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The dawn of the Chemical Industry in Portugal

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ACADEMIA DAS CIÊNCIAS
DE LISBOA

LISBOA • 2024

Título: The dawn of the Chemical Industry in Portugal
Edição: Academia das Ciências de Lisboa
Data de edição: 2024
DOI: https://doi.org/10.58164/hagg-sc27

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ABSTRACT

The birth of the chemical industry and its development during the Industrial Revolution are the subject of a brief analysis, before turning to the case of Portugal. Here, the chemical industry arrived in 1829, when Joaquim Pedro Quintela (later to become the Count of Farrobo) obtained the privilege to introduce the production of soda ash and sulfuric acid in the country. The subsequent events are discussed until the birth of the CUF Group (in the 1920's), which was to become the largest Portuguese business group of the 20th century. The technological evolution (raw materials, production processes and products) is described, highlighting its main protagonists.

O alvorecer da Indústria Química em Portugal

RESUMO

O nascimento da indústria química e o seu desenvolvimento durante a Revolução Industrial são objecto de uma breve análise, antes de abordar o caso de Portugal. Aqui, a indústria química de base chegou pela mão de Joaquim Pedro Quintela (mais tarde Conde do Farrobo) que, em 1829, obteve do Governo o privilégio de exclusivo para introduzir no País o fabrico de soda e ácido sulfúrico. Nesta comunicação descreve-se o desenvolvimento subsequente da indústria química até à formação do Grupo CUF, na década de 1920, que se viria a afirmar como o maior grupo económico português do século XX. Refere-se a evolução tecnológica (matérias-primas, processos de fabrico e produtos), e destacam-se os seus principais protagonistas.

INTRODUCTION

The chemical industry was born in Great Britain during the Industrial Revolution, in the mid-18th century, due to the growing demand for acids and bases from other booming industries (textiles, glass, soap). Sulfuric acid was the first base chemical product to be produced on a large scale. Until then, it had been obtained in laboratories, and metal pickling was its main application[1].

The process that was implemented on an industrial scale was based on the combustion of a mixture of sulfur and saltpeter (potassium nitrate); the resulting gases, in contact with water, produce sulfuric acid. This process was already used by Johann Rudolf Glauber in the mid-17th century and was introduced in England by Joshua Ward in 1736. Instead of a laboratory retort, Ward used 50-gallon glass vessels, increasing the scale of production from grams to kilograms. But it was John Roebuck who invented a technology capable of producing sulfuric acid on the scale of tons, using large chambers lined with lead (a metal that is not attacked by the acid). As a result of this increase in scale, the price of sulfuric acid went down a hundredfold. The first sulfuric acid plant by the lead chamber process was set up in Birmingham in 1746 (*Birmingham Vitriol Manufactory*), but three years later the company moved to Scotland (*Prestonpans Vitriol Manufactory*). The chamber process soon reached other European countries (France, Bohemia, Germany) and the United States[2-4]. The acid produced in the lead chambers had a relatively low concentration (62-68%), which was nevertheless sufficient for most applications in the textile and metallurgical industries, and in the new chemical processes that emerged in the first half of the 19th century (soda and superphosphates).

In 1774, the Swedish chemist Carl Scheele discovered chlorine by reacting hydrochloric acid ("*muriatic acid*") with pyrolusite (a mineral made up of manganese dioxide) and observed the discoloring properties of the gas. At that time, chemistry was still interpreted in terms of the "*phlogiston theory*", and Scheele, considering that the gas was a compound, gave it the name "*dephlogisticated muriatic acid*". In France, Claude Berthollet used chlorine (which he called "*oxygenated muriatic acid*") to prepare bleach (sodium or potassium hypochlorite), marketed from 1786 under the name "*Eau de Javelle*". However, it was in Great Britain that the chlorine bleaching process was implemented most quickly, after Scottish entrepreneur Charles Tennant obtained a solid product by chlorinating dry calcium hydroxide. "*Chloride of lime*" (more precisely, calcium chloride-hypochlorite) was patented in 1799 under the name "*bleaching powder*" and began to be produced in the plant that Tennant built near Glasgow (*St. Rollox Chemical Works*)[5-7]. This was an ingenious invention, as it allowed the gas (chlorine) to be transported and handled safely in the form of a solid product. By adding an acid to the powder, the gas is released *in situ*, which justifies the product's common name in Spain: "*polvos de gas*"[8].

The scarcity of natural alkalis (obtained from plant ashes, or from the mineral "*trona*" or "*natron*") was felt at the end of the 18th century, especially in France, where there was an important glass industry. It was there that Nicolas Leblanc patented (in 1791) a process for making soda (sodium carbonate) from common salt (sodium chloride). Industrial production began shortly afterwards but was interrupted during the "period of terror" of the French Revolution. Once again, it was in Great Britain that the process was implemented on a large scale, especially after the salt tax was abolished in the 1820's [9-11].

The organic chemical industry emerged in the mid-19th century, when the properties and applications of aromatic compounds obtained from the distillation of coal tar began to be studied. Tar was a widely abundant raw material, being a by-product of the pyrolysis of coal for the production of metallurgical coke (introduced in Coalbrookdale in 1709) and lighting gas (London became the first gas-lit city in 1812). Particularly important in this context was the methodology developed at Liebig's school, in Giessen[12], where August Wilhelm von Hofmann studied the properties of aniline. Hofmann continued this work at the Royal College of Chemistry, in London, of which he became the first director (from 1845 to 1865). It was there that, in 1856, his young assistant William Perkin accidentally synthesized the first dye from aniline. Synthetic dye factories soon appeared in the UK and in France; however, by the end of the 19th century the German industry had already taken the lead, accounting for over 90% of the world's production of synthetic dyes[13-15].

Figure 1 shows the chronological evolution of the chemical technologies implemented in the period in question (at the bottom), while the most relevant scientific discoveries are marked at the top: discovery of oxygen (Scheele, 1771, and Priestley, 1774); theory of combustion (Lavoisier, 1777); discovery of chlorine (Scheele, 1774) and production of hypochlorites (Berthollet, 1786); process for producing artificial soda (Leblanc, 1791); interpretation of the lead chamber process for sulfuric acid production (Desormes & Clément, 1806); establishment of the elemental nature of chlorine (Humphry Davy, 1811); patent for the contact process (platinum catalyst) to produce sulfuric acid (Phillips, 1831), a process that would not be developed until the end of the century, without fame or profit for its inventor; identification of a new chemical phenomenon, "catalysis" (Berzelius, 1836); the organic synthesis experiments in Liebig's laboratories in Giessen (>1840), carried out, among others, by August Wilhelm von Hofmann, which would later lead to the discovery of the first synthetic dye (Perkin, 1856); and a new process for making soda from salt (Solvay, 1861), which was more efficient and less polluting than the Leblanc process[1]. As can be observed, these scientific discoveries took place in various European countries (Sweden, the United Kingdom, France, Germany, Belgium); but most of the industrial developments of this period occurred in the United Kingdom, which, by 1870, led the world's chemical industry, with annual productions of 300 kt of soda and 590 kt of sulfuric acid [10,16].

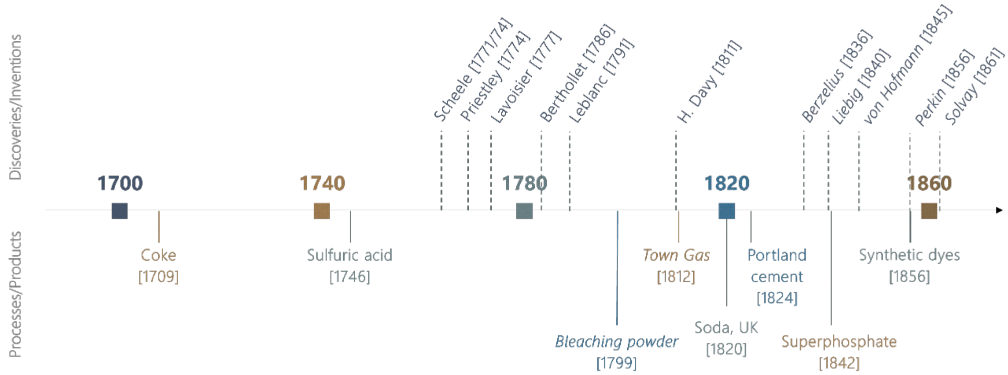

Figure 1. Chronology of chemical technologies implemented during the period of the industrial revolution (bottom), and most relevant scientific discoveries (top).

The lead chamber process was the first catalytic process used on an industrial scale (a rare example where the catalyst is a gas), long before the role of nitrate in the reaction mechanism was correctly interpreted[17]: it is NO_2 (resulting from the decomposition of nitrate) that promotes the oxidation of SO_2 (resulting from the combustion of sulfur). In other words, NO_2 is the catalyst for the reaction (Figure 2). But all this happened before Berzelius had identified the phenomenon of catalysis (1836), which in turn would only be properly explained 60 years later, by W. Ostwald! Thus, the lead chamber process is one of those examples where technology preceded science[18].

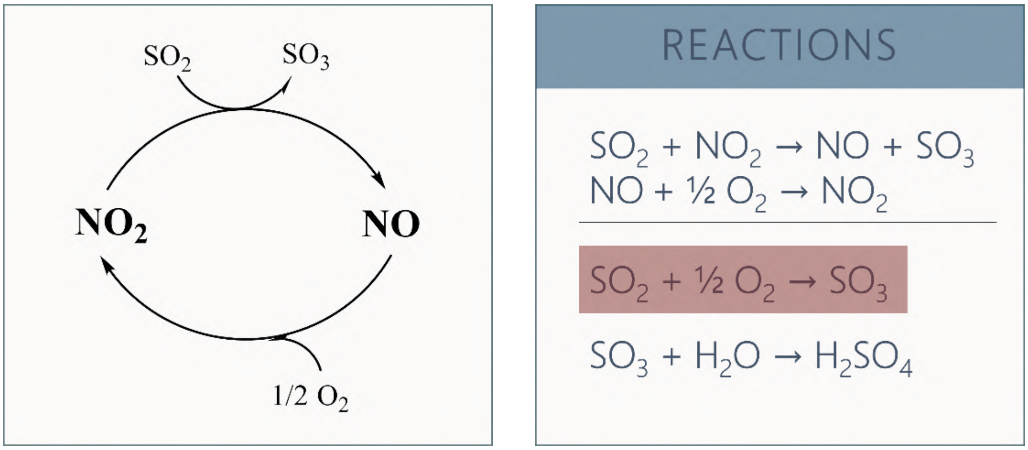

Figure 2. Simplified catalytic cycle and reactions in the production of sulfuric acid[1].

The first chemical plants in Portugal

The pioneer of the chemical industry in Portugal was Joaquim Pedro Quintela (1801–1869), 2nd Baron of Quintela, who received the title of Count of Farrobo in 1833 (Figure 3)[1]. Although he is mostly remembered as a great patron of the arts, the Count of Farrobo was also the protagonist of several business initiatives. In 1829, he obtained authorization from the government to introduce the production of soda ash and sulfuric acid in the country (with an exclusive privilege granted for 14 years). The plant was to be set up at *Quinta da Verdelha* (in Alverca), but it only started working in 1836, as its construction was interrupted during the Civil War (1832–1834). At the Exhibition of Portuguese Industry in 1838, the *Verdelha Factory* presented three products: "*oil of vitriol*" (sulfuric acid), "*green vitriol*", also known as "*copperas*" (iron sulfate), and refined soda (sodium carbonate). However, the regular production of soda only started after 1847, when Professor Júlio Máximo de Oliveira Pimentel was hired as a technical consultant[1]. The factory's production had a significant impact on imports of refined soda, which fell from 119 t in 1843 to just 25 kg in 1848. In the same period, imports of "*barilla*" (a natural product rich in soda, made from the ashes of *salsola soda*, a plant grown on the coasts of Alicante) fell from 80 to 20 t. A greater diversity of products was presented at the Portuguese Industry Exhibition of 1849, where Verdelha received an honorable mention, with emphasis on the quality of its soda, oxalic acid and chloride of lime. That same year, the Count leased the factory to his friend and accountant Ignácio Miguel Hirsch. Verdelha was also present at the Universal Exhibitions in London (1851) and Paris (1855), winning a 1st class silver medal in the latter, for which the entrepreneur (I. Hirsch) was decorated by King Pedro V[1,19].

Figure 3. Portrait of the Count of Farrobo, by Julien-Paul Delorme (1823). National Museum of Ancient Art (photograph by Luísa Oliveira) [D.G. of Cultural Heritage/Photographic Documentation Archive].

The information available about the equipment installed at Verdelha is scarce. It is known that a platinum "boiler" had already been imported in 1831, valued at four *contos de réis* (4000$000 rs); this apparatus was used to concentrate sulfuric acid[19]. Otherwise, there is only reference to two lead chamber apparatuses for producing sulfuric acid "by the English process"[20]. In 1827, Gay-Lussac had already introduced an absorption tower at the St. Gobain factory (Chauny, France) to recover and recycle the catalyst (NO_2), but this innovation only became widespread in the 1870's, after it had been complemented by the Glover tower (invented in 1859), making the process much more efficient. We can therefore infer that Verdelha's equipment consisted only of simple lead chambers (of unknown capacity), with external combustion of the sulfur and nitrate mixture (the number of furnaces is also unknown), this being the usual set-up since the beginning of the 19th century. There was also a boiler to inject water vapor into the chambers[21-23]. The production of soda by the Leblanc process involves two steps: first, sodium chloride reacts with sulfuric acid to yield sodium sulfate ("salt cake") and hydrochloric acid; the "salt cake" is then mixed with limestone and coal and calcined, giving a solid product known as "black ash" (mainly composed of sodium carbonate and calcium sulfide), from which the carbonate can be extracted with water and is finally obtained by evaporation[1]. There is no information on the type of furnaces and other equipment used to manufacture and refine the soda and other products from the plant. However, the costs of the raw materials and of the various operations are known in detail. Verdelha imported sulfur from Sicily, coal from England, and sodium nitrate was also imported. The salt came from the salt pans owned by the Count in the region, and the limestone (chalk) was obtained on the outskirts of Lisbon and crushed in the factory using a mill powered by a steam engine. The salaries of the various workers ranged from 200 rs/day for unskilled personnel, to 240 rs/day for the workers in the lead chambers and in drying of the refined soda, 280 or 320 rs/day for the furnace operators in the production of sulfate and raw soda ("black ash"), respectively, and 800 rs/day for the "master plumber", responsible for the maintenance of the lead chambers. It is known that the plant consumed 1000 kg of sulfur and 100 kg of nitrate to produce the equivalent of 2500 kg of commercial acid at 66°Baumé, i.e., with a concentration of 93%. (It should be noted that the acid came out of the chambers at 50°Baumé, equivalent to a mass concentration of 62%.) The production of 100 kg of commercial acid cost around 1$920 rs. In turn, the cost of producing 100 kg of refined soda was around 6$980 rs[21-23]. According to the Industrial Statistics of December 31st, 1852, the Verdelha factory employed 46 workers[22]. At that time, it produced about 900 t/year of sulfuric acid and more than 250 t/year of soda, as well as various acids (hydrochloric, nitric and oxalic) and sulfates (of sodium, copper and iron), and chloride of lime. The soda was mostly consumed by the soap factory, but it also supplied the glass, textile and paper industries. The Verdelha plant went into decline after the introduction of the new customs duty in 1852, and by 1862 it had already closed down[19].

Lighting gas was introduced in Lisbon in 1848 by the *Companhia Lisbonense de Iluminação a Gás*, with a factory on *Av. 24 de Julho*. The Count of Farrobo was one of the company's main shareholders. The gas was produced by pyrolysis of coal, stored in gasometers and distributed by underground pipes[1]. Until the end of the 19th century, three more inorganic chemical plants had been established[24]:

– The *Trafaria Factory*, owned by *Companhia de Guano Químico de Peixe* (founded in 1858), produced a phosphate fertilizer (identical to superphosphate) by treating fish and bone waste with sulfuric acid. It had a lead chamber apparatus and also produced iron and copper sulfates. By 1878 it had already closed down.

– The *Póvoa de Santa Iria Factory*, owned by *Sociedade Geral de Produtos Químicos* (founded in 1859 and financed by *Companhia do Crédito Móvel Português*), was intended to produce sulfuric acid, soda and derived products. A 1,300 m^3 lead chamber apparatus with six furnaces for burning sulfur was installed. There was also a platinum still, boilers, a steam engine, several furnaces for the production and refining of soda, and a testing laboratory. However, the society was dissolved in 1862 due to funding difficulties. The factory was acquired in 1867 by Ferdinand Deligny (a French industrialist with interests in the *São Domingos* mines), and modified to use pyrites as raw material, instead of sulfur. It was equipped with two lead chamber apparatuses (each with a capacity of 2,400 m^3) and two batteries of six furnaces for roasting pyrites and produced sulfuric acid (1,200 t/year) and soda (120 t/year), as well as nitric acid, hydrochloric acid, iron sulfate, sodium sulfate and chloride of lime[23]. In 1884, *Companhia Real Promotora da Agricultura Portuguesa* bought the factory to produce fertilizers, namely calcium superphosphate. However, it also continued to manufacture the products mentioned above, except for soda and chloride of lime, which had been exempted from import duties. In 1898 the factory was leased to *Henry Bachofen & C^a*, who operated it until 1914, when the company went bankrupt. Subsequently, it passed into state ownership.

– The *Casal das Rolas Factory,* in Cabo Ruivo, was set up in 1888 by *Tinoca Ltd*, a Scottish company that operated the *Tinoca mine,* in Arronches. The aim of the factory was to recover copper from low-grade pyrites (by a pyrometallurgical process), using the sulfur to produce sulfuric acid and derived products. It had two lead chamber apparatuses of 2,000 m^3 each, 16 furnaces for roasting pyrites, a Gay-Lussac tower and a Glover tower, a platinum still, and a laboratory. This was probably the first sulfuric acid plant in Portugal to include catalyst recovery.

It is worth highlighting the eminent figure of Júlio Máximo de Oliveira Pimentel (1809–1884), 2nd Viscount of Vila Maior (Figure 4), the first Chemistry professor at the Polytechnic School (a position he held from 1837 to 1864), and also at the Industrial Institute of Lisbon (1854–1858), and rector of the University of Coimbra (1869–1884). In September 1844, he obtained a scholarship to practice at Péligot's laboratories (at the *Conservatoire national des arts et métiers*), in Paris, where he also attended chemistry courses by Chevreul (at *Manufacture des Gobelins*) and Dumas (at the *Sorbonne*). He visited various universities and chemical plants in France and other European countries. After his return to Lisbon, in February 1846, he became a technical consultant for the soap factory in Marvila (until 1851) and the chemical products factory in Verdelha. In 1859 he founded the *Póvoa de Santa Iria Chemical Products Factory*, of which he was Director. In addition to numerous scientific works and his "Lectures on General Chemistry and its main applications" (3 volumes, published between 1850 and 1852), Júlio

Figure 4. Portrait of the Viscount of Vila Maior [Courtesy of the University of Coimbra].

Pimentel wrote a series of articles on various chemical processes, in which he also reported on new developments (published in *Archivo Universal* in 1859), as well as the "scientific lectures", published between 1859 and 1861 in *Revista Contemporanea de Portugal e Brasil*. His contributions, as a teacher and scientist, had a major impact on the development of the chemical industry in Portugal in the mid-19th century[1,26].

The *Industrial Statistics* of December 31st, 1852, refer several factories of organic products[22]: six tallow candle factories; one stearic candle factory; one vegetable oil factory; and one soap factory; however, it should be mentioned that only those establishments with at least 10 workers were considered "factories".

The stearic candle factory had been set up by the Count of Farrobo in *Bom Sucesso* (Belém, Lisbon), in 1840. Compared to the manufacture of tallow candles, the technology used was more advanced, involving the hydrolysis of fats. In 1847, the Count of Farrobo bought from Júlio Pimentel the patent rights to use palmitin, extracted from palm oil, to make candles. At the time of the Inquiry, the factory employed 36 workers and was leased to *Ignacio Miguel Hirsch & Bros*. It took part in the Universal Exhibition in Paris in 1855, where it received an Honorable Mention, and it was still in operation by 1869[19]. The vegetable oil factory in Alcântara, with 32 workers, belonged to the firm *Viúva Burnay & Sons*, and produced oil from the seed of purging nut (*Jatropha curcas*) imported from Cape Verde, an exclusive privilege granted in 1842 for a period of 15 years[27]. Before gas lighting was implemented, purging nut oil was used in Lisbon's public lighting. The manufacture of soap was a Crown monopoly[30], and the factory in Marvila (Lisbon) employed 64 workers. This monopoly ended in 1857, and since then many soap factories were built all over the country, with an inventory of 65 factories as of 1889[24].

In 1865, José Dias Leite Sampaio (1804–1870), Viscount of Junqueira, set up *Companhia União Fabril (CUF)*, which took over the operation of his *Fábrica União* (established in 1857, and producing stearic candles, soaps and vegetable oils) located in *Largo das Fontaínhas*, in Alcântara. Nearby was *Fábrica Sol*, owned by *Companhia Aliança Fabril*, managed by Alfredo da Silva (1871–1942) since 1893, which was dedicated to the same business of candles, soaps and vegetable oils. The merger of the two companies, in 1898, was the solution found to face the economic constraints arising from the national situation and the limitations of a small and very competitive market. Alfredo da Silva (Figure 5) took over the management of the new CUF and, without neglecting the traditional productions, immediately began manufacturing and marketing various types of fertilizers, using sulfuric acid acquired from the *de Santa Iria Factory*[24,29,30].

Figure 5. Photograph of Alfredo da Silva (1902). Jorge Maria de Mello Collection [Courtesy of the Amélia de Mello Foundation].

The large-scale production of chemical fertilizers, particularly calcium superphosphate, was going to be Alfredo da Silva's major stake[29-31]. This was a fully justified option, given the increasing imports of chemical fertilizers (23 kt in 1998, 45 kt in 1901, 100 kt in 1906) and the small domestic production of superphosphates (about 8 kt/year, in the two factories in operation at the beginning of the 20th century, namely the *Póvoa de Santa Iria Factory* and the *Sociedade Tinoca Factory* in Cabo Ruivo).

The industrial complex of CUF in Barreiro

Once the decision had been made to go ahead with the large-scale production of superphosphates, it was necessary to find a site for the expansion of CUF, as the project envisioned by Alfredo da Silva was not compatible with the limited space of the Alcântara factories. So, in 1907, a land plot was bought in Barreiro, and a French chemical engineer (Auguste Lucien Stinville) was hired to design and supervise the construction of the factories[32]. That same year, CUF entered the olive oil business, acquiring a factory in Alferrarede (Abrantes). From then on, and under the motto "*more and better*", the Barreiro industrial complex developed at a breathtaking pace (Figure 6): in 1908, *Companhia de Tecidos Aliança* was acquired and transferred to Barreiro (in order to ensure the production of sacks for the fertilizers), an olive pomace oil extraction plant was set up, and construction began on two sulfuric acid plants (30 t/day) and the superphosphate factory, as well as the *Santa Bárbara* living quarters for workers; in 1909, the sulfuric acid and superphosphate plants began operating; in 1910, a pier was built to facilitate the supply of raw materials (phosphate rock shipped from North Africa and pyrites from the *São Domingos mine,* in the Alentejo) and the expedition of finished products; the recovery of copper from pyrite ashes (by lixiviation) started in 1911, and two more sulfuric acid plants were built; the production of hydrochloric acid and sodium sulfate was initiated in 1912, as well as the metalworking activity; and the production of copper sulfate (a product used as a pesticide to treat vineyards) began in 1913, under a monopoly regime granted until 1924.

On the eve of the Great War, more than 100 kt/year of superphosphates were already being produced in Barreiro. In 1915, CUF acquired the ship *Lusitano*, to face difficulties in the supply of raw materials and fuels; one year later, *Tecelagem do Rato* was bought, marking CUF's entry into the textile sector[29,33]. CUF managed to overcome the problems arising from the world conflict, establishing itself as one of the largest Portuguese industrial companies in terms of its assets (9,150 *contos*) and number of workers (>2,100)[34,35].

The lead chambers installed at CUF were classic "box chambers", fed by multiple hearth furnaces where the pyrites were roasted (7.5 t/day/furnace), with a Glover tower upstream and two Gay-Lussac towers downstream[31]. As the nitrogen oxides were efficiently recovered in the towers, it was only necessary to supply NO_2 at the start of the process, and to replenish the inevitable losses during operation. The reactions involved in the manufacture of sulfuric acid are strongly exothermic, so the chambers were built on wooden frames to facilitate air circulation, and their walls were cooled by sprinkling with water[1]. The chamber plants were imposing structures, "*veritable cathedrals of timber and lead*"[33].

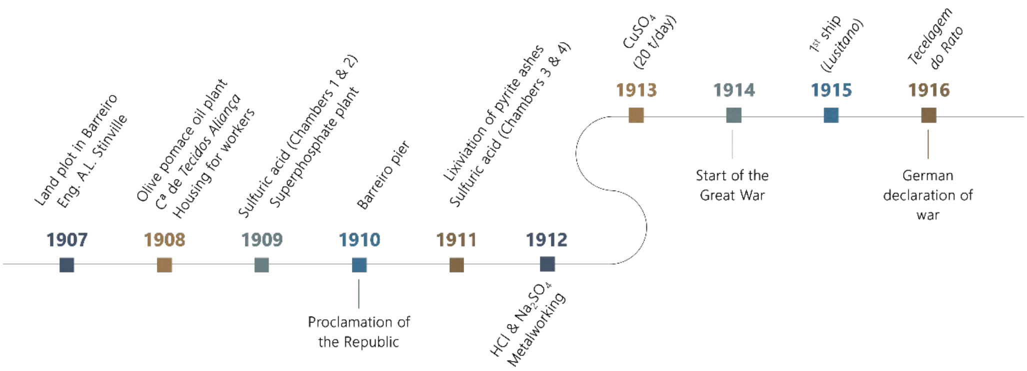

Figure 6. Development of the CUF industrial complex in Barreiro, in the period 1907-1916.

The production of normal superphosphate (i.e., with a concentration of available P_2O_5<22%) involves three steps: a) mixing the phosphate rock with sulfuric acid; b) "denning", where the resulting slurry is kept in a container (the "den") until it solidifies; and c) "curing", where the product is stored for the time needed to complete the reactions. The objective is to convert tricalcium phosphate (insoluble) into monocalcium phosphate (soluble and easily assimilated by plants). Noxious gases containing fluorine are evolved during the process, and must be collected for disposal or to obtain useful by-products [1].

Once the war was over, CUF continued to grow and to diversify its activities, under the motto "*What the country needs, CUF provides*" (Figure 7). With the creation of *Sociedade Geral de Comércio, Indústria e Transportes* (SGCIT, in 1919), and participation in banking (*Casa Totta*, in 1921), the "CUF Group" began to take shape, entering the oilseed trade (through *Soc. António da Silva Gouveia, Lda*), shipping (through *Sociedade Geral*), copper metallurgy, tobacco manufacture (*A Tabaqueira*), shipbuilding (by leasing the Lisbon shipyards), and later, insurance (*Companhia de Seguros Império*, in 1942)[29].

As far as fertilizers were concerned, competition arrived in the 1920's. In Póvoa de Santa Iria, CIP (*Companhia Industrial Portuguesa)* started to operate the Fertilizer and Chemical Products Factory (*Fábrica de Adubos e Produtos Químicos*), leased from the State, as well as a glass and crystal factory, while SAPEC (*Société Anonyme de Produits et Engrais Chimiques*), a Belgian-owned company, was established on the outskirts of Setúbal and started operating in 1928. However, when the "Wheat Campaign" began (in 1929), encouraging the use of chemical fertilizers in agriculture, CUF already dominated the superphosphate market, with 63% of the national production (the market shares of SAPEC and CIP were 28% and 9% respectively). That same year, 185 kt of superphosphates were produced in Portugal, meeting almost all of the country's needs (only 6.4 kt were imported)[1].

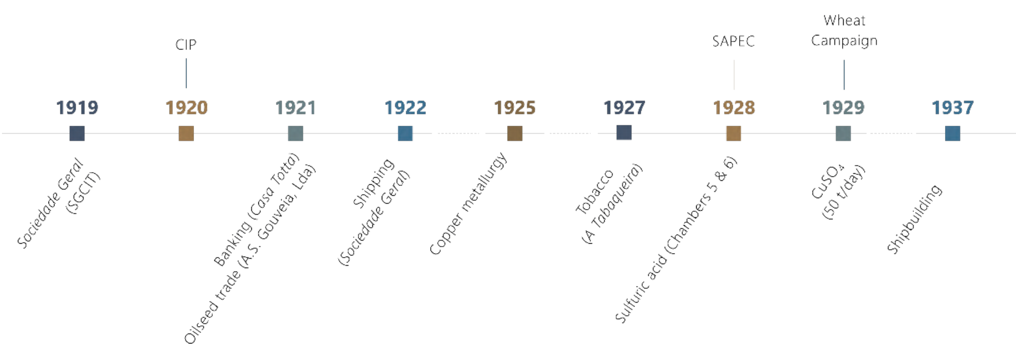

Figure 7 – Formation of the "CUF Group" and diversification of its activities after the Great War.

Alfredo da Silva was undoubtedly the great tycoon of the chemical industry in Portugal in the first half of the 20th century, leaving in Barreiro a unique work, comparable to the one that Charles Tennant built, a hundred years earlier, on the outskirts of Glasgow (*St. Rollox Chemical Works*). As he himself said, when the ships *Creoula* and *Santa Maria Manuela* were launched on May 10th, 1937: "*O que tivémos de lutar para tal conseguir foi muito, não é ocasião para se enumerar. Conseguiu-se: é o principal*"[1]. ("*We had to work hard to achieve this, but there is no need to list it. It was achieved: that's the main thing*").

Acknowledgments: This text is based on the book "The Relevance of the Chemical Industry and its Contribution to Society", published with the support of the Amélia de Mello Foundation (FAM). I would like to acknowledge, in particular, the invaluable collaboration of Dr. Jorge Quintas (FAM), and to thank my collaborator Dr. Raquel Pinto Rocha for formatting the text and producing the figures. The work was carried out within the scope of the ALiCE and LSRE-LCM Research Units, financed by *national funds through FCT/MCTES (PIDDAC): LA/P/0045/2020 (ALiCE), UIDB/50020/2020 and UIDP/50020/2020 (LSRE-LCM).*

COMUNICAÇÃO APRESENTADA À CLASSE DE CIÊNCIAS
NA SESSÃO DE 21 DE SETEMBRO DE 2023

COMUNICAÇÃO RECEBIDA A 16 DE OUTUBRO DE 2023

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