aluminum polychloride. The product will allow to obtain the advantages of the existing synergies between both ions, and at the same time it can be presented as a polymerized final product.

Compared to conventional PAC, the new product gives important advantages in terms of applicability and functionality. Specifically, the main advantages are the following:

- Lower degree of turbidity of the water once treated. (Patent, CN207756141U, 2018)
- Better reduction of COD, BOD and heavy metals. (Patent, CN106395917A, 2017)
- Fast solubility (1 millisecond) and therefore acceleration of the coagulation and sedimentation process. (Patent, CN106395917A, 2017)
- Higher working pH range and therefore less chemical agents to adjust the pH of the solution. (Patent, CN207756141U, 2018)
- Lower residual aluminum contents in treated water. (Patent, CN207756141U, 2018)

For all the aforementioned, PAFC has been selected as the coagulant to be manufactured, instead it is not widely used but still in the testing and development phase, because it has substantial advantages over the rest of coagulants. Furthermore, in the future it is expected that this coagulant will be able to largely replace the classic coagulants currently used.

4. PAFC MANUFACTURING PROCESS

Current section develops the production process of polyaluminum ferric chloride (PAFC), detailing each of the phases as well as the products involved in each of them. In addition, the choice of the selected process as well as the raw materials used in the process will be justified.

Add that this project aims to be as sustainable and ecological as possible. Environmental aspects require special attention when designing the facilities. Even, using by-products in the production of PAFC is an added value from environmental and profitability point of view.

4.1. RAW MATERIAL SELECTION

The first important decision to be made prior to the design of the industrial installation has been the choice of the raw material, as well as the methodology to be used in the production process. Currently, the production of both ferric chloride and PAC can be done in different ways, using different raw materials in each case and therefore different reactions. For this reason, the present possibilities have been analyzed and an assessment of each one of them has been carried out in order to select the more ecological and profitable option.

The first source of aluminum considered has been aluminum hydrate ($\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$). This product is currently used as the usual raw material for PAC production at an industrial level. Hydrate is a final product easily obtainable in the market in large quantities. It is obtained from the alumina purification process and is used in many industries. (Company, 2000)

However, this product is not a byproduct of any industrial process, but a final product, so we would not be achieving one of the objectives of the approach, which lies in outputting by-products from other industries to minimize the ecological impact generated. In addition, being a refined product, it does not contain iron in its composition, so an alternative source of iron would be required.

A considered alternative is obtaining the aluminum hydroxide as a byproduct of some of the processes carried out in the aluminum industry. Different by-products have been found in the

literature that contain a large amount of $\text{Al}(\text{OH})_3$ in their final composition. Unfortunately, the quantity that can be obtained of these materials is relatively low, so we couldn't cover expected market demand. In addition, these aluminum industry by-products usually contain some undesirable heavy metals, so the process should include some removing step for these heavy metals. (Patent CN102774889A, 2012)

A reference to blast furnace dust (BFD) has also been found in the literature as an alternative source of both aluminum and iron. BDF is a solid waste produced by the iron and steel industries with a general composition of iron oxides (approximately 50%) and small amounts of refractory oxides such as aluminum, silicon, calcium and magnesium. Now, producing coagulants from BDF is difficult to achieve at an industrial level, so its use for our PAFC production plant has been rejected. (Yanli et al., 2015)

Regarding the iron source of the process, there are some viable options in the market. The main sources of iron for ferric chloride production are scrap iron (obtained as retails of steel coils transformation) and mill scales (by-product from mechanical pickling of steel coils), since they are easy to obtain by-products. Other usual iron source found is the spent FeCl_2 liquor (SPL) coming from the pickling process with HCl in steel industry. Their iron content is (8-14% Fe^{+++}) but is a liquid form, easy to include in the process at different stages. Nevertheless, none of these by-products contain aluminum in their composition, so it would require a source of aluminum to develop a mixed salt. (Patent, CN108675418A, 2018)

An also considered aspect in the choice of raw material has been the need for chlorine. A large part of the industrial processes observed in the literature have this product in some of its stages. Since chlorine is a highly demanded element by the chemical industry, it has a high cost. In addition, it's a product with limited availability in our market and a high seasonal demand (conversion to sodium hypochlorite during summer seasonal demand), so our plant could be affected by chlorine market shifts. Also, chlorine is very dangerous, difficult to handle and usually induces reluctances and limitations in local authorities. (Lindquist, 2003)

Considering above mentioned aspects, bauxite tailings has finally been chosen as the raw material for the process. This is one of the solid products generated in the alumina production process. It represents a by-product easy to find in large quantities, and additionally it provides in the same raw material both the iron and aluminum needed to produce our mixed salt. This by-product, unlike conventional bauxite, does contain an enough percentage of iron by itself to

produce our coagulant, without any need for adding a second source of iron, which makes it possible to speed up the manufacturing process.

Even so, it should be considered that the PAFC production process from bauxite tailings produces sludge as a result of the presence of SiO_2 in its composition, so the process will require an intermediate stage of decantation to eliminate said sludge. (Dongyang et al., 2013)

Specifically, the composition of this raw material will be as follows:

Table 1. Bauxite tailings composition

Compound	wt. %
Al_2O_3	49,87
SiO_2	22,45
Fe_2O_3	13,1
TiO_2	2,61
K_2O	2,25

In order to calculate the balances, which will be presented in the following section, it was necessary to define the molar mass of the bauxite tailings. This calculation has resulted in a molar mass value of 98.95 g/mol and this value will be used in the following sections.

Moreover, the second major chemical compound necessary for the industrial process of PAFC synthesis will be hydrochloric acid. This will act as a source of chlorine in the absence of Cl_2 and it will help us to regulate the pH of the solution, keeping it in an optimal working condition. The concentration of HCl will be determined by the purity of its commercial forms. Therefore, it has been considered that the HCl content will be between 31% and 33%, because these are the most common market values. In this way, it will not be necessary to pre-treat the HCl. (Aznar, 1997)

Finally, in addition to these two compounds, a third essential element, calcium aluminate, appears in the synthesis process of PAFC. This, as will be detailed later in the present work, will be essential to carry out correctly the polymerization of the final product. Moreover, this reaction produce an extra AlCl_3 quantity. (Dongyang et al., 2013)

4.2. MANUFACTURING PROCESS SYNTHESIS

The PAFC production process has three basic elements such as the reactor, the decanter and the polymerization of the solution. (Dongyang et al., 2013)

The next figure shows the flow diagram of the process, which will be described in this section.

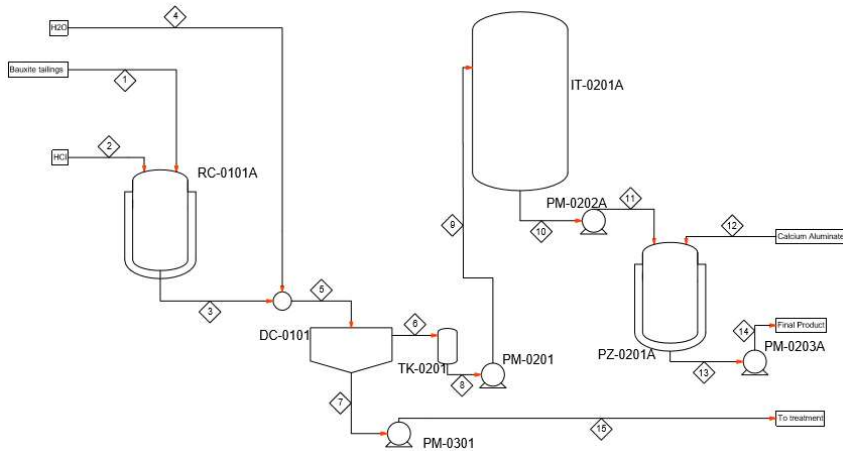
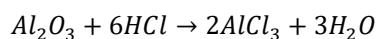


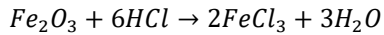
Figure 2. PAFC flow diagram.

In order to explain in deep the PAFC synthesis process, the installation has been divided into 3 sections. The first refers to the synthesis of $AlCl_3$ and $FeCl_3$, including the dissolving process and the liquid-solid separation in the settling tank. In this section we still find an important number of solid particles in suspension from bauxite tailings. The second section includes the polymerization of the product, the entire process from the decanter, using clarified liquor. This phase does not include suspended mud particles, because they have been removed through the decanter. Finally, section 3 refers to the rejected solids leaving the decanter as a waste, as well as its later treatment.

4.2.1. Reaction

The first step in PAFC production is aimed at the formation of ferric chloride and aluminum chloride. This step is performed inside a reactor where solid bauxite tailings react with liquid HCl. This process pretends to dissolve the maximum as possible Al_2O_3 and Fe_2O_3 with HCl. Specifically, the reactions that will take place inside the reactor are the following:





Consequently two distinct solid-liquid phases will appear. The liquid phase will contain the mixing of both chlorides with the residual HCl of the process, while the solid phase will contain mostly SiO₂ along with the other not reacting elements of the residual bauxite tailings.

As can be deduced from the reactions, the reactor should be able to work in a very acidic medium, which will require working with a vitrified type reactor. This type of reactors are able to withstand very low pH preventing the deterioration of the equipment. In addition, the reactor must have a thermal jacket able to keep the reaction temperature between 75°C-100°C. (Dongyang et al., 2013)

4.2.2. Decantation

Before decanting, it will be necessary to add water to the reactor outlet stream. This stream has a high content of Al³⁺, and if it has a concentration greater than 9% the aluminum may crystallize. This might be problematic in the decantation stage of the process if it appears because we may lose product in the mud. As a result, this effect would produce a substantial decrease in process performance. (Lindquist, 2003)

As explained, the second stage of the PAFC production process is the decantation of SiO₂ sludge. This stage aims to separate the two phases present at the outlet of the reactor by settling. Thus, a clarified solution of AlCl₃ and FeCl₃ free of solid bauxite tailings waste is achieved together with the residual HCl.

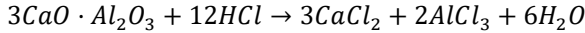
4.2.3. Polymerization

Prior to polymerization it will be necessary to consider an important concept. The polymerizers are reactors that work by batch, while the previous stage, the decanter, works in continuous. Therefore, it will be necessary to size and design an intermediate tank capable of storing the clarified product from the decanter and distribute it among the different polymerizers. In addition, it must be able to store product in the event of shutdown or maintenance of any of the reactors.

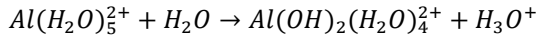
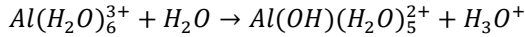
The last step of the PAFC production process is the polymerization of the resulting solution. This process consists of hydrolyzing the molecules with the objective of forming an hydroxyl polymer of Al and Fe.

In order to produce hydrolyzed forms correctly, the excess of acid present in the solution must be counteracted, otherwise the only produced forms will be $Al(H_2O)_6^{3+}$ and $Fe(H_2O)_6^{3+}$. To neutralize the acidic medium calcium aluminate ($CaO \cdot Al_2O_3$, $CaO \cdot 2Al_2O_3$) will be used, so that the pH of the solution increases and polymerized and copolymerized can be formed. The more basic the medium, the greater the degree of polymerization of Fe^{3+} and Al^{3+} . (Aznar, 1997)

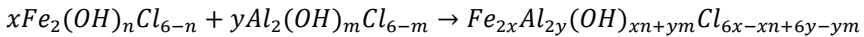
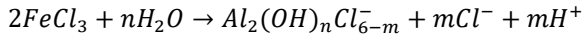
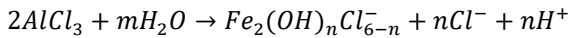
Specifically, the neutralization reactions that occur when adding calcium aluminate to the mixture will be the following:



Thus, as the medium is basified, different hydrolyzed forms appear as the example shown below.



Consequently, the overall process for both aluminium and iron will be as follows:



At the end of the polymerization process, most of Al and Fe will be in polymeric form as PAFC. (Dongyang et al., 2013)

5. PLANT DESIGN AND SIZING

Within this section, the detailed sizing of the entire installation will be carried out, including the individual sizing of each involved equipment.

5.1. BALANCE CALCULATION

First of all, a production of 2 m³/h of FeCl₃ has been used as a calculation basis in order to size and design the full process. This value is based on data obtained from the sector with the aim of dimensioning an installation with a production capacity comparable to a FeCl₃ production plant located in Tarragona, which represents the biggest coagulant production plant in Spain. (Lindquist, 2003)

As it was described previously the process includes 3 principal stages and those effect the balance calculation. The main aspects to consider are described below.

- **Reactor.** It has a HCl mole fraction of 0.84, the rest being bauxite tailings. Furthermore, it is assumed that the conversion of Fe₂O₃ and Al₂O₃ will work with an excess of HCl and will be dissolved 100%, although not all the raw material present in the bauxite tailings will be able to dissolve. It has been accepted that the limitation percentage for Fe₂O₃ will be 95%, while for Al₂O₃ it will be 65%. This value will be important because, even if a 100% conversion is carried out due to excess HCl, not all the raw material will react in the reactor. (Dongyang et al., 2013)
- **Decanter.** The operation of the decanter has been considered as ideal. Therefore, it has been established that all the bauxite tailings mud (red mud) is extracted through the bottom of the decanter. By the top of the settling tank just the aqueous full clarified phase is extracted, which contains the remaining HCl together with AlCl₃, FeCl₃ and water.

- Polymerizer. It has been considered that the HCl reacts completely when adding stoichiometric amounts of calcium aluminate, which will allow the polymerization process to be carried out.

For the correct understanding of the process and description of the different streams present in the process, the material balances are presented below.

Table 2. Stream material balance.

[kg/h]	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
HCl	0	22692	8273	0	8273	8273	0	8273	8273	8273	8273	0	0	0	0
Fe ₂ O ₃	1515	0	75	0	75	0	75	0	0	0	0	0	0	0	75
Al ₂ O ₃	5798	0	2029	0	2029	0	2029	0	0	0	0	0	0	0	2029
SiO ₂	2629	0	2629	0	2629	0	2629	0	0	0	0	0	0	0	2629
AlCl ₃	0	0	9864	0	9864	9864	0	9864	9864	9864	9864	0	10972	10972	0
FeCl ₃	0	0	2928	0	2928	2928	0	2928	2928	2928	2928	0	2928	2928	0
H ₂ O	0	0	0	49743	49743	7146	42597	7146	7146	7146	7146	0	7146	7146	42597
Calcium Aluminate	0	0	0	0	0	0	0	0	0	0	0	1123	0	0	0
CaCl ₂	0	0	0	0	0	0	0	0	0	0	0	0	1384	1384	0
Total	9942	22692	25798	49743	75541	28211	47330	28211	28211	28211	28211	1123	22430	22430	47330

Regarding the final product, PAFC, the present project allows the production of a mixed product of FeCl₃ and AlCl₃, which means obtaining an aqueous solution of aluminium and iron hydroxychloride in polymeric form. It should be noted that, in the industrial sector of water treatment, the purity criterion and the viability of the product as a coagulant is measured by the aluminium and iron content of the final product. In this case the final content of aluminium is 9.7%, while that of iron is 4.5%. These values are acceptable from a technical point of view and give to PAFC the possibility of competing with the current products on the market. (Company, 2000)

In addition, the limiting factor in that product is the aluminium content, which should be around 9%. If this value is bigger, aluminium may precipitates, generating destabilization problems on the mixture and affecting its use in water treatment plants.

Finally, the next page presents the PFD of the plant.

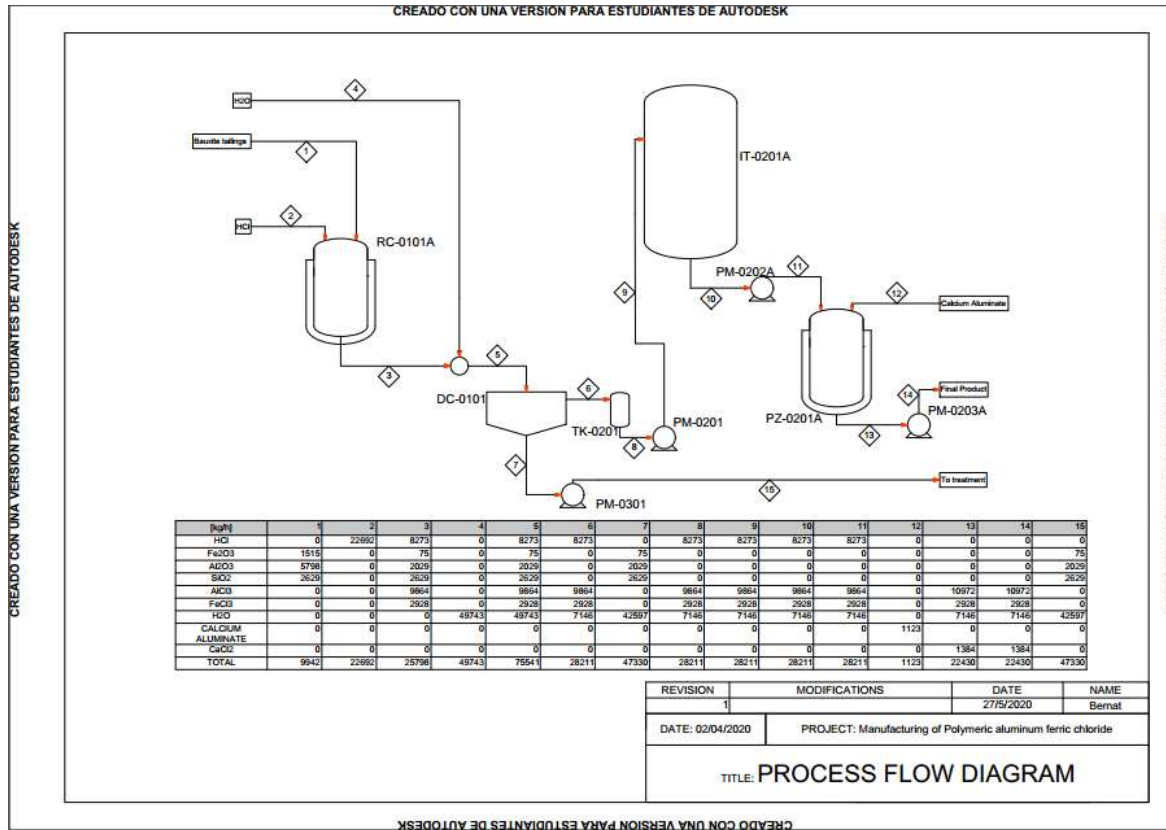


Figure 3. PAFC PFD.

5.2. EQUIPMENT SIZING

Next, we will describe the dimensioning of the different equipment included in the production process for polymeric aluminium ferric chloride. The sizing of all the equipment has been carried out to scale from the different bibliography consulted, which has allowed me to adapt the different equipment to the needs of my industrial process.

5.2.1. Reactor.

The reactor is the main element of the plant, because it carries out the transformation of the raw material. In this case, the raw material will be bauxite tailings, which as described above represent an important by-product within the alumina production process. (Dongyang et al., 2013) Thus, in the reactor will take place the transformation of Al_2O_3 and Fe_2O_3 present in the raw material into liquid AlCl_3 and FeCl_3 through the use of HCl.

The first consideration is to determine the type of reactor required. In this case, we can calculate the amount of HCl necessary to carry out the reaction analysing the different compounds that participate in it. As shown in Table 2, 621 kmol/h of HCl 32% (22692 Kg/h) is needed, which implies an acidic medium with very low pH.

Facing these low pH conditions, the most effective solution in coagulant production processes is using vitrified reactors. They are stirred tanks type reactors with an internal glass coating. It should be noted that these vitrified reactors work discontinuously, so the first phase of the process must work discontinuously. (Stendahl, 1990)

In order to size the reactor, a real reactor from PAC production will be scaled with the operating conditions of this process. As it has been presented in the previous section, the inlet flow rate of the reactor is $15 \text{ m}^3/\text{h}$ while, according to the bibliography consulted, the residence time should be 3 hours. The residence time has been established with the purpose of maximizing the dissolution ratio of the bauxite tailings, achieving the dissolution percentages presented in the calculation of balances, 95% in the case of iron and 65% in that of aluminium. Moreover, with the same purpose, the temperature inside the reactor should be $80 \text{ }^\circ\text{C}$. As a result, the process needs a jacketed reactor. (Dongyang et al., 2013)

Considering the inlet flow, the reaction time and knowing that the designed reactor works discontinuously, initial dimensioning established a total reactor volume of 45 m^3 . However, it has

been found that is difficult to maintain a homogeneous agitation in such big stirred tanks. Moreover, it has also been observed in the bibliography that at such high volumes it is difficult to maintain the suspension of bauxite tailings, so the dissolution percentage previously proposed could be affected. As a result, I considered different alternatives with the aim of solving this problem.

First option is a series system of 3 tanks of 15 m³ each one. This system is capable of solving the problems previously described, achieving better agitation. Moreover, it is considered the possibility of reducing the inlet flow to 5 m³/h to reduce the reactor volume. Despite the improvements it presents, the solutions have the same problem because it is difficult to evacuate solid waste continuously.

For this reason, the best alternative is a parallel discontinuous tank system to simulate continuous operation. It has been estimated that the total time per charge will be 5 hours, considering that the reaction time is 3 hours, the estimated charge time is 1 hour and the estimated discharge time is also 1 hour, so 5 reactors have been designed. In addition, a margin has been set in the working volume of the reactors in order to be able to increase the production volume if it is necessary in the future. Specifically, it has been established that the reactor will work at 75% of its capacity, which being 15 m³ the input volume to the reactor implies designing 5 reactors with 20 m³.

In this way, the overall process can be assimilated to a continuous workflow and the flow rates are adapted to the decanter, which, as will be presented below, will preferably work continuously. This design option allows to maintain a continuous and consistent dimensioning for the entire plant.

Through the specialized bibliography, it has been possible to determine that an optimal h/d ratio for the correct performance of the reaction and the mixing process. This relation is 5/3 (Lindquist, 2003). With this relation it will be sized each reactor with 2.5 m in diameter and 4 m in height.

The next step was search for the best supplier. Pfaundler is a company that presents a range of vitrified reactors suitable for implementation in my installation. Other companies, such as De Dietrich, present reactors capable of working at high pressures as well as at high temperatures, but there are not in their portfolio any reactor that meets the coating specifications necessary to cope with the resulting corrosion of HCl. The Pfaundler reactor would also be applicable to the described operating conditions, without the need to design a

custom reactor, because they cover volumes of between 4 and 90 m³ and reactors being 20 m³. (www.pfaudler.com/es, 04/03/2020)



Figure 4. Pfaudler reactor.

(<https://www.pfaudler.com/es/productos/reactores-quimicos#lightbox>, 10/04/2020)

These Pfaudler reactors feature a technology called Glasteel which is corrosion resistant through the incorporation of a glass coating inside a reactor manufactured using AISI 316L. Furthermore, the glass used is highly resistant to abrasion, which is extremely important because inside may appear alumina dust, which is an extremely abrasive compound.

Regarding the turbine used in the mixer, comparing the different available geometries I chose a sloped blade turbine. These turbines allow to work at a high volume of pumping and with a medium that includes suspended solids, as our case is. Moreover, this turbine will create a type of axial flow within the reactor. (www.pfaudler.com/es, 04/03/2020)

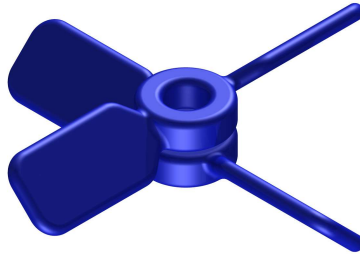


Figure 5. Turbine type.

(<https://www.pfaudler.com/es/productos/componentes-de-mezclado>, 10/04/2020)

5.2.2. Decanter.

For the correct sizing of the decanter, it is also necessary a scale of a real settling tank used in the decantation of red mud. This has been the only viable dimensioning option due to the difficulty of carrying out experiments to know the sedimentation speed or the diameters of the different particles present in the red mud. Thus, it has been chosen to scale the real reactor described in Preparation of Polymeric aluminium ferric chloride from bauxite tailings. (Dongyang et al., 2013)

Following the information provided in this article, it has been established that the best option is the use of a circular decanter, with a 30° wall inclination. It allows us to ensure the correct operation of the decantation and the precipitation of the particles in suspension.

Moreover, the V/q ratio used in the patent has been established as a scaling parameter, which gives us a residence time of 2 hours. Applying this relationship to my inlet flow of 22.8 m³/h leads to a 50 m³ decanter. Specifically, the dimensions will be 4.7 m in diameter by 4.1 m in height of the conical section and 1.5 m in the cylindrical section, defined from the calculation parameters established in the article. With this size the plant only needs one settling tank to carry out the whole process.

5.2.3. Intermediate tank.

As described above, an intermediate tank is needed between the decanter and the polymerizer, because the decanter works continuously, while the polymerizer works discontinuously. In order to have time enough to stop the process in case unexpected stop of polymerizer a buffer tank is needed, with a total volume equal to twice the polymerizer, 80 m³. Therefore, two 40 m³ tanks have been dimensioned to give greater flexibility and adaptability to the plant.

The material for these buffer tanks must be able to face corrosion. In this case, in order to reduce costs, polyethylene tanks were chosen. After searching for the different alternatives presents on the market, a model from Röchling has been selected. This company uses a mixture of high-density polyethylene called Polystone®, which is capable of coping with corrosion derived from HCl.

Furthermore, as will be seen later in the piping section, it will be necessary to pump the fluid from the decanter to the intermediate tank. Because the decanter works by overflow may be subject to small variations that could negatively affect the operation of the pump. Therefore, it has been necessary to size a small tank capable of ensuring the flow to the pump.

Specifically, this tank has been dimensioned for a total volume of 3 m³, which will be enough to guarantee the correct operation of the installation. For this volume, a height of 2 m and a diameter of 1.4 m have been established.

5.2.4. Polimerizer.

The polymerizer is a reactor under some specific conditions of pressure and temperature in order to obtain a mixture of the different hydrolyzed species of the compounds.

In this case, the sizing of the reactor has been carried out analogously to that described above for the dissolution reactor, with the same h / d ratio. Thus, for a total volume of 25 m³ a diameter of 2.7 m and a height of 4.4 m have been determined. (Company, 2000) Regarding the reaction time, the optimum point is estimated at a reaction time of 3 hours. This time allows establishing a relative basicity (ratio Al/OH) percentage of 70%, optimally developing the polymerization of both compounds following the reactions presented above. It has been

detected that a higher percentage of basicity can lead to the appearance of precipitated forms that produce destabilization of the final product. (Dongyang et al., 2013)

Although the final product of the reaction will not present an acidic pH, the entrance of the stream 11 does present a significantly acidic pH value, associated with the presence of the remaining HCl. For this reason, as in the reaction step, it will be necessary to work with a vitrified reactor. In this case, the new designed reactor also has 80°C design conditions, but additionally operates at high pressure, because these conditions have been shown to be favourable to achieve 70% basicity. Specifically the polymerizer will work at 4.4 bar pressure (Dongyang et al., 2013)

Finally, as in the case of the reactor, in order to achieve continuous operation of the installation, the possibility of working with different reactors in parallel has been considered. Therefore, it has been decided the implementation of 5 identical reactors since residence time, filling time and discharging time are the same as in the reactors.

5.2.5. Cooling jacket.

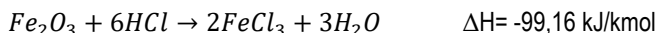
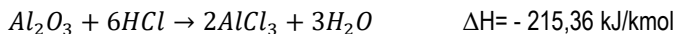
As previously described, the reactors and the polymerizers must have an operating temperature of 80°C. They have been modelled as discontinuous reactors of perfect mixing in an isothermal regime with a cooling jacket to keep the temperature inside. The calculation and dimensioning of both jackets is analogous in both steps.

To design the jacket, the first thing to do is calculate the transfer area required. Cooling tower water is used as cooling fluid, which enters the jacket at 25°C and exits at 40°C.

To calculate the necessary transfer area we use the design equation of a heat exchanger.

$$A = \frac{Q}{U \cdot \Delta T_{ml}}$$

Where Q is the heat given off in the reaction. Two reactions take place in the reactor, both exothermic, which release heat:



Thus, the heat is calculated from the following formula:

$$Q = |\Delta H_1 R_1 - \Delta H_2 R_2|$$

To calculate ΔT_{ml} , it will be considered that the fluid enters the reactor at 25°C and leaves at 40°C.

$$\Delta T_{ml} = \frac{\Delta T_1 - \Delta T_2}{\ln \frac{\Delta T_1}{\Delta T_2}}$$

Finally, the U will be estimated from tables.

According all these data, it has been possible to estimate that the total heat transfer area will be 36 m², which is equivalent to the lateral and base of the tanks. Also the water flow must be 170 kg/h.

In the case of polymerizers it has been established in an analogous way that the reaction is as follows:



Thus, in this case the heat will be deduced only from one reaction.

$$Q = |\Delta H_1 R_1|$$

Considering that the reagents inlet temperature is 25 °C, the interior and outlet temperatures must be 40 °C and an exchange area of 43 m², with a refrigerant fluid flow of 200 kg/h has been established.

6. DETAILED DESIGN

The following section includes the detailed design of the plant's secondary facilities as well as the auxiliary systems necessary for its proper operation.

6.1. DIAGRAMS

In order to help the correct understanding of the different diagrams that appears in this section, it has been decided to describe the abbreviations and references that appear in them.

6.1.1. Equipment.

For the identification of the different equipment in the diagrams, a nomenclature that includes letters and numbers has been chosen. The code follows a basic structure type AA-0000X. The first two letters allow us to identify what type of equipment we are talking about, be it a reactor, a decanter or a polymerizer for example. The first two numbers immediately after the bar indicate the section of the floor where we are. The plant has been divided in 3 different sections. The two numbers below are used to identify the different identical equipment that can be found. Finally, the last letter will only be included to identify equal teams with the same function.

Reference nomenclature for equipment:

Table 3. Equipment abbreviation.

Equipment	Abbreviation
Reactor	RC
Decanter	DC
Intermediate Tank	IT
Polymerizer	PZ
Pump	PM
Tank	TK

6.1.2. Pipelines.

For the identification of pipes in the diagrams, a specific nomenclature has been used that follows the model 00 - A / B - 0000 - AB. The first figures refer to the nominal diameter of the dimensioned pipes. Then the letter or letters in the following section allow us to identify the main component of our fluid. The set of numbers below serve to identify the piping within the facility. The first two numbers refer to our section of the plant, while the following two are responsible for numbering the pipes within the installation. Finally, the final code is used to identify the material of our pipes. In the case of the developed installation and as described in the piping section, all the pipes have been designed in polypropylene, so this sequence will be PP.

The main components in the flows will be:

Table 4. Compounds abbreviation.

Compound	Abbreviation
HCl	AC
$\text{Fe}_2\text{O}_3/\text{Al}_2\text{O}_3/\text{SiO}_2$	BT
$\text{AlCl}_3/\text{FeCl}_3$	CL
H_2O	W
Calcium aluminate	CA

6.2. PIPING

The next section describe the selection of the material used in the pipes, as well as the need for pumps and the type of pump depending on the section of the plant.

6.2.1. Pipe material.

In order to decide the material used in the piping system designed for the plant, various considerations have been considered, including the characteristics and properties of the material and the products that must circulate through the pipes.

When deciding the proper material of the pipes, low pH and corrosion are the main existing limitations. In addition, in the first section of the plant, there are a mixture of suspended solids. Therefore, it will be necessary to use a material that is not affected by the resulting abrasion. In reactors, it has been decided to design vitrified wall coverings, but this solution is not applicable to pipes. Thus, the pipes made of alloy stainless steels have been rejected, since most of them would not be able to cope with the pH of the solution. In addition, the few steels capable of coping with this situation are too expensive. (Aznar, 1997)

Therefore, pipes of different materials have been preferred, as polyethylene and polypropylene pipes. These types of polymeric pipes have a wide range of advantages compared to traditional one derived from different alloys: lightness, resistance against corrosion and inorganic acids, the lowest pressure drop, electrical insulation, flexibility and ease of maintenance and repair. In addition, they have a low cost of installation and maintenance.

Another great limitation of the process has been the temperature. By operating both the reactor and the polymerizer at 40°C, the resulting fluid is at an excessively high temperature for most polymer pipes. It has been a key factor in the final choice of material for the pipes. Despite the fact that some ranges of polyethylene, such as high-density polyethylene, allow working at relatively high temperatures, they present certain difficulties, as in the vast majority of cases the lifetime of the material is severely affected. For this reason, polypropylene pipes have finally been chosen, which have advantages in this regard. (<https://www.polysan.es/>, 01/05/2020). Specifically, HDPE pipes, because this material has a higher temperature resistance.

Once the basic material of the pipes has been determined, the different alternatives present in the market has been studied. In this regard, it must be said that this type of pipes has a very widespread use within the drinking water sector, because it also allows very satisfactory hygienic conditions. (Lindquist, 2003)

Polysan is a company within a portfolio including different polypropylene-based polymeric pipes. Specifically, two models have been compared, the PP-R and the PP-RCT. The difference between both lies in the structure of the polymer. PP-RCT is Polypropylene Random Copolymer with a modified crystalline structure, which gives to the final material greater resistance against temperature.

Specifically, the PP-RCT has a resistance of 5MPa to the critical stress required at 700°C and 50 years, while the PP-R only had 3.2 MPa. The improvement in the qualities of the PP-RCT also allows a more economical adaptation in the sizing of the plant, which allows to select thinner wall pipes and smaller diameters. In addition, by using a lesser amount of raw material, energy savings and resource conservation are achieved for the benefit of a sustainable environment. (<https://www.polysan.es/>, 01/05/2020)

6.2.2. Pumps.

By placing the reactors at a higher level than the decanter, it will not be necessary to pump the reactors liquor into the settling tank, the process uses the force of gravity. On the contrary, at the outlet of the decanter, it will be necessary to pump the fluid to the intermediate tanks, using the PM-0201 pump. In this case, in addition, it will be necessary to have a small tank capable of temporarily storing the contents of the decanter outlet stream. That is necessary because otherwise fluctuations in said stream could affect and damage the pump.

Moreover, there are other necessary pumps through the plant. Those pumps will be located at the outlets of the intermediate tanks and the polymerizers, as well as at the outlet of the sludge stream from the decanter. These should pump the fluid to the following equipment, because all of them being placed on the same level and cannot be carried out by gravity. These pumps are PM-0202A / B, PM-0203A / B / C / D / E, and PM-0301.

Regarding the pumps used throughout the process, it has been necessary to select a type of equipment capable of supporting a highly corrosive fluid. In addition, it is important to select pumps able to cope with a fluid that contains suspended particles, making the fluid highly

abrasive as well as corrosive. With these conditions, a study of the different types of pumps available in the market is needed. It must be considered that the main line, which includes sections 1 and 2, will use a type of pump, while the sludge line (section 3) must incorporate a second type of specific pumps for the transport of sludge. (Lindquist, 2003)

Specifically, after a market availability study, a supplier has been found capable of providing both pump types. This supplier is EGGER, which has two pumps in its portfolio with design characteristics very suitable for our designed process. The first of these is a propeller pump conditioned to work in highly corrosive media. This pump has a high performance and a great resistance against corrosion and allows working at high temperatures, so this factor will not be a problem. Workflow in brochure goes up to 8.300 l/s, much higher than the flow rates designed in the previously detailed production process. In addition, it is resistant to the transport of suspended solids. (<https://www.eggerpumps.com/en-us/default.aspx>, 12/05/2020)



Figure 6. Elbow pump for sections 1 and 2.

(<https://www.eggerpumps.com/en-us/products/elbow-pumps-rpp-rpg>, 20/05/2020)

The other selected pump has been a specially designed vortex pump for the transport of sludge, the Turo® Vortex T-Series pump. This pump has been designed to be abrasion-proof and anti-clog for the handling of fluids loaded with solid particles. In addition, they are corrosion resistant pumps, so if HCl escapes from the decanter due to a failure, they would not be affected. Regarding the design flow, these pumps have a maximum design flow of 200 m³/h, as in the previous case, well above the design flow of the production process. (<https://www.eggerpumps.com/en-us/default.aspx>, 12/05/2020)



Figure 7. Vortex pump for section 3.

(<https://www.eggerpumps.com/en-us/products/turo-vortex-pumps-t>, 20/05/2020)

6.3. CONTROL SYSTEM

Control in an industrial installation is important because it allows to regulate and monitor all the processes that are carried out along the production line. In this section describes the control system designed for the synthesis of polyaluminum ferric chloride (PAFC), describing the systems developed for each equipment. To complete the information about control system you will find the P&ID of the different equipment, as well as the general one, with the aim of completing the correct understanding of the designed control system.

6.3.1. Reactor.

This equipment, as already described previously, is the main one in the plant, where it takes place the dissolution of the iron and aluminium present in the raw material in the form of ferric chloride and aluminium chloride. As described in previous sections, it will necessarily work at constant temperature, needing a control system capable of regulating this aspect through the flow rate of the refrigerant fluid.

The inner temperature is measured in the output stream. If this is higher than desired, the transmitter sends a signal to the controller and the controller to the poppet valve, which will

result in an increase in the refrigerant flow. Thus, the parameter to control will be the temperature, while the control variable will be the refrigerant inlet flow rate. To ensure the correct operation of the installation in case of failure, a bypass is designed, in which the fluid flow would be controlled through a manual valve. This would also allow an emergency stop in the event of any failure.

In the reactor it will also be necessary to control the entry of HCl according to the bauxite tailings flow to ensure the correct operation of the reactor. Thus, we will control the homogeneity of the mixture by manipulating the HCl flow inlet. In the same way, it will be necessary to regulate the H₂O flow rate by measuring the outlet flow rate from the reactor. This point is of vital importance because water will be necessary to avoid precipitation of the aluminium in the outflow, as well as the correct operation of the decanter.

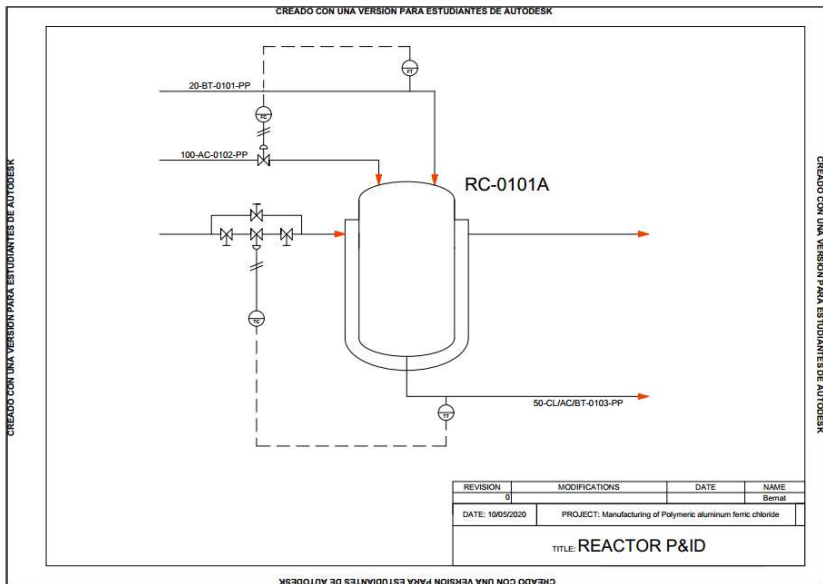


Figure 8. Reactor P&ID.

6.3.2. Decanter.

In this case, it will not be necessary to control the interior volume, since it works by continuous overflow. However, it will be necessary to control the concentration of sludge inside the decanter. The sludge outlet flow is controlled by means of a suspended solids meter located

at a certain height inside the settling tank. Thus, in the event of overcome a set up concentration at this height, a signal from the transmitter to the controller allows the output flow line to be regulated with a control valve.

On the other hand, as mentioned in the previous section, at the outlet of the decanter it is necessary a pump capable of propelling the fluid to the intermediate tanks. A small pumping tank is designed in order to protect the pump to be unburdened. In this tank, it will be necessary a level alarm, which alerts the control panel when it drops below a set limit.

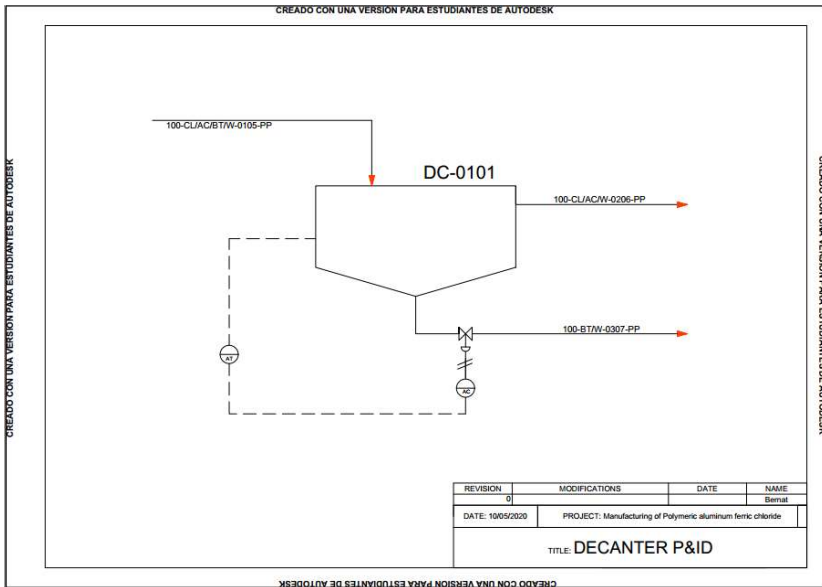


Figure 9. Decanter P&ID.

6.3.3. Intermediate Tank.

For intermediate tanks, it is necessary to control the inner level. In this case, instead, on/off valves have been used in the inlet flow of the tanks, which will be closed when a previously determined liquid height is detected. In this way, the flow is redirected to the second intermediate tank without interrupting the production process.

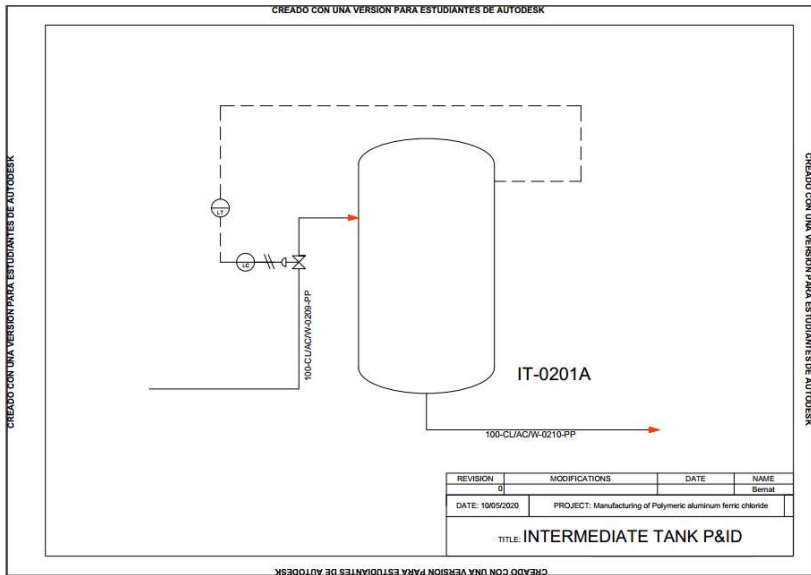


Figure 10. Intermediate tank P&ID.

6.3.4. Polymerizer.

As in reactors, the main variable to control within the polymerizer is the temperature, so we will have the same control loop as in the case of the reactor. The temperature inside is measured from the temperature of the output stream. In case of overcome a set point the transmitter sends a signal to the controller and the latter to the poppet valve, which translates into an increase in the refrigerant flow. As in the reactors, to ensure the correct operation of the installation in case of failure, a bypass has been designed in which the fluid flow would be controlled by one manual valve. In addition, it allows a manual shutdown in case of emergency.

Another important control point in the polymerizer is the entry of calcium aluminate, that is necessary for the correct polymerization of the product. Therefore, the inlet flow rate of calcium aluminate into the polymerizer is controlled by measuring the product inlet flow rate. That flow, as previously described within this project, will be regulated by an on/off valve which will be opened manually once the discharge is complete.

Moreover, unlike the reactor, the polymerizer will work at a pressure higher than atmospheric, so it's necessary install a safety valve that opens in case of emergency.

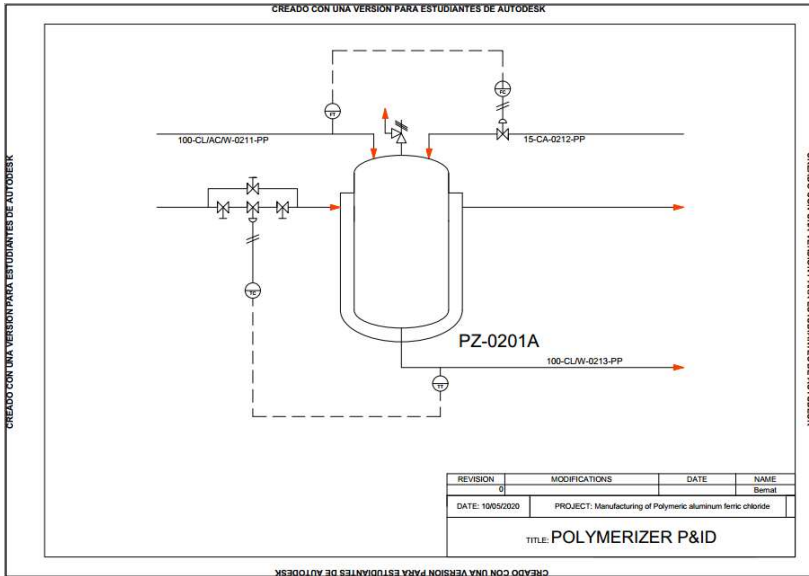


Figure 11. Polymerizer P&ID.

As it can be seen, most of the control loops described correspond to feedback loops, which allows control by feedback. This means that the control variable is manipulated when a disturbance occurs in the parameter to be controlled. The only exception is the control loop established in the decanter which corresponds with a feedforward loop, because it allows to carry out an anticipatory control of the sludge concentration at the fluid outlet. For this reason, when implementing feedback and feedforward loops within the plant we can consider an advanced regulatory control.

Another aspect affected within the control section has been the selection of the control valves used in the plant. After carrying out a detailed study of the different types of valves on the market, the 35000 Camflex II model from Masoneilan was chosen.

The Camflex II valve is based on a spherical, eccentrically rotating plug, contained in a free-flowing body. The seat surface of the shutter is connected by flexible arms to the hub, which can slide on the rotating shaft. The shutter is free to self-center along the axis of the tree. A good

seal between the shutter and seat is achieved due to the elastic deformation of the shutter arms. The seat ring is attached to the valve body by a threaded retainer. The shutter and shaft rotate at a 50° angle by means of a lever connected to a powerful spring-loaded diaphragm actuator with an opposing spring. (<https://valves.bakerhughes.com/masoneilan>, 18/05/2020)

In this way, working at high flow rates is improved by working with a plug that is not pressurized on the passage section of the valve. In addition, these types of valves allow work with high flow rates while maintaining high precision in fluid control. In parallel, they allow incorporating shutters and seats resistant to corrosion and the appearance of suspended solids, which are highly interesting aspects to take into account in the designed production process. (<https://valves.bakerhughes.com/masoneilan>, 18/05/2020)

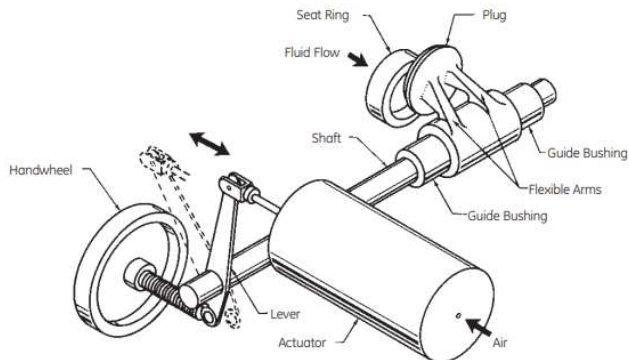


Figure 12. Camflex II valve.

(<https://corona-control.se/wp-content/uploads/2016/11/MN-35002-Camflex-IOM-GEA19538B-English.pdf>, 28/05/2020)

As a summary, the general P&ID of the plant is presented, in which all the control elements described above can be observed together.

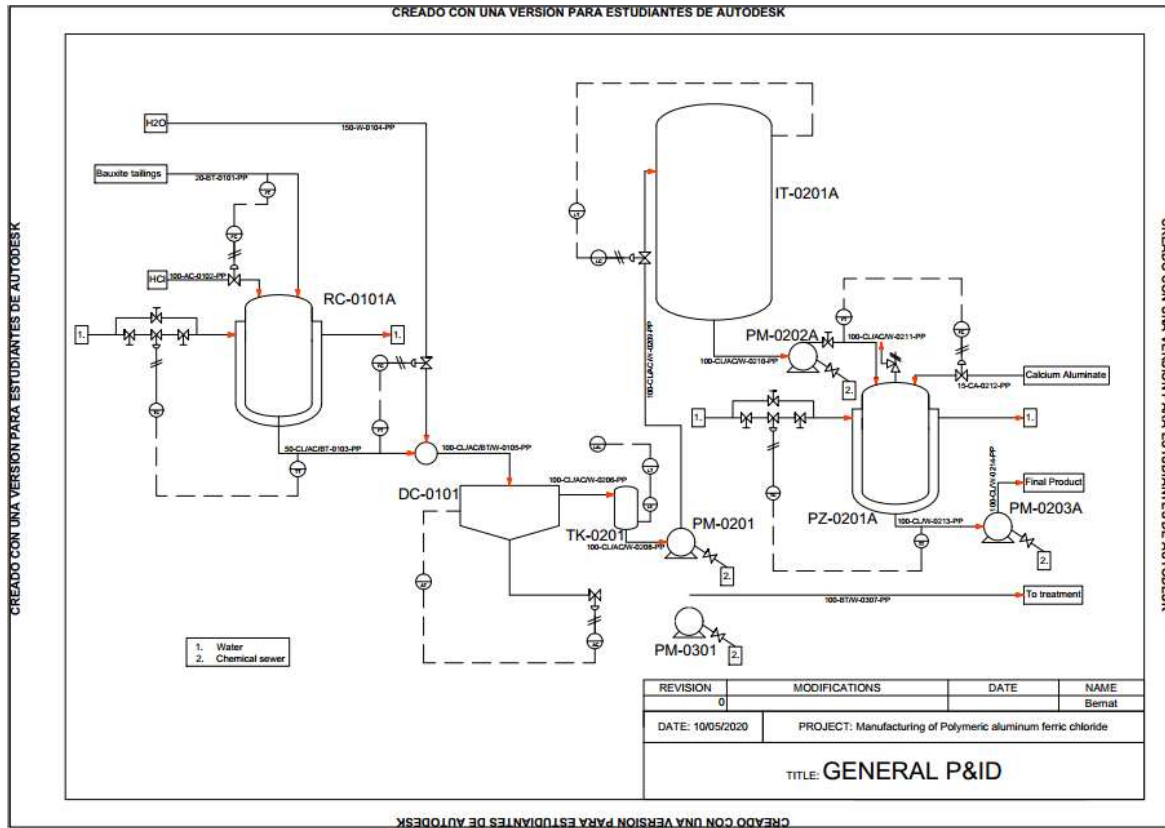


Figure 13. PAFC General P&ID.

6.4. LAY OUT

With the aim of distributing the equipment within the plant and providing an overview of it, the Lay Out of the plant has been carried out which is included below.

It has been chosen to design the plant on two levels, which will allow gravity to be used to transport the fluid through the first section. Therefore, the 5 reactors have been arranged on the upper level of the plant. This level is 7 meters above the ground and has a total height of 6 meters. At the lower level and located just below the reactors are the decanter and the little tank, so that the fluid circulates by gravity up to this point. From here and for the rest of the plant sections it will be necessary to use pumps.

The upper level on which the reactors are located, does not cover the plant entirely, but is only available along a surface of approximately 200 m². This is mainly due to the total height of the intermediate tanks, which being 8 meters and exceeds the first level. In total, the lower level of the plant will occupy approximately an area of 470 m², which includes the 200 m² located below the first level plus the 270 m² of the second part of the plant.

Specifically, the new designed PAFC manufacturing plant will be approximately 36 meters long by 13 meters wide. The next figure presents the lay out of the plant.

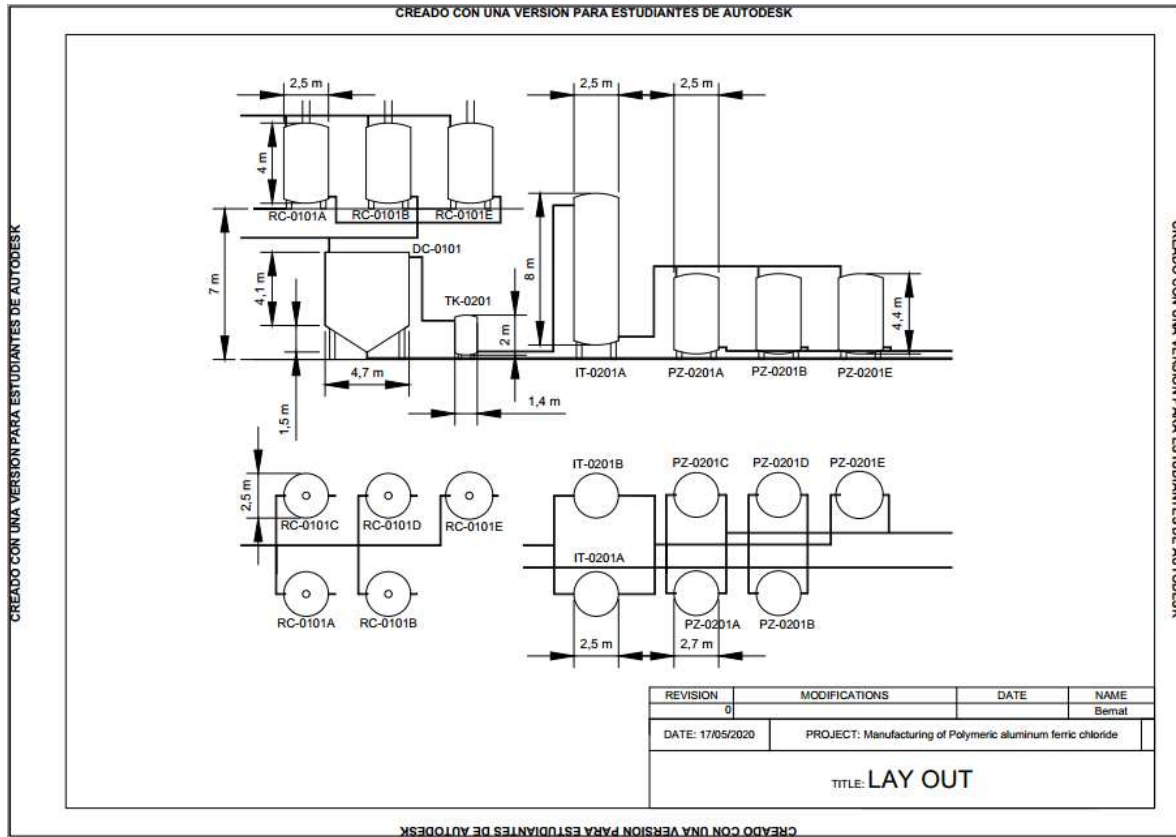


Figure 14. Lay Out.

6.5. PLANT SAFETY AND RISK ASSESSMENT

As a method of evaluating plant safety and predicting possible failures and action procedures, HAZOP has been chosen. Through this tool, and using guide words, you can make a systematic analysis of the process variables deviations. For this reason, it can be analysed the possible deviations, their most probable causes, their consequences and the necessary corrective measures and actions in each case. Therefore, this method allows going back in a systematic and controlled way, analysing the different equipment in operating conditions.

Below is the HAZOP for the plant.

Table 5. Hazop.

Guide Word	Meaning	Parameter	Deviation	Causes	Action Required
LESS	Quantitative decrease of the parameter	Temperature	Temperature drop	Possible heat losses. The activation energy would be reduced, directly linked to the kinetics of the reaction, the reaction time would have to be increased by altering the dissolution of the bauxite tailings and the formation of chlorides	Shut off the refrigerant stream so that the system temperature returns to its operating state
		Pressure	Decreased pressure	Bomb cavitation	Check for leaks, reduce output flow
		Level	Lower liquid level height in the reactor, polymerizer and tanks	Electronic error of the measuring instruments	Repair of measuring instruments. Close the PM-0201 pump to avoid problems with it
		Flow	Decreasing flow	Pump failures, small leaks, in-line sediment or possible valve blockages	Open valves and check for possible blockages. In the event of a leak, the installation is stopped

MORE	Quantitative increase of the parameter	Temperature	Temperature rise	Exterior fires, equipment blockages, reactor explosion or uncontrolled reaction. The activation energy increases, and therefore the reaction time is shorter. The proportions of Polyaluminum ferric chloride would also be affected, the desired ones would not be obtained	Reduce the flow of the cooling fluid in the case of falling film and increase the flow of refrigerant in the case of the reactor
		Pressure	Pressure rise	Decrease the flow of the cooling fluid in the case of the valve stuck or too open. Wrong gauge reading, cavitation and wrong instrument readings	Increase the output flow. Relieve pressure by opening the safety valve

		Level	Level above control limits	Error in the operation of the pump, incorrect opening of the valves, erroneous reading of control instruments	Decrease flow and check for possible stuck valves
		Flow	Flow higher than established	Electronic failure of control measuring instruments	Open valve of outlet stream. Divert the inlet stream to another of the intermediate tanks
NO	Absence of the parameter to which it is applied	Pressure	In vacuum there are no pressure	Pump failure, valve closed, or leak	Open inlet or outlet valve and repair pump
		Flow	Not flow	Blocking: pump failure; valve closed or stuck; leak, valve open, control failure. (Without the desired flow of reagents, the reaction cannot be carried out)	Repair valve or open valve for flow restoration
		Level	emptiness	Sediment obstruction	Clean pipes

OTHER THAN	Activity other than normal operation, complete replacement of the desired function, or anything that may happen other than normal operation	Impurities	Flow with unwanted compositions	Entry of external contaminants such as air, water or oils, corrosion products, insulation failure or presence of materials due to internal leaks	Purge the system
		Extraordinary added phase	Start up, operation with low performance or excessive consumption, alternative mode of operation, inspection or weather conditions	Failures in start up	Review of the operating conditions and change
		Composition	Wrong composition and wrong products used	Incompatibilities	Purge
PART OF	Qualitative decrease, get only a part of the design intent, any difference from what it should be	Composition	Decrease of polymerized forms in the final product	Too high concentration of HCl in the mixture, change in the feed or entry of contaminants	Modification of the calcium aluminate flow and readjustment of the operating conditions of the polymerizer

MOREOVER	Qualitative increase, you get more than just design intentions, more components in your system than there should be	Composition and Impurities	Increased concentration, impurities, corrosion or contamination	Entry of external contaminants such as air, water or oils, corrosion products, insulation failure or presence of materials due to internal leaks	Modification of flows and cleaning of equipment and pipes. Renewal of these if the corrosion has been too strong
		Extraordinary added phase	There are more phases than expected	Decanter malfunction and decreased residence time	Mud purge through the larger opening of the outlet valve
REVERSE	Analyse the investment in the direction of the variable, obtaining the opposite effect to that intended or the opposite of the design purpose	Flow	Reverse flow	Pump failure, back siphon, pump reversal, check valve that fails or is improperly installed in the pipeline	Stopping the pump and reopening/closing the valve
		Chemical reaction	Reverse reaction	Wrong operating conditions in the reactor	Modify reactor conditions
FAILURE OF	Study error for process improvement	Material	Material election	Corrosion, erosion, stress, etc	Change and/or repair of the material

		Material	Breakage of the vitrified coating	Breakage of the vitrified coating of reactors and polymerizers as a consequence of their use and the corrosion produced by the acidic medium	Replacement of damaged reactors with new reactors
		Services	Services failure (air, water, steam, electricity)	Lack or significant decrease in any of them.	Plant stop
		Operations	Operations failure	Malfunction of some of the unit operations	Plant stop

6.6. SERVICES

Obviously, some services are needed in order to operate correctly the plant. Specifically, it will be necessary to have cooling water, steam and electricity.

It is necessary to refrigerate in two equipment, in the reactor and in the polymerizer, using in both cases the same fluid under the same conditions. Cooling water at 25 °C will be used in the reactor, which will come out of the jacket at 40 °C and the flow will be about 170 kg/h. As for the polymerizer, where water also keeps 25 °C with a flow rate about 200 kg/h.

For the instrumentation and control of the plant it requires air with a very low humidity. The air is dried by adsorption, placing a dew point around -40°C in the resulting air. Compressed air is very common in the industry and has advantages over hydraulic systems because pneumatic systems are faster. This is a key factor in process control, because corrective action must be quick in order to avoid deviation from the setpoint value.

In addition, obviously, to operate correctly all the equipment needs electricity.

6.6.1. Geographic location.

Regarding the geographical location of the plant, it is obviously preferable that the plant is placed in a point easy to access and with important commercial communications. Specifically, when large amounts of raw material are required, especially bauxite tailings and HCl. Bauxite tailings is provided in bulk by vessels, so it would be interesting for the plant to have a direct connection by sea. Another easier alternative is be placed close to an alumina refinery. (Lindquist, 2003)

Moreover, it must be considered having a large area of land to disposal the high volume of solid waste generated in the form of sludge. As previously described, the plant uses industrial waste as raw material, and despite the advantages that this implies it involves a greater amount of waste compared to processes based on pure raw materials. When using pre-treated raw materials, a smaller quantity of sludge is produced because it contains a smaller quantity of impurities. (Dongyang et al., 2013)

7. CONCLUSIONS

In this work, the design of a production plant for polyaluminum ferric chloride, a coagulant capable of incorporating aluminium and iron salts in its composition in polymerized form, has been carried out. For the design of the plant the following stages have been covered:

- Through a bibliographic study, the product to be manufactured has been selected. Different types of coagulants have been studied, as well as their different mechanisms of action and main applications. As a result of this study, it was decided to produce a mixed salt of aluminium and iron in its polymerized form, a third-generation coagulant with higher performance than those currently on the market.
- Based on a study of the different alternatives available for the manufacture of polyaluminum ferric chloride, it has been chosen the use of bauxite tailings as a raw material. In this decision, the importance of giving a second utility to a by-product has been considered. This by-product is very available in the industry, since it comes from the production process of alumina. In addition, through the use of a single product, it has been possible to provide the quantities of iron and aluminium necessary to produce a competitive coagulant at a purity level.
- Through a study of current production and consumption of coagulants and taking into account processes of companies in the sector, an adequate production volume has been established. In order to cover current demand, and also mirroring the main production plant of coagulants in Spain, a FeCl_3 production flow of $2 \text{ m}^3/\text{h}$ has been set.
- Using process synthesis techniques, the main operations for the process have been selected. They consist on an initial chemical reaction providing dissolved iron and aluminium, a settling of solids step, and finally a polymerization of the clarified solution of iron and aluminium salts. In this way, separating the polymerization from the reaction stage and carrying out continuous decantation in the middle of the process, a more

adaptable process is achieved, limiting the problem of working with a high content of suspended particles and minimizing the abrasion that occurs in the equipment.

- The basic design and dimensioning of the main equipment has been carried out, considering mainly the need to work with corrosion resistant reactors resulting from working with high amounts of HCl. For this, it has been chosen to work with polypropylene pipes and with vitrified reactors.
- The detailed design of the rest of the secondary systems of the plant has been carried out: the control system, the piping of the plant and the lay out of the plant.
- Finally, considering the needs and limitations of the plant, a description has been made of the conditions necessary to determine the location of the plant.

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APPENDICES

APPENDIX 1: SPECIFICATION SHEETS

MANUFACTURING OF POLYMERIC ALUMINUM FERRIC CHLORIDE			
REACTOR SPECIFICATION SHEET			
TYPE: AGITATED TANK REACTOR			
PLANT	MANUFACTURING OF POLYMERIC ALUMINUM FERRIC CHLORIDE		
SECTION	01.		
SERVICE	ALUMINUM FERRIC CHLORIDE MANUFACTURE		
ABBREVIATION	RC-0101		
UNITS	5		
SHEET	1	OF	1
DESIGN DATA			
RESIDENCE TIME	3		h
DIAMETER	2.5		m
HEIGHT	4		m
INPUT TEMP.	25		°C
OUTPUT TEMP.	80		°C
COOLING FLUID	WATER		
COOLING FLUID FLOW	170		kg/h
COOL FLUID INLET TEMP.	25		°C
COOL FLUID OUTLET TEMP.	40		°C
TRANSFER AREA	36		m ²

MANUFACTURING OF POLYMERIC ALUMINUM FERRIC CHLORIDE

DECANTER SPECIFICATION SHEET

TYPE: CIRCULAR DECANTER

PLANT	MANUFACTURING OF POLYMERIC ALUMINUM FERRIC CHLORIDE		
SECTION	01.		
SERVICE	ALUMINUM FERRIC CHLORIDE AND RED MUD SEPARATION		
ABBREVIATION	DC-0101		
UNITS	1		
SHEET	1	OF	1

DESIGN DATA

RESIDENCE TIME	2	h
DIAMETER	4.7	m
CONE HEIGHT	4.1	m
CYLINDER HEIGHT	1.5	m
WALL INCLINATION	30	°

MANUFACTURING OF POLYMERIC ALUMINUM FERRIC CHLORIDE

POLYMERIZER SPECIFICATION SHEET

TYPE: AGITATED TANK REACTOR

PLANT	MANUFACTURING OF POLYMERIC ALUMINUM FERRIC CHLORIDE		
SECTION	02.		
SERVICE	ALUMINUM FERRIC CHLORIDE POLYMERIZATION		
ABBREVIATION	PZ-0201		
UNITS	5		
SHEET	1	OF	1

DESIGN DATA

RESIDENCE TIME	3	h
DIAMETER	2.7	m
HEIGHT	4.4	m
PRESSURE	4.4	Bar
INPUT TEMP.	25	°C
OUTPUT TEMP.	80	°C
COOLING FLUID	WATER	
COOLING FLUID FLOW	200	kg/h
COOL FLUID INLET TEMP.	25	°C
COOL FLUID OUTLET TEMP.	40	°C
TRANSFER AREA	43	m ²

