The Early Development of the Casale Process for the Production of Synthetic Ammonia (1917-1922). The protagonists, the technology, and a link between Italy and the United States.

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Abstract

The Haber-Bosch process for the industrial synthesis of ammonia, originally intended for use in manufacture of nitrogen fertilizer, was inaugurated by BASF of Germany in 1913. During the First World War the process proved to be of tremendous value to Germany for the production of munitions. This was appreciated by the Allied nations, but, despite great efforts, they were unable to replicate the industrial process prior to the cessation of hostilities. Notwithstanding tremendous postwar demand for nitrogen products to ensure national security in both munitions and fertilizers, BASF refused to license its ammonia process. This ultimately forced inventors and firms elsewhere to innovate based on their wartime research efforts.

This paper provides an account of the emergence of the first successful rival to the BASF Haber-Bosch synthetic ammonia process, that of the Italian inventor Luigi Casale. To accomplish this goal, Casale in 1917 gained the support of the wartime chemical manufacture Idros, at Terni, north of Rome, headed led by the Franco-American entrepreneur René Leprestre. However, better facilities for development of a working process, including byproduct hydrogen, were available at the works of the Rumianca company. Casale moved there in 1919. Leprestre brought in representatives of a group of investors from the United States to observe Casale's process in action at the Rumianca works. However, disputes quickly emerged. Agreements were broken, followed by lengthy litigation, and the return of Casale to Idros at Terni. At Terni there were different problems. There, barriers to the supply of electricity were created, in part because the ammonia technology was perceived to be highly disruptive to another, well developed, local nitrogen process, that of calcium cyanamide. Compromises were reached. The outcome was the almost simultaneous foundation in 1921 of two companies, Ammonia Casale SA, in Lugano, Switzerland, which handled international licensing, and the Società Italiana per l'Ammoniaca Sintetica which absorbed the Idros works. In 1922 SIAS acquired a mothballed hydroelectric factory at Nera Montoro. It would serve as the industrial testing site for improvements in Casale's process. The widespread, and rapid, dispersal of Luigi Casale's highly successful synthetic ammonia process would become an outstanding example of technology transfer from Italy during the early 1920s. This transfer included, through Leprestre, to the United States, as recently described in *Substantia*. The origins of the transatlantic interest can be discerned in the very earliest attempts by Casale to develop his novel ammonia technology, as described here.

Keywords: Luigi Casale, René Leprestre, Nitrogen fixation, Italian Chemistry, Terni, Casale Ammonia Process

Introduction

The three most important plant nutrients are nitrogen, phosphorus and potassium. While mineral sources of phosphorus and potassium are abundant and readily accessible, this is not the case for nitrogen even though it constitutes the greater part of the earth's atmosphere. In the nineteenth century the main source of nitrogen fertilizer was South America, first the native guano (accumulated bird droppings), and then sodium nitrate, extracted from rocks in the desolate Atacama Desert. Concerns in Europe that the nitrate might become depleted early in the twentieth century stimulated research into methods for fixing atmospheric nitrogen. Two processes requiring inexpensive electricity were introduced in 1905. However, they were restricted to regions where continuous sources of cheap hydropower was available. The situation changed in 1913 with the inauguration of the Haber-Bosch process for the production of synthetic ammonia by BASF in Germany. It is considered to be one of the greatest triumphs of twentieth century technology. However, the process was proved not through its ability to feed the worlds' population but through its use in the manufacture of munitions by Germany during World War I. The Allied nations failed in their elusive quests for breakthroughs in replicating the German technology. After the Armistice, BASF refused to license the Haber-Bosch process, which was widely considered essential for maintaining national security in both agriculture and defence. This encouraged the development of wartime research in order to achieve success in the fixing of nitrogen as synthetic ammonia. As a result, by the early 1920s, three processes had been developed, by Georges Claude in France, and Luigi Casale and Giacomo Fauser in Italy. It is noteworthy that the Claude and Fauser processes feature more in the technical and historical literature than the Casale process*. Perhaps this is because they were associated with, and promoted by, large corporations (Air Liquide and Montecatini, respectively). Yet the Casale process, by completely overcoming problems of fractures in the outer walls of converters (as in the Claude process), was the first to achieve a high level of sustained performance, and, moreover, through licensing arrangements and supply of the complex machinery (unlike Montecatini at least until the mid-1920s), to launch the global synthetic ammonia industry. This was no mean achievement, requiring safe operation under brute force conditions and the production of pure gases, nitrogen and hydrogen. How the lone-inventor Luigi Casale was enabled to achieve this pre-eminence is the remit of this paper. It involves Casale's engagement with two wartime startup companies, the influence of a Franco-American entrepreneur, who was also an influencer in gaining business from the Italian government, disputes over contracts, and litigation, and negotiations with a firm that saw the Casale process as a disruptive technology and prevented access to the critical hydroelectric power needed for driving machinery and for electrolysers that produced pure hydrogen. The outcome was the founding in 1921 of two companies that represented Casale interests, one to promote licensing outside Italy, the other to take up manufacture and onsite testing on a scale that overcame the limitations of Casale's Terni workshop.

The nitrogen problem with special reference to Italy on the eve of the First World War

From the 1840s, an important source of nitrogen as agricultural fertilizer was guano, vast amounts of bird droppings, imported mainly into Great Britain from the Chincha Islands off Peru, and marketed throughout Europe. The peak of imports into Europe was reached in 1870 (280,000 tons); the progressive depletion of the richest deposits of guano led to its replacement from around 1880 with sodium nitrate, also exported from South America, mainly to Great Britain, but now from Chile.[1] The nitrate was also exported to North America. A significant advantage of Chilean nitrate was its flexibility of use: in addition to being used as fertilizer for agriculture, it could be transformed into nitric acid for the manufacture of explosives and organic products such dyestuffs.[2]

Relevant here is the fact that in the early 1900s only a small fraction of nitrate, about 50,000 tonnes per year, were imported into Italy. This resulted in insufficient fertilization of Italian land; in fact, the average consumption of nitrate per hectare in Italy was significantly lower than in other European nations, representing one twenty-fourth that of Belgium, one eighth that of Germany, and about half of that consumed by France.[3] The deficit contributed to low agricultural productivity, with a grain yield of barely 20 quintals per hectare in some regions, a figure which, although similar to the German average, was still below that of Great Britain. As a result, dependence on foreign imports of wheat remained high, highlighting the need for more intensive agricultural activity and for the production of nitrogen fertilizers in Italy in order to ensure food self-sufficiency.

Another important fertilizer in Europe in the late nineteenth century was ammonium sulphate, obtained mainly from gas works where coal was distilled for the production of illuminating or coal gas. For the gas and coking plants, the recovery of ammonia and the production of the sulphate was considered a secondary activity compared to the more profitable production of gas. Nevertheless, in 1913, 433,000 tons of sulphate were produced in Britain, most of which was exported. Of this amount, only 30,000 tons was destined for Italy, where national production of sulphate was estimated at around 5,000 tons.[4]

Chile’s sodium nitrate retained its role as the main source of nitrogen fertilizer for Italy and for much of the world. However, at the beginning of the twentieth century there was a widespread fear of the depletion of nitrate reserves within just a few decades. This created a nitrogen problem, that took on considerable urgency in industrialized countries, where projections of population growth suggested that they were heading towards a food crisis of potentially catastrophic dimensions. In 1898 Sir William Crookes, then president of the British Association for the Advancement of Science, in a highly publicised address before the association, made an urgent appeal for a solution to what he called the “Wheat Problem.” There was, he opined, "a glimmer of light into darkness and despair," namely through the *fixation of atmospheric nitrogen.*[5]

The fixation of atmospheric nitrogen, especially its combination with other elements to create stable compounds suitable as fertilizers, captured the close attentions of several scientist and entrepreneurs.

Though the reaction between nitrogen and hydrogen to produce ammonia is represented by a simple equation, it presented considerable difficulties. The first attempts for production of “synthetic nitrates,” involved combination of nitrogen with oxygen rather than hydrogen and took place in electric arcs.

In this method, introduced successfully in 1905, atmospheric nitrogen was reacted at temperatures of about 2,000-3000 °C with oxygen under the action of powerful electric discharges produced by a direct current ($N_2 + O_2 \rightarrow 2NO$). This process generated oxides of nitrogen which were further oxidized and then dissolved in water, forming a solution of nitric acid from which a wide range of nitrates could be derived ($3NO_2 + H_2O \rightarrow 2HNO_3 + NO$). Because the main disadvantage of the electric arc process was its high consumption of electricity, factories using this technology were predominantly located in Norway, where there was sufficient low cost hydropower. The main product was *Norgesalpeter*, a form of calcium nitrate $Ca(NO_3)_2$ that was widely exported (about 115,000 tons in 1913, of which 3,000 tons arrived in Italy) and that had an efficacy in soils similar to that of sodium nitrate.[6] However, because market prices were determined by the price of Chilean nitrate, this process was not successful elsewhere due to the prohibitive energy costs.

Another means for fixation of atmospheric nitrogen was by the electrothermal production of calcium cyanamide from calcium carbide in electric furnaces, a process invented in Germany by the chemists Adolph Frank and Nikodem Caro.

$CaC_2 + N_2 \rightarrow CaCN_2 + C$

Again, the main constraint was represented by the high energy consumption. However, this electrothermal process was important in Italy, which though it suffered from a scarcity of fossil fuels, particularly coal, possessed the water resources needed to produce hydroelectric power. The cyanamide process, significantly, required much less electricity than the arc process (12,000 kW per ton of nitrogen compared to 61,000 kW), and cyanamide was a useful nitrogen fertilizer. Also, treatment of calcium cyanamide with superheated steam gave ammonia, from which ammonium sulphate fertilizer could be produced.[7] The first large cyanamide factories were set up north of Rome in the industrial city of Terni, in the Conca Ternana region of southern Umbria, also in 1905.

At Terni, surrounded by hills and mountains, the Nera and Velino rivers were two of the main engines of Italian industrial development, favouring the construction of a weapons factory and the nation’s first steel-making factory, where rails for the expanding railway network were manufactured. Exploitation of hydraulic energy (hydropower), known as "white coal", for the production of cheap electricity, led to the establishment of the first plants for the production of calcium carbide by the Società Italiana per il Carburo di Calcio e Altri Gas (Carburo, established in 1896).

Carburo emerged as a leader in the Italian and international markets for calcium carbide and its derivatives, in particular acetylene, which was widely used for public lighting in cities. After the efficient transmission of electricity over great distances became available, the use of acetylene declined, and carbide took on a new role. Carburo acquired the patents of Frank and Caro for the conversion of carbide to calcium cyanamide. Frank and Caro, with mainly German backers, established the Società Generale per la Cianamide, in Rome, an organisation that licensed the cyanamide process worldwide. In 1914, 15,000 of cyanamide tons were produced in Italy.[8]

However, the main breakthrough in nitrogen fixation occurred in Germany, at the technical institute in Karslruhe. There, in 1909, the physical chemist Fritz Haber brought about the direct synthesis of ammonia from its element on the laboratory bench.[9] Because the yield was low, the process was designed to be continuous, with recirculation of unreacted gases. The apparatus, including the reaction chamber (converter) was made of steel. BASF, of Ludwigshafen, the leading chemical company in Germany, took on the challenge of industrial scale up of Haber's invention. This included design of the necessary equipment to withstand the high pressures and temperatures of a reaction that took place in the presence of an active but inexpensive catalyst, and apparatus for production of pure hydrogen and nitrogen. A team of chemists and engineers under the direction of the engineer Carl Bosch overcame a slew of difficult technical problems, especially overcoming difficulties with the steels then available, and in the design of innovative gas compression machinery. The best catalyst was found to be iron, containing promoters. In September 1913 the full-size industrial plant, at Oppau, near Ludwigshafen, produced the first quantities of synthetic ammonia. The gas was cooled by refrigeration to give the anhydrous product. What became known as the Haber-Bosch process operated at a pressure of 250 atm and a temperature of about 500° C.

$N_2 + 3H_2 \rightleftarrows 2NH_3$.

The converter was the core of the Haber-Bosch process. The mixed nitrogen-hydrogen gas, under the high pressure, was introduced into its lower part and forced through a tubular heat exchanger where it was preheated by gas leaving the reaction area. The hot charge of gas then rose through a central tube to the top of the converter and was directed down into the catalyst chamber where the reaction took place. Here, around 8% of the gas mixture was transformed into ammonia. The mixture of gases passed through the preheat tube bundle, after which it was expelled at the base of the converter.[10]

Shortly after war broke out in 1914, Germany's supply of Chilean nitrate was cut off by a British naval embargo. Germany then turned mainly to the Haber-Bosch process for the nitrogen products required in manufacture of munitions. The Allies took note. With the urgent increase in nitrogen demand for the production of explosives and fertilizers during the war period, solving the "nitrogen problem" became critical among all major belligerents. It soon became apparent that in the production of synthetic ammonia the combined technological and chemical capabilities of the Allies was decidedly inferior to that of Germany. Shortages of nitrogen products meant shortages of both explosives and bread. In Italy, in particular, the conflict made imports of sodium nitrate difficult, while production of calcium cyanamide and ammonium sulphate was insufficient to meet the new requirements imposed by the war.[11] In addition, Italy suffered from a shortage of wheat from Russia and Romania following the closure of the Dardanelles.[12] These factors highlighted the need to develop an industry for the production of synthetic nitrogen compounds. But, as elsewhere, there was very little progress, even immediately after cessation of hostilities.

The possibility of adopting the Haber-Bosch process in Italy after 1918, as was the case elsewhere, encountered obstacles due to the policy of BASF in refusing to grant licenses. There was also the need of coal to produce hydrogen as well as nitrogen, according to the BASF processes. Coal was a scarce raw material in Italy. This would have meant dependence on the import of coal to meet nitrogen requirements. However, there was an alternative, production of hydrogen from water by electrolysis, even though electrolysis required a vast amount of electricity.[13] Nevertheless, for Italy, for strategic reasons, the choice between the two processes, coal-based or electrolytic, was not difficult. Production of synthetic ammonia would draw on electrolytic hydrogen, ensuring that an Italian nitrogen industry would be independent of imports of fossil fuels. Also, the large-scale increase in the production of synthetic ammonia would lead to a significant reduction in unit costs of nitrogen as compared with the arc and cyanamide processes.[14]

As a result, several Italian chemists undertook research to develop a process for the synthesis of ammonia from electrolytic hydrogen. Among them, Luigi Casale emerged as the most prominent figure.

Luigi Casale and the pilot plants in Terni and Pieve Vegonte

Luigi Casale was born on 22 November 1882 in Langosco Lomellina (fig. 1).[15] He graduated in chemistry at the University of Turin, where he took the Advanced Course of Physics, Chemistry and Electrotechnics, under Professor Arturo Miolati. Subsequently, he began his academic career as an assistant at Turin, and later at the Institute of General Chemistry and Electrochemistry. During 1912-1913 he undertook research in Berlin under the guidance of physical chemist Walther Nernst, famous for his studies on physical chemistry, including the equilibria in gas reactions when carried out under various conditions, and to a great extent paralleling the studies of Fritz Haber.[16] In Berlin, Luigi was joined by his wife Maria Sacchi, also a chemist.

After Italy joined the Allies in 1915. Luigi Casale moved to Naples and joined the university, where he collaborated with Oscar Scarpa, who taught physical chemistry, and had a strong interest in nitrogen fixation. Scarpa was also director of the Electrochemical Laboratory of the Royal Polytechnic High School of Naples. In addition to their joint interests, Casale engaged in research into asphyxiating gases and protective measures for the war effort at the university's Institute of Pharmaceutical Chemistry and Toxicology, directed by Arnaldo Piutti.

Figure 1. Luigi Casale as a young man. (Collection of Francesco Casale)

While undertaking this research, Casale became the victim of severe poisoning while manipulating a toxic gaseous mercury compound. This incident, which took place in 1917 (and would lead to his untimely death in 1927), forced him to abandon the study of asphyxiating gases and he decided to devote himself to the fixation of atmospheric nitrogen as synthetic ammonia, at first working closely with Scarpa. Both Miolati and Scarpa had taken up an interest in nitrogen fixation even before the war at a time when chemistry departments in Italian universities and technical institutes were increasingly dealing with industrial problem solving. This new direction for Casale was probably suggested by his mentor, Miolati, a prominent figure in the commissions established during the war to solve the urgent needs related to war materials of various kinds, including explosives and products for the chemical industry and agriculture. But even more significant was the fact that Casale's time in Berlin had introduced him to the physical chemistry of gas reactions, including between hydrogen and nitrogen.

Casale soon moved to the city of Terni, where, no doubt on the recommendation of Miolati, he joined Idros, an important chemical company that by electrolysis of water produced hydrogen for the Italian navy for use in dirigible and other aerial balloons, and oxygen for oxyacetylene welding. Società Idros (its name derived from idrogeno and ossigeno) was founded in Bologna on 19 February 1916.[17] The objects of establishment of Idros included, in addition to production of hydrogen and oxygen, the extraction of nitrogen from the atmosphere and generation of electricity. Prominent members of the board of directors were René Lepreste, as president, the lawyer Mario Santangelo, as CEO, and Carlo Andreucci, as technical expert.[18] Leprestre, a Franco-American entrepreneur, based in New York City, had played a key role in the creation of Idros specifically for production of certain industrial and military chemicals (fig. 2).

Figure 2. Portrait of René Leprestre, in 1916. (Elizabeth Burrows [family] Collection)

Leprestre profited from various business activities conducted during the war. He appears to have travelled abroad frequently. In the summer of 1916, he visited France, Great Britain and Italy to organise and manage the sale of horses and mules from the United States to European armies. Aware of the facilities offered to industrial enterprises engaged in the war effort by the special war legislation, Idros in November 1916 signed a contract with the Italian Military Administration for the supply of 36,000 240 mm bombs at the price of 123,6 lire each, for a total order amount of £4,490,000[19], and another contract for the supply of 237.75 cubic metres of oxygen per day at the price of 1.1 lire per cubic metre.[20] Around the same time, Idros began receiving orders from the Italian Air Force for production of electrolytic hydrogen at an auxiliary plant. The need for nitrogen products, essential both as fertilizers and for the production of explosives, was particularly critical. Through the inventor Carlo Andreucci, Idros became interested in synthetic ammonia.

Towards the end of 1916 the Idros facility in Terni was enlarged. Shortly after, Carlo Andreucci obtained a patent for the extraction of atmospheric nitrogen by burning a mixture of air and hydrogen.[21] It is therefore likely that Casale moved to Terni in 1917 in order to collaborate with Andreucci on a process for synthetic ammonia that drew on Casale's studies in Berlin and followed the basic chemistry and workings of Haber's method. By July 1917, with the support of Santangelo, who was interested in the technological and economic developments of nitrogen fixation, Casale and Andreucci had developed a method for the continuous production of ammonia in a closed apparatus through recirculation of unreacted gas.[22] Their small experimental apparatus produced a few kilograms of ammonia. The reaction was carried out at temperatures between 350 and 600° C in the presence of a catalyst, as documented in the patents filed in Italy on 18 July 1917 and in France on 19 October 1917 on behalf of Casale, Andreucci and Santangelo.[23] Although the piping of the ammonia unit was declared suitable for resisting high-pressure gases, the pressure range used was not specified, presumably to prevent possible imitations. Only later would it be revealed that Casale employed a considerably greater working pressure than Haber, with as, Casale calculated, a much greater yield. The nearby Terni steelworks provided a suitable steel pressure vessel, as converter, which according to later accounts was the redundant barrel of a naval cannon from the battleship *Dante Alighieri*.

However, under the wartime conditions, Idros, could not spare the hydrogen necessary for Casale's pilot plant research. This was because rights to practically all the local water resources were controlled by Carburo. Idros had access to sufficient water from the Nera to enable production of gases required by the military. Apart from this consideration, Idros might have considered the ammonia process a somewhat speculative venture.

As a result, Casale, on 16 July 1918, entered into a collaborative arrangement for further development with the engineer Alfonso Vitale, the CEO of the chemical manufacturer Rumianca. Rumianca, founded in 1915, was engaged in the production of alkali (caustic soda) and chlorine using the electrolytic Kastner-Kellner process, as well as the manufacture of war gases, including phosgene.

Substantial quantities of hydrogen, for which apparently there was no demand, were produced as a by-product of the electrolysis process. Also, the Rumianca factory included a suitable working area, with idle equipment, including compressors and pumps, and vessels capable of withstanding high pressures. Casale probably already had in mind development of the ammonia process alone (without Andreucci) but he needed adequate equipment and in particular hydrogen. Therefore, the interests of both parties merged: Rumianca's wartime plant included machinery that Casale needed as well as pure hydrogen; Rumianca could possibly benefit from the discoveries of Casale.

The facilities seemed ideal for demonstrating the industrial feasibility of Casale's ammonia process. According to the agreement, Rumianca undertook to provide Casale with the necessary resources to register relevant patents. Casale agreed to keep confidential the practical details of the production process, and Rumianca would cover the related expenses.

Towards the end of 1918, Casale moved to the Rumianca factory, at Pieve Vegonte, in the Piedmont region, where he undertook the construction of his first 100 kg per day ammonia unit. By May 1919, the plant was operational, albeit with intermittent stoppages. Casale filed his first patent relevant to the process on 12 July 1919 (granted on 12 July 1920) for an apparatus suited to the production of mixtures of hydrogen and nitrogen for the ammonia synthesis by burning air in a hydrogen atmosphere. The burning took place inside a combustion chamber, from which the resulting mixture (of water, hydrogen and pure nitrogen) was passed through a refrigerant, enabling ready isolation of the reactant mixture of nitrogen and hydrogen.[24]

The success with the 100 kg unit led to the drawing up of a formal agreement between Casale and Vitale on 17 May 1919. According to this agreement, valid for a period of five years, Rumianca would provide suitable premises, technical and scientific equipment, and qualified personnel for the construction and operation of a semi-industrial synthetic ammonia plant capable of producing 1,070 kg per day. To this end Rumianca would supply 3,000 cubic metres of hydrogen to match up with the requirements of the intended ammonia production. Casale would receive a royalty of 0.1 lire per kg of ammonia produced, reduced to 0.08 lire per kg if the production exceeded 1,500 kg per day. In addition to the royalty, Casale was guaranteed a monthly allowance of 1,500 lire, valid until 31 March 1920.

This agreement took place at a time when Italian factories were going through a major, and expensive, crisis in converting and reverting to peacetime needs, which included seeking opportunities to exploit by-products. It was anticipated that the market for nitrogen fertilizers, especially in Italy, would be dominated by calcium cyanamide. Ammonia was not considered a major contender for satisfying the nitrogen fertilizer demand. Synthetic ammonia was a gamble for Rumianca, and probably considered suitable for distribution to the alkali industry and for refrigeration, and certainly not for large-scale production as would be required in the manufacture of fertilizer. Rumianca in any case had to restrict production of ammonia to the maximum amount of byproduct hydrogen (3,000 cubic metres per day). There were no plans to increase production of alkali which would enabled production of more ammonia. These were difficult times. Because of the post-war crisis, Rumianca, in common with other Italian chemical companies, was facing financial difficulties.

This is perhaps why, in exchange for his services and commitments to Rumianca, Casale was granted the right to bring in other parties prepared to invest in Rumianca's ammonia. There was, however, an important caveat in the agreement, regarding certain business conditions: It had to be ratified by 30 September, at which time Rumianca would need to have provided all the machinery and made available the necessary daily supply of hydrogen to enable production of ammonia at the rate of a calculated 1,070 kilograms per day.

Notwithstanding the confidentiality agreement with Rumianca, Casale maintained close relations with Idros, especially with Lepreste, who had been busy developing business networks with political and banking representatives in Italy and the United States. In 1917, during his stay in Italy, Leprestre had established important political and military relations, in particular with the Minister of Trade Francesco Saverio Nitti.[25] Their negotiations focused mainly on agreements aimed at accelerating the process of Italian industrialization to support the war effort, especially through the use of hydroelectric energy, and to ensure the supply of war material from the United States. This no doubt brought in more business for Idros and may have led to Leprestre's meeting with the Italian-American banker Vincent Handley. Handley was company secretary of the American Italian Commercial Corporation, established at the behest of Banca Commerciale Italiana in New York in 1917 to facilitate trade in steel and ships destined for Italy.

By May 1919, Lepreste, from whatever information he had gleaned, understood that the ammonia method developed by Casale was proving successful and could potentially constitute a means to challenge the monopoly of the BASF Haber-Bosch process for production of synthetic ammonia. On 11 July 1919, Casale and Lepreste signed an informal agreement, within the terms that Casale had agreed in May with Rumianca, giving Leprestre the right to visit the ammonia plant at Rumianca. Three days later, on 14 July, Lepreste accompanied a group of American investors interested in Casale's process on a visit to the Rumianca plant. The participants were a Mr Scheffey, a Dr Fred Chamier and Vincent Handley. With them was Giuseppe Bruni, professor at the Royal Polytechnic of Milan and the Royal University of Milan, who was charged with providing testimony and advice concerning Casale's process as an independent expert. On a second visit a week later, the same

American group was impressed by the claim that an average rate of production of 1,070 kg per day for three months had been achieved (which was not the case).

Consequently, on 2 August 1919, Casale and Lepreste signed a formal, secret agreement for the commercial exploitation of the Casale process for the synthetic production of ammonia, before the notary Agatocle Mollaioli of Rome.[26] In the contract, Casale clearly specified that he excluded Carlo Andreucci and Mario Santangelo from any involvement, reserving exclusively for himself and Lepreste ownership of the two Casale inventions through their patents, one for the production of synthetic ammonia and the other for production of a mixture of hydrogen and nitrogen, for both Italy and abroad. Casale also declared in the agreement with Lepreste that he had entered into an agreement with Rumianca, conditional on ratification within sixty days, specifying the quantity of ammonia produced, and accepting the clause covering protection of industrial secrecy. However, Casale retained the right to ask third parties to examine the existing or future facilities he developed at Rumianca, with a view to investing in Rumianca's ammonia business.

In addition, according to the agreement [with Leprestre], Casale would allow Bruni, the technical expert, in accord with the Casale-Rumianca agreement, to check data from the plant until late September 1919, in order to determine the industrial cost for each kg of synthetic ammonia produced. On the basis of a satisfactory report prepared by Bruni, Lepreste would be committed to pay Casale the sum of one hundred thousand lire as compensation, in addition to an additional four hundred thousand as the initial payment for the acquisition of processes and related patents, for use both in Italy and abroad. It was then reiterated that the agreement with Rumianca would remain in place subject to its meeting the conditions of its contract.

The sums specified by Leprestre as payable to Casale would be handed over only after Casale had explained, in front of Lepreste and two expert chemists designated by the American group, the recipe for the composition of the catalyst, demonstrate the process of its preparation, and provide a satisfactory estimate for the cost of operation. The demonstration would involve placing the catalyst in a sealed tube, under the supervision of those present, and its insertion into the converter, followed by verification, by observation, of its industrial performance. Once the industrial cost per kg of anhydrous ammonia and the performance of the catalyst had been demonstrated on a semi-industrial scale, the American group would undertake to: acquire and protect the patent; guarantee to invest ten million lire in Rumianca for developing a process capable of manufacturing ammonia at the rate of 20 tons per day; and also promote the process in the United States.[27] The American investors would invest in the Casale process by selling licenses and generating royalties in the United States. Rumianca would receive an injection of funds in order to scale up production to 20 tons of synthetic ammonia per day, and perhaps the investors would acquire a controlling interest in Rumianca, because ten million lire was four times the share capital of Rumianca. In these circumstances, Rumianca would quickly gain the synthetic ammonia monopoly in the Italian market.

If, however, Rumianca was unable to fulfil its part of the agreement, that is, by 30 September, Casale would declare the prior agreements void, and move to Idros at Terni to build an industrial ammonia unit. There, Lepreste would provide all the necessary means in order to allow Bruni to gather data on the spot. In addition to the total compensation of five hundred thousand lire, Lepreste would pay Casale 20 % of the profits deriving from the commercialization of patents. In addition, Lepreste would provide Casale with all the necessary means to establish a chemical laboratory in Terni for the industrial development of the catalyst, and supply the recipe to the companies that adopt the Casale process. The laboratory would also conduct further studies and research under the direction of Casale, who would receive a monthly allowance of 1,500 lire and 20 % of the net profits, in addition to the foregoing amounts, on income arising from innovations made at the laboratory.

It was probably in August 1919 that relations between Casale and Vitale began to deteriorate, probably as a result of the CEO of Rumianca learning of the secret contract between Casale and Lepreste. This would explain why Vitale started getting in the way of Casale's efforts. As a result, in a letter dated 25 August 1919, Casale reminded Vitale that the conditions agreed in May had not yet been realised. The earlier agreement was based on the strict caveat: the deadline for the finalisation of the agreement, based on the working of the process, was 30 September. Casale stated that in the absence of the agreement being concluded he would cease all cooperation with Rumianca.[28] Vitale's response, on 13 September 1919, emphasized the need to make some amendments to the convention, since, in his opinion, the industrial feasibility of the Casale process had not yet been demonstrated.[29]

In October, Casale replied, informing Vitale that he had appointed his legal counsel, the lawyer Mario Santangelo, to negotiate with the vice president of Rumianca, the prominent engineer Lorenzo Allievi, to permit only those changes deemed fair and reasonable. Santangelo was confronted with an outright rejection by Vitale. The parties could not come to an arrangement for permitting the requested changes. Rumianca filed a lawsuit against Casale, claiming that he had reneged on their contract. Casale submitted to the court a detailed expert report drawn up by Professor Bruni. The litigation would continue for a decade and is described later. Casale moved to Terni.

The return to Terni and the agreement between Casale and Carburo

In contrast to the experimental plant at Rumianca, for which less power was available, and where operation was intermittent, and whose converter under the high-pressure conditions was subject to cracks when exposed to temperatures above 400º C (as a result of hydrogen attacking ordinary steel), the Terni plant operated with regularity and stability thanks to the design of an internally heated catalyst tube, according to the specification patented in September 1920.[30]

Luigi Casale's converter, as perfected after his return to Idros, became the standard model.[31] It was comprised of a number of concentric tubes. By flushing the inner wall of the outer tube, or shell, of the converter with cold synthesis gas, it was maintained at a temperature which greatly reduced the stress on the tube. This was the feature which permitted the use of ordinary steel for the outer wall and overcame the problem of fracture, caused by hydrogen. The innermost tube was the catalyst bed. It surrounded the electrical heating element. Heat from the catalyst bed warmed up the incoming gas mixture. In the words of the chemical engineer Bruno Waeser, "The synthesis tube [converter] is constructed so as to ensure efficient heat interchange, and the pressure container [outer tube] itself never attains a temperature higher than 200℃. This low temperature enables special steels to be dispensed with in the design of the catalyst bomb".[32]

The compressed nitrogen-hydrogen gas-feed entered at the bottom of the converter, and flowed up, in the annular space between the inner wall of the converter shell and the corrugated partition. At the top, the gas reversed its direction and flowed down the next annular space, where it came into contact with the outer wall of the inner catalyst tube. Here it absorbed heat from the exothermic reaction. At the bottom the gas again changed direction, this time entering into the catalyst tube where, within the catalyst bed, the ammonia was formed (fig. 3).

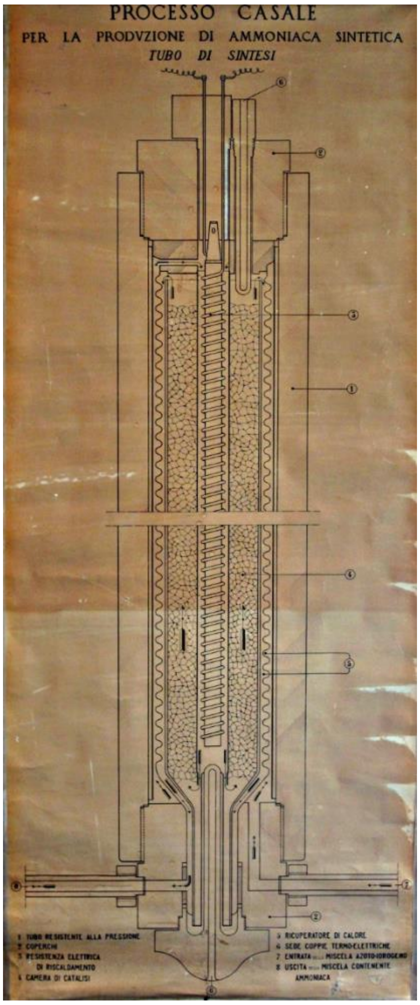

Figure 3. Casale ammonia converter, 1920s.

As in the Haber-Bosch process, a gas recirculation pump, for return of the unreacted gas mixture to the converter, was incorporated into the early synthesis loop.

Another important advantage of the use of very high pressures, as Casale had calculated, was the increased yield, 15-18%, compared with around 5% for the Haber-Bosch process. The yield, in fact, was greater than expected by theory. This meant that less gas was pumped and circulated to produce the same volume of ammonia as in the Haber-Bosch process. In the synthetic ammonia processes at the lower (Haber-Bosch) pressure range, at 200 to 350 atm, the iron (magnetite) catalyst had to be specially formulated with the addition of promoters, or activators, which were oxides of certain metals, to ensure high activity. At very high pressures, a less active catalyst, though still incorporating promoters, was used, as was the case in the Casale process, which required around one-seventh the amount of catalyst as compared with the Haber-Bosch process. At Casale's working pressure of 800-850 atm, it was possible to separate out anhydrous liquid ammonia from the gas mixture by simply cooling with water. Thus, because of the high working pressure, there was no need for a refrigeration compressor for separating out liquid ammonia; the condenser operated at the water-cooling temperature. This was a major advantage over the Haber-Bosch process, for which expensive refrigeration equipment was necessary in order to produce anhydrous, liquid ammonia from the gaseous product mixture. Moreover, direct production of liquid ammonia under Casale's conditions involved more compact units than were required in the Haber-Bosch process.

Lepreste showed full satisfaction and provided additional funding to Casale for the study and erection of a plant capable of taking 3,000 cubic metres of hydrogen daily, equivalent to the design capacity of the Rumianca facility. For this purpose, a limited liability company, Nitram, was established, which appears to have been intended as a syndicate involving a group of investors brought together created by Lepreste and Handley in order to provide liquidity for Casale's work at Idros. Lepreste's enthusiasm was such that he went to the United States in order to promote the Casale ammonia process, with the intention of attracting investors.

There was one major stumbling block remaining that prevented industrial scale production. In order to generate the necessary electricity for the electrolysers and for driving compressors and other machinery, Idros was required to negotiate with the Municipal Electricity Company of Terni. However, the electricity company was forbidden from supplying energy to any chemical company competing with the cyanamide manufacturer Carburo, according to a 1910 agreement between the Municipality of Terni and Carburo. As a result, Carburo invoked the agreement, thus preventing the supply of electricity to Idros.[33] This move most likely followed information provided by Lorenzo Allievi, of Rumianca, to Carburo, following the falling out with Casale. In addition, the Casale process was now seen as a strong competitor to the cyanamide process. Delicate negotiations were required to bring about a change at Carburo. If Carburo became involved with the Casale process, it was argued, Carburo would have a cost and energy saving alternative to the production of ammonium sulphate, as compared with calcium cyanamide. Another consideration was that the Frank-Caro cyanamide patents were about to expire.

The outcome of the deliberations was a compromise. For Idros, which desperately needed the energy to operate the semi-industrial plant of Casale, it became necessary to meet certain demands of Carburo. On 3 January 1921, Carburo and Idros signed an agreement that allowed the former to inspect the Idros unit for the production of synthetic ammonia. Once convinced of the effectiveness of the Casale process, Carburo acquired exclusive rights to the Casale patents, valid for Italy, for the countries of the former Austro-Hungarian Empire, and for Romania. On 23 April 1921, Carburo and Idros together established a new company.[34] This was based on reorganisation of Idros as a new entity, the Società Italiana per l'Ammoniaca Sintetica (SIAS), in which the Carburo would direct the industrial programme, while Casale would assume the role of scientist-entrepreneur. Nitram was absorbed into SIAS. Idros would increase its capital to 23 million lire, divided into 12 million invested by Carburo (4 million of which would be used to pay off Idros's debts, and 8 million remaining available for investment in new plants); 5 million as compensation for the Casale patent; and 6 million as the previous capital of Idros.[35] The Board of Directors of SIAS was composed of President Riccardo Bianchi, engineer and former head of the Italian State Railways, Arturo Bocciardo, engineer and CEO of the Terni – Società per l'Industria e l'Elettricità, Luigi Casale, and Pietro Fenoglio, engineer and board member of the Banca Commerciale Italiana; the CEO was Vincent Handley, the secretary was Ranieri Pontecorvi, and the technical director was engineer Osvino Ranieri Tenti.

A few days later, on 27 April 1921, Casale and Leprestre founded Casale Ammonia SA in Lugano (Switzerland) with the aim of acquiring, exploiting and protecting patents abroad for the production of synthetic ammonia according to the processes devised by Casale. Of a total of two hundred 500-franc shares issued, Casale held seventy-eight and Leprestre ninety. The Board of Directors was composed of three members, namely Lepreste (president), Giuseppe Albisetti (colonel and merchant of Massagno), and Tommaso Quadri (architect of Lugano).

Table 1. Events in the Development of the Casale Ammonia Process

Year	*Main events*
1915	Establishment of Rumianca, at Rumianca (Pieve Vegonte).
1916	Establishment of Idros at Terni, for production of hydrogen and oxygen for the military. The president is René Leprestre.
1917	Casale joins Idros, on recommendation of Miolati. Casale collaborates with Carlo Andreucci on synthetic ammonia.
1918	Casale moves from Idros to Rumianca because Terni does not produce surplus hydrogen. For ammonia studies. Rumianca makes available redundant equipment, and by-product hydrogen from its alkali plant.
1919	In May, Casale demonstrates synthesis of synthetic ammonia at the rate of 100 kg per day. Casale and Rumianca draw up a contract, signed on 17 May, specifying production by 30 September 1919 of one ton per day. Casale still owns his patents but agreed not to reveal confidential technical information for working the process developed at the Rumianca factory. Casale is allowed to deal with outside investors. In July, Leprestre visits Rumianca with Giuseppe Bruni, his expert, and representatives of a US group prepared to invest in Rumianca's ammonia, and promote the process outside Italy, subject to

	satisfaction of the conditions laid down in the Casale-Rumianca contract. Things fall apart because on 11 July Leprestre and Casale came to a separate agreement over control of the ammonia patents, and this was discovered by Rumianca, Rumianca in any case could not achieve the agreed output. The Casale-Rumianca agreement was declared void. Late in 1919 Casale returns to the now closed Terni works where he now has sufficient working space, and a supply of hydrogen from the electrolysers there.
1920	In April, Casale's converter produces about 200 kg of ammonia daily. In June Casale completes the Terni ammonia unit and in July and September he obtains two patents for the ammonia process.
1921	Ammonia Casale SA and SIAS are established. SIAS is created from Idros and calcium cyanamide manufacturer Carburo. Casale receives his first order for industrial scale synthetic ammonia machinery from the Japanese cyanamide manufacturer Shitagau Noguchi.
1922	A 2 metric ton per day converter is installed at Terni (September). SIAS acquires access to larger premises at Nera Montoro for testing and development in collaboration with Ammonia Casale.

The official constitution of SIAS took place on 22 May 1921, with headquarters in via Due Macelli 66 in Rome, the headquarters of Carburo.[36] On 29 September 1921, the SIAS factory in Terni was visited by ministers Alberto Beneduce (Minister of Labour) and Luigi Gasparotto (Minister of War), who paid tribute to Casale for his hard work and dedication and gave great praise to Lepreste for recognising the potential of the Casale process and for investing considerable sums for its development.[37] Around the same time, Casale was negotiating a licensing agreement with Japanese engineer Shitagau Noguchi, an entrepreneur active in the production of calcium carbide and calcium cyanamide on the island of Kyushu. This agreement was finalized in December 1921 with the payment of 10 million lire to Ammonia Casale.[38] Noguchi had been introduced to Casale by Enrico Cairo of Carburo.

In September 1922, a 2-metric ton per day Casale ammonia converter was installed at Terni. This proved of tremendous value in demonstrating the capabilities, and reliability, of the Casale process. To develop and test larger converters it became necessary to gain access to additional space. Carburo had already given full backing to the expansion of SIAS activities. In 1922, SIAS leased the substantial Nera Montoro electrochemical plant that had been inactive for some years.[39] This would enable development work on ammonia converters, in 1923 of 7.5-tons per day capacity, equivalent to 24,000 tons per year of nitrogen, and, within three years, to converters of 20-tons per day capacity.

The selection of the Nera Montoro site was not random. The Montorese plain is characterized by the rich availability of hydropower, ideal for the production of electrolytic hydrogen, the most important raw material for the synthesis of synthetic ammonia, and of electricity for operating the machinery, including electrolysers. The factory site, owned by Società Idroelettrica di Villeneuve e Borgofranco (Villeneuve), was specifically designed for electrochemical production, such as chlorine and aluminium. Therefore, much of its existing machinery could be readily reused or adapted for production of hydrogen. In addition, the local staff of peasant origin had accumulated considerable experience in chemical processing during the war and were a valuable source of cheap and semi-skilled labour. Nera Montoro was the ideal location for expanding and developing the electrochemical process associated with Luigi's Casale's ammonia process. Within a short time, orders were placed for the additional equipment needed in the synthesis of ammonia at Nera Montoro.

The Litigation between Rumianca and Luigi Casale

Following the inability of Rumianca to meet the 30 September 1919 production target, and Casale's refusal to allow extra time, Rumianca built up a legal case for claiming compensation for loss of business from Casale. The resultant claim went as follows: Casale challenged Rumianca on two main issues. The first concerned an estimate of the potential production of synthetic ammonia at Rumianca from September 1919 to May 1934, taking into account both the existing equipment and what could have been developed using around 3,000 cubic metres of hydrogen per day. At the same time, it was necessary to determine the percentage of royalties owed to Casale, based on this higher production. The second issue concerned the impediment placed by Rumianca towards Bruni, in charge of verifying the industrial effectiveness of the Casale process on behalf of an American group. Moreover, it was argued, Rumianca's inability to satisfy the conditions of the contract was contested because it had led to delays in the implementation of new plants and in entering into licensing agreements based on the Casale patents. Consequently, the crucial aspect was to determine whether, and for what specific reasons, Casale had lost the competitive advantage in cornering the production and consumption of ammonia in different countries when compared with other similar and competing processes, and if this had harmed Casale's interests by causing financial loss.[40] Rumianca's lawyers took Casale to trial in order to contest his claims.

The litigation was first tried before the Court of Pallanza, and then before the Court of Appeal of Turin. The Court of Pallanza, in elaborating on its verdict, declared that Casale had provided exhaustive evidence of the efficiency of his process.[41] The two court rulings, delivered on 30 December 1922 and on appeal on 2 July 1923, respectively, established that Rumianca was obliged to compensate Casale on the basis established by the agreement of 17 May 1919 for ammonia that could have been produced, as well as for damages due to the failure to conclude the contract, amounting in all to 179 thousand lire for unpaid consultancies and a further 660 thousand lire for unpaid royalties. The final judgement was issued by the Court of Appeal in April 1929, after review of a series of statements and investigations requested by a panel of experts appointed by the court the previous year. This panel of experts was composed of professors Angelo Menozzi and Felice Garelli, as well as the engineer Eduardo Ferrua.

In the defence memorial presented by Rumianca, and signed by the then CEO Ostilio Severini, it was explained that the measure of success of the plant could be determined only by the prospects for marketing ammonia. It was also argued that it would be easy to replicate the same experimental plant in Terni, stressing that Casale could make further improvements by virtue of the specifications of his 1920 patent, which was earlier than those of Fauser and Claude. Therefore, the defence of Rumianca argued that the alleged losses complained of by Casale were totally non-existent.[42]

In response, Casale, represented by Bruni, argued that the failure to comply with the agreements had caused a delay in the development and expansion of the Casale process both in Italy and abroad, thus nullifying the technological advantage. In fact, it was already clear in May 1919 at the Rumianca plant that the industrial efficiency of the process could then have been demonstrated, and this was well before the time required for the completion of the Terni plant in June 1920 (started in November 1919). The need to regain Lepreste's trust, and the agreements with the companies that led to the establishment of SIAS in July 1921 to exploit a daily production of one ton, resulted in a delay of two years in the industrial application of the Casale process. Meanwhile, Claude (whose patent was filed in March 1917, just before that of Casale) had already started a plant in Montereau and on 2 February 1920, had demonstrated a working unit before the French Academy of Sciences with production of 6-7 litres of liquid ammonia per hour. Claude had exploited very high pressures, around 1,000 atm, to obtain higher concentrations of ammonia but confronted considerable difficulties in constructing the necessary equipment. On 26 November of the same year, by means of a different catalytic tube system, he was able to produce 60-70 litres per hour. Finally, on 21 April 1921 a plant was under construction for production of 5 tons of anhydrous ammonia per day. Fauser, in the meantime, had obtained his patent and was working to persuade his potential backers. Consequently, if the Casale process had been properly consolidated two years earlier at Rumianca, it could have established itself considerably earlier as a predominant method worldwide by virtue of its technical merits.[43]

According to Bruni, the construction of a plant similar to that of Rumianca in Terni was initially delayed due to the lack of hydrogen and adequate equipment (compressors and refrigeration pumps), but especially the converter, which had already been ordered by Rumianca from the engineering firm Ansaldo.[44] The panel of experts recognized that the installation of the catalytic tube (converter) would have made the process operational by the end of October 1919, thus allowing an effective production of synthetic ammonia from 1 November. The main plant, designed to absorb the 3,000 cubic metres of hydrogen, if made available, could have operated at full capacity by 1 November 1920. As a result, it was decided that from this date onwards full production capacity based on the amount of hydrogen available should be taken into consideration. With Casale's estimate that 2.6 cubic metres of hydrogen were needed to produce one kg of ammonia, a total daily production of about 1,150 kg of ammonia was calculated. It was estimated that in the fifteen-year period 1919-1934 about 5,400 tons of ammonia could have been produced. This production would have generated for Casale an estimated income of 580,000 lire, calculated on the basis of 0.1 lire per kg, and rounded up to include interest payable.[45]

In 1929, the panel of experts also recognized delays forced on Casale in developing his invention and the resulting loss of pre-eminence to Claude and Fauser. This situation led to considerable difficulties in acquiring the markets for production and consumption, both because of the emerging competition and the discredit that Casale suffered by the actions of Vitale, in addition to the widespread defamation in judicial, scientific and industrial circles. Moreover, the obstacles encountered by Casale following the legal dispute—and taking into account Claude's possible claim on priority—may have influenced Montecatini's decision to opt for Fauser. Guido Donegani, the Montecatini's CEO, was an advisor to Carburo and therefore had an intimate knowledge of the Casale process.[46]

However, determining precisely the extent of the material and non-material damage proved complex, except for the loss of the income relating to the 20-ton plant, which was to have been financed by American investors but which, because of the complications introduced by Rumianca, was never realized. Consequently, the experts, with a majority of two to one, came to the conclusion that Rumianca was responsible for the non-fulfilment of the conditions of its agreement with Casale and therefore Rumianca was ordered to pay the sum of two million lire to Casale. In total, it was estimated that the company would have to compensate Casale for an approximate sum of two and a half million lire. According to the ISTAT converter,[47] two and a half million lire of 1929 would correspond to more than two and a half billion euros today (to be precise, 2,635,649.99 euros).

Conclusion

According to heroic narratives, mixed with a little pure mythology, in the history of science and technology, several individuals are credited with breakthrough inventions. While this may be true, bringing an innovation to market requires multiple actors. Typically, to accomplish this goal the inventor has to convince potential investors and those who place resources at his/her disposal for development of a viable process. The path to success is, invariably, not smooth. There might be disputes over contractual arrangements. Some companies, at least initially, might have little or no incentive to invest in new technologies, especially if they might constitute a disruptive influence. They may even place barriers in the way of potential competitors that are engaged in the same or similar markets. In these circumstances, diplomacy must come into play to overcome prejudices. All these elements are prominent in the development of the synthetic ammonia process of Luigi Casale. The arrangement of his steel converter tube, packed with other tubes, one containing a catalyst, and others that heated and cooled gases—preventing rupture of the outer wall of the converter—and including a heating element, was the basis of a technology that rivalled BASF's Haber-Bosch process. A major incentive for the development of the Casale process was the fact that BASF tightly guarded the secrets of its technology, and its conditions for licensing were so onerous that the Haber-Bosch process was not taken up elsewhere. Casale in contrast, licensed his process globally, and was the first to do so, with considerable success. In that sense he benefitted greatly from the BASF strategy. At the same time, with René Lepreste's encouragement and backing, he was enabled to garner international interest at a time when the Claude process was facing operational difficulties and the Fauser process was serving the needs of Montecatini alone, and within the borders of in Italy

The entrepreneur Leprestre enters the story as the head of the Italian company Idros at Terni founded during wartime for the express purpose of producing gases for the Italian military. Leprestre was a wily businessman who drew on his contacts with prominent Italian politicians to gain contracts for the supply of bombs. He was intrigued by Luigi Casale's ammonia invention and provided support for its development at Terni. However, at Terni the facilities there were inadequate for Casale's work. So Casale moved to Rumianca, which had idle equipment suited to ammonia research, and by product hydrogen that was available. Casale was semi-independent and he did not break his links with Leprestre. Casale's 1919 contract with Rumianca, subject to the firm fulfilling its part of the bargain, gave Rumianca certain exclusive rights, but it allowed Casale to bring in outsiders who would invest in and promote and license the process elsewhere. Casale and Leprestre drew up a separate, and secret, contract for control of Casale's ammonia the patents. Leprestre gathered together a small group of American investors who were enabled to observe the Casale process at work. As soon as Rumianca found out about the Leprestre-Casale agreement, it put a stop to the visits. Casale returned to Idros where Leprestre was now in a better position to back the ammonia studies. At Terni, Casale overcame the several teething problems that he had experienced at Rumianca. However, as soon as Idros planned to manufacture ammonia it was confronted with a stumbling block put up by the cyanamide manufacture Carburo, which denied Idros access to hydroelectricity. This difficulty was overcome after Carburo was brought into a partnership with Idros, to exploit synthetic ammonia, through the newly created SIAS.

SIAS was founded in 1921 at almost the same time as the establishment of Ammonia Casale, by Lepreste and Casale, and whose main objective was licensing the ammonia process abroad. Because the Terni site was inadequate for industrial scale development of the Casale process SIAS took over the Nera Montoro power station and its associated chemical works which had been out of action since the end of the war. Much of the machinery could be readily adapted to the needs of the Casale process. Additional machinery, including special compressors and apparatus for burning air in hydrogen to produce nitrogen was purchased and installed. In 1925, Nera Montoro began producing ammonia. From this time Ammonia Casale and SIAS collaborated closely in the further improvements in the process, especially the enlargement of converters capable of producing 20 tons per day, and more, of synthetic anhydrous ammonia. Further trials and tribulations followed. As for Rumianca, the company claimed compensation from Casale according to the agreement of 17 May 1919.

According to the May 1919 agreement between Casale and Vitale, of Rumianca, Casale would increase production from the 100 kg experimental plant to an industrial unit of 1 ton per day, using byproduct hydrogen made available by Rumianca at the rate of 3,000 cubic metres per day. In the event, Ruminaca was unable to provide all the necessary hydrogen, and thus was unable to demonstrate the scaled-up process, according to its agreement with Casale. This is evidenced by the fact that when Casale and Lepreste signed their secret agreement among the clauses it was specified that the American group would increase the share capital of Rumianca only if the specified higher daily capacity was attained.

Casale claimed that Rumianca had held up development of the Casale process by not fulfilling the conditions of the May 1919 contract. Most especially Rumianca's resources were shown to be insufficient to meet the targeted production. Casale's expert witnesses argued in reports placed before the court in litigation that Casale had lost a two-year headstart against the main rivals, Fauser and Claude, and deserved compensation for loss of business. On appeal, the court found in Casale's favour. Had Rumianca not prevented Casale from demonstrating his apparatus before outside interested parties in 1919, then both sides would have benefited. Significantly the absence of competition outside Italy from Fauser and Claude at the time of the lawsuit was not raised in the court case between Rumianca and Casale.

There were also the American investors. Though they would appear to have disappeared from the scene not long after Casale departed from Rumianca, it is highly likely that Leprestre, having gained their trust, was able to raise funds for development of the Casale process in Terni from the United States. Vincent Handley, one of the observers in 1919, and who had worked at an Italian bank in New York, was appointed the secretary of Ammonia Casale and with Leprestre arranged funding. In addition, Leprestre through his war-related businesses in the United States and Italy probably gained considerable wealth, enabling him to be the principal shareholder of Ammonia Casale in its early years. His reputation was such that in mid-1923, as a resident of New York City, he raised considerable capital from American investors for an enterprise based on manufacture in the United States and international commercialisation of the Casale process. However, the venture did not succeed. More success was achieved in Japan, from 1923, where converters of 7.5 tons daily capacity were installed. This gave a tremendous boost to the Casale process. Several licenses were acquired by French and other chemical companies during 1924. The licensees used Italian-made converters or converters based on the Casale design. By just after the mid-1920s, Terni and Nero Montoro engineers were testing new 20-ton converters under the high-pressure conditions. A novel feature invented in 1923 and introduced in 1927, the year of Casale's death, was the ejector, which did away with a number of moving parts. The simplicity and efficiency of the Casale process was such that the basic model devised by the late 1920s was, with few changes, widely taken up until the 1950s. Ammonia Casale fell behind in the 1970s but was revived in the 1980s through introduction of a novel converter that incorporated both radial and axial flows of gas. Rebranded as Casale SA in 2014, the firm today is a global leader in ammonia and related technologies.

* In recent years, the authors have contributed towards unravelling the hidden history of the Casale process, so far rarely mentioned in the historiography of technology. In particular, Anthony S. Travis has examined the development of the Casale process in the United States, which was adopted by Du Pont in the late 1920s (A. S. Travis, *Substantia*, **2021**, *5*, 55-77), in addition to describing the technological innovations that accompanied the expansion of Ammonia Casale SA, now Casale SA (A. S. Travis, *Catalysis Today*, **2022**, *387*, 4-8).

Lorenzo Francisci has concentrated on the development of the Casale process in Italy, with particular reference to impact on the industrial policies of Terni - Società per l'Industria e l'Elettricità (TERNI). Francisci has highlighted the role of the Nera Montoro plant, which at the time of its construction in

1923 was the world's largest facility for the production of synthetic ammonia (L. Francisci, *Proposte e Ricerche*, **2020**, *84*, 121-132). Francisci has examined the industrial dynamics and the competition between TERNI and Montecatini over control of the Italian nitrogen industry, leading to the primacy of the Fauser process (L. Francisci, *Memoria Storica*, **2023**, *61*, 103-130). Ammonia Casale pioneered, in connection with the Italian autarchy programme, the use of ammonia as an automobile fuel (R. Covino, L. Francisci, *Ricerche Storiche*, **2020**, *1*, 41-57).

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14. With the Haber-Bosch process, coal consumption was one tenth of the arc process and one third of the calcium cyanamide process, while electricity consumption was fifty times lower and twelve times lower, respectively. See C. Rossi, *Giornale di Chimica Industriale e Applicata*, **1920**, *6*, 301-307.

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22. As for the catalyst, it was a compound of inorganic and organic matter (salts, oxides, metals, alkali metals, earth alkaline metals, earth metals, carbon and metalloid oxides). See Accademia Nazionale delle Scienze detta dei XL Historical Archive, *Nicola Parravano Fund*, folder 22, dossier 221, copy of the patent “Metodo industriale ed apparecchio per la produzione sintetica dell’ammoniaca, mediante un nuovo catalitico di grande rendimento” of Luigi Casale, Carlo Andreucci, Mario Santangelo, 18 luglio **1917**.

23. Ibid.

24. Ibid, copy of the patent “Metodo ed apparecchio per produrre da idrogeno miscele di azoto, idrogeno od azoto” of Luigi Casale, 12 July **1920**.

25. A. M. Falchero, *Italia contemporanea*, **1982**, *146-147*, 67-92.

26. See ref. 22, copy of the agreement between Casale and Lepreste, 2 August **1919**.

27. Ibid, copy of the judgement of the Turin Court of Appeal in the Rumianca-Casale case, April **1929**, p. 2.

28. State Archive of Terni, *Siri Fund*, folder 3699, letter from Luigi Casale to Alfonso Vitale, 25 August **1919**.

29. Ibid, Letter from Luigi Casale to Alfonso Vitale, 13 September **1919**.

30. See ref. 22, copy of the patent "Apparecchio di catalisi per la sintesi dell'ammoniaca" of Luigi Casale, René Lepreste, 21 September **1920**.

31. Ibid. See also copy of the patent "Perfezionamento nell'apparecchio catalitico per la sintesi dell'ammoniaca" of Luigi Casale, René Lepreste, 15 February **1921**.

32. B. Waeser, *The Atmospheric Nitrogen Industry: With Special Consideration of the Production of Ammonia and Nitric Acid* (transl. Ernest Fyleman), vol. I, P. Blakiston's Son & Co., Philadelphia, **1926**, p. XVII. Fyleman was a chemist associated with the London-based J.F. Crowley and Partners engineering consultancy of B. Waeser*, Die Luftstickstoff-Industrie, mit besonderer Berücksichtigung der gewinnung von ammoniak und salpetersaure*, Otto Spamer, Leipzig, **1922**, with an introduction by J. F. Crowley. Crowley and Partners represented Ammonia Casale in the UK.

33. Acciai Speciali Terni Historical Archive, *Archives Acquired Fund*, *Carburo*, register 64, minutes of the meeting of the Board of Directors of Carburo, 2 May **1921**.

34. Ibid, minutes of the meeting of the Board of Directors of Carburo, 27 April **1921**.

35. Ibid.

36. Acciai Speciali Terni Historical Archive, *Archives Acquired Fund*, *SIAS*, register 1, minutes of the meeting of the Board of Directors of SIAS, 6 February **1922**.

37. Ibid, minutes of the meeting of the Board of Directors of SIAS, 8 October **1921**.

38. B. Molony, *Technology and Investment: The Prewar Japanese Chemical Industry,* Mass: Council on East Asian Studies/Harvard University Press, Cambridge, **1990**. See also https://www.treccani.it/enciclopedia/l-industria-dei-composti-azotati_(Il-Contributo-italiano-alla-storia-del-Pensiero:-Tecnica)/ (accessed on 10/05/2024).

39. See ref. 36, minutes of the meeting of the Board of Directors of SIAS, 27 June **1922**.

40. See ref. 22, copy of the judgement of the Turin Court of Appeal in the Rumianca-Casale case, April **1929**, p. 2.

41. Ibid, pp. 8-12.

42. Ibid, pp. 14-16.

43. Ibid, pp. 16-18.

44. Ibid, pp. 59-64.

45. Ibid, pp. 104-106.

46. Ibid, pp. 101-103.

47. https:///inflationhistory.com/it-IT/?currency=ITL&amount=2500000&year=1929 (accessed 25 May 2024).