PAGINE DI STORIA

Carlo Perego Senior Advisor Former director of the Institute G. Donegani (2007-2018) caregoperlo54@gmail.com

GIACOMO FAUSER AND THE CATALYTIC HYDROGENATION OF ALBANIAN OIL

In the 1930s, the Novara R&D center of Montecatini, under the direction of Giacomo Fauser, developed a process to increase gasoline and lubricants yields of the Albanian oil. The process was based on a two stage catalytic hydrogenation and was industrially applied in the refineries of Livorno and Bari. The paper summarizes the historical and technical aspects of this story.

Introduction

56

At the conference of the Lombard Section of the Italian Association of Chemistry, held in Milan in 1937, Giacomo Fauser presented the research activities devoted to the development of a hydrogenation process for the production of gasoline and lubricants [1]. Taking into account the lack of oil production in Italy and the difficulty of its imports from abroad - being the oil production and supply under the control of the Anglo-Americans and being Italy subjected to international sanctions after the war in Abyssinia - it became important to have a technology able to produce gasoline and lubricants from poor raw materials, available in Italy and in "friendly" countries.

Since 1932 the AIPA, *Azienda Italiana Petrolio Albania* (Italian Albanian Oil Company), was producing crude oil at the Albanian field of Devoli with a daily capacity of 1,000 t^a. This was an asphaltic crude oil, with a high sulfur content (6%): therefore by distillation and thermal cracking only 40% of gasoline could be obtained. This gasoline being characterized by high sulfur content and low stability (due to the high olefin content).

Starting from the synthetic gasoline production experience of Germany, through the high pressure hydrogenation of lignite, invented by prof. Friedrich Bergius (Nobel Prize in Chemistry, 1931), also in Italy it was hence evaluated the possibility of increasing the yield and the quality of gasoline of this asphaltic crude, through catalytic hydrogenation. As a matter of fact, in Germany the chemical giant IG Farben had realized a first plant at Leuna Werke producing 1,000 t/day of gasoline from lignite in 1927^b.

The hydrogenation studies of Albanian crude were first assigned to the Fuels Section of the Institute of Industrial Chemistry of the *Regio Politecnico di Milano*^c, under the supervision of Mario Giacomo Levi^d. Levi was a true pioneer in this sector, having undertaken studies of hydrogenation of fuels of different origin since 1920s'. After these preliminary studies, the process development was carried out

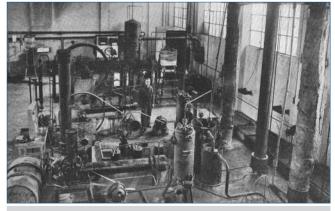


Fig. 1 - Semi-commercial unit for Albanian oil hydrogenation at Novara [2]

^aAIPA had discovered an oil field in Devoli, with an estimated capacity of 12 million t.

^bThe maximum production during the II World War was in 1944, when Germany produced 6.5 million t of synthetic fuels, mainly through the Bergius process, and only in small part through the Fischer-Tropsch process (0.5 million t).

°The current Stazione Sperimentale Combustibili was born from this section.

^dMario Giacomo Levi was removed from the chair of Industrial Chemistry of the Polytechnic because of the racial laws in 1938.

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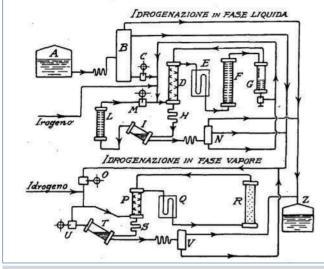


Fig. 2 - Hydrogenation process flow-sheet [1]

by the research laboratory of Montecatini in Novara. As a result of these R&D studies a semi-commercial unit was built (Fig. 1) [2], able to produce up to 80% of gasoline with low sulfur content and high octane number from the Albanian crude. This technology was later applied for the valorization of Albanian crude and also for eventually other poor raw materials, *e.g.* oil shale, oils and asphaltic rocks and tars obtained by low-temperature distillation of Italian lignite. To such a purpose the new company ANIC was founded, belonging fifty-fifty to Montecatini and the Italian State, with the commitment to build-up two industrial plants at Bari and Livorno for the production of 120,000 t/y of gasoline, together with lubricants, paraffin and LPG (liquefied petroleum gas).

The process of catalytic hydrogenation

As shown schematically in Fig. 2, the hydrogenation process developed at the research laboratory of Novara was based on two catalytic steps: one in the liquid-phase and one vapor-phase [1].

The crude oil from tank A was first fractionated in the rectification column B, to separate the gasoline, the middle-distillate and the heavy residue. The residue was then fed, by a high pressure pump C, together with hydrogen in the heat exchanger D, then in the superheater E and finally in the reactor F. The hydrogenated products were sent to a hot separator G. The heavy fractions were then recycled to the hydrogenation reactor, while gasoline and light oils in the vapor state, together with hydrogen in excess,

were sent to the heat exchanger D to heat up the fresh feed. After that this stream was refrigerated in H and finally sent to a cold separator, where the vapor-phase was separated from the liquid-phase. The hydrogen was recovered from the gas containing the light hydrocarbons produced in the reaction, by oil washing in column L and it was recycled by means of the compressor M. The liquid products leaving the cold separator were subjected to a new distillation in column N; gasoline was sent to the tank Z, while the middle-distillate was subjected to a further hydrogenation in the vapor-phase. As shown in the scheme the step of hydrogenation in vapor-phase of the middle-distillate is similar to that of the liquid-phase, but without the hot separator.

The middle-distillate not converted during hydrogenation was separated from the gasoline in the distillation column V and recycled. The hydrogen utilized in the process was produced starting from the light hydrocarbons recovered in the process, after purification from H_2S . The light hydrocarbons were reformed to syn gas (CO/H₂). After reforming, CO was converted to CO₂ by reaction with steam (Water Gas Shift Reaction). Finally, the hydrogen was compressed to 300 bar and purified from residual CO by cupro-ammoniacal washing.

The hydrogenation was performed at a pressure of 300 bar. The temperature of the vapor-phase hydrogenation was in the range 425 and 500 °C [3], as disclosed by Giuseppe Pastonesi^e.

The detailed scheme of the hydrogenation of middle-distillate in the gas phase is shown in Fig. 3 [4]. The overall scheme of Albanian crude oil process is summarized according to Fauser in Fig. 4 [1].

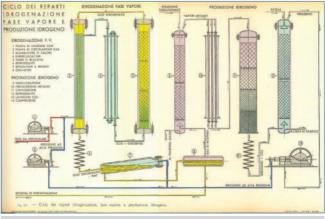


Fig. 3 - Vapor-phase hydrogenation process flow-sheet [4]

57

^eTechnical Director of ANIC-Novara and professor of chemical engineering at the Milan Polytechnic.

PAGINE DI STORIA

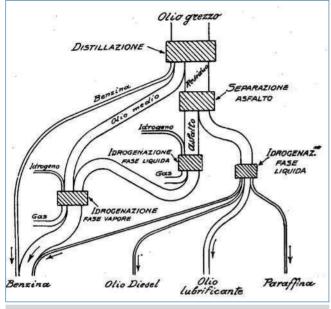


Fig. 4 - Simplified scheme of gasoline and lubricants production from low value crude oil by hydrogenation [1]

Catalysis and catalysts

58

Concerning catalysis and catalysts, Fauser reported: «In the liquid-phase is preferably used a catalytic mass intimately mixed with the liquid, in the vaporphase instead are adopted "fixed catalysts", which have a great resistance to poisoning, and then allow to work continuously for more than a year without a significant decrease of their activity» [1].

In a later paper [5], Sandro Doldi of the ANIC of Novara, wrote: "In the case of oil as the Albanian one, the liquid-phase is mainly aimed to convert the asphalt into oil. Therefore, oxygen and nitrogen are eliminated as water and ammonia (practically ammonium sulfide); the sulfur for the most part as hydrogen sulfide, and then ammonium sulfide. Currently the hydrogenation in the liquid-phase is accomplished with finely divided catalyst in the oil". Beside concerning the hydrogenation catalyst, he reported: "The target is to have a very active catalyst, even at low concentration in the feed, able to hydrogenate the asphalt, as well as to remove organic sulfur, nitrogen and oxygen, while preventing the separation of a large amount of asphalt". And again: "Sulfur balance shows that the hydrogenation of the liquid-phase removed 9/10 of the sulfur content in the feedstock. Beside nitrogen balance shows that the hydrogenation in liquid-phase removed 60% of the nitrogen content in the feedstock".

Unfortunately the papers published by La Chimica e l'Industria did not disclosed which type of catalysts where utilized both in the liquid and in the vapor-phases. However these papers reported to the work of Levi at the Polytechnic of Milan and in particular to the that published in the collection of Fuels Section [6]. In this work, after having distilled of the light fractions up to temperatures ranging between 200-280 °C, the residues (fuel oil) were hydrogenated in liquid-phase with a catalysts powder dispersed in the medium. The hydrogenation products were again distillated obtaining gasoline, a middle-distillate and a residue containing the catalyst. The middle-distillate was than hydrogenated in vapor-phase. In this case the catalyst was supported. The second hydrogenation step was aimed to increase the gasoline fraction.

The catalysts tested included Cu, Co and Fe oxides and natural molybdenite (Mo sulfide). In the first hydrogenation in the liquid-phase, the catalysts were finely pulverized and dispersed in the medium. Among the other the paper reports tests performed with molybdenite that resulted to be a very active hydrogenation catalyst. In this case the test was performed on the residue of crude distillation up to 200 °C, in a Bergius bomb under stirring. The operating conditions were: maximum temperature 430 °C, maximum pressure 238 bar.

For the second step of hydrogenation in vaporphase, at a temperature of 500-510 °C and 200 bar, the catalyst (molybdenite powder) was supported on pumice granules. The overall yield of gasoline (first and second step) was over 70%.

Discussing the results Levi reported interesting comments on the role of the catalysts. Comparing the experiments with and without catalysts, Levi observed that with catalysts no significant improvement of light product yields were achieved. However in the presence of catalysts the coke formation was suppressed and the residue remaining after distillation still containing the catalyst, could be further hydrogenated to produce distillates, by recycling it in the hydrogenation reactor and adding other fresh feedstock. Hence introducing the concept of catalyst recycling with the unconverted residue.

According to the evidences reported by Levi, the best performances were obtained using molybdenite. However at Novara factory, ANIC produced and uti-



lized different catalysts, based on the technology developed in Germany for the hydrogenation of coal. IG Farben started to work on the technical commercialization of the high pressure hydrogenation of heavy hydrocarbons since 1924, after acquiring the patents of Bergius. Matthias Pier ultimately solved the technical problems of the Bergius process by (i) developing durable sulfur-tolerant catalysts, and (ii) dividing the process into two stages, the first one of which (liquid-phase, *Sumpfphase*) produced a so-called "middle-oil", boiling above 325-350 °C, with the second (gas, or vapor-phase) converting this middle-oil into the desired products. The first commercial plant for the liquefaction of lignite coal at the Leuna site was started-up in 1927.

For the first liquid-phase stage, Mo-based catalysts were considered. The catalysts were normally added to the coal/heavy oil (paste) feed in a finely divided state, as a powder or as a colloidal solution, and they were not (could not be) reclaimed. However the economics of using Mo look decidedly unfavorable. The search for a cheaper alternative ended to Fe catalysts **[7, 8]**.

For the second-stage the catalysts were applied in fixed-bed reactors, which entailed a strong emphasis on physical strength and stability. In the beginning, the catalyst best performing was still based on Mo, like that employed in the first stage, but in form of pellets. Then a new catalyst based on W sulfide with an over-stoichiometric amount of sulfur (WS₂₁₅) was introduced. This catalyst labeled Kt. 5058 (Kt. from Kontakt Masse) was a very active catalyst, it allowed a three-fold increase of throughput vis-à-vis the Mo one, at about a 50 °C lower T. But other problems now emerged: (i) the catalyst contained too much W, as this element was also needed in the steel industry, and (ii) its hydrogenation performance was in the end too brilliant, and led to a too strongly saturated gasoline. The trick to solve these two problems consisted in dispersing 10% wWS_{2.15} on Terrana, a montmorillonite earth, treated with 10% hydrofluoric acid. The resulting catalyst, labeled Kt. 6434, was about as active as Kt. 5058 itself, but the gasoline showed a 5 points higher octane number [7]. The catalysts Kt. 5058 and Kt. 6434 were also produced by ANIC in Novara as reported in the next chapter.

Production of catalysts at Novara factory during the war

In 1944, IG Farben, which was supporting the Nazi war effort through the production of synthetic gasoline, was facing the problem of catalyst lack. Given the alliance between fascist Italy and nazi Germany, IG Farben required the ANIC factory of Novara to supply some catalysts.

This story is testified by documents collected in the archives of microfilm Technical Oil Mission, the allies gathered in Germany during the war [9]. In these archives there are several documents related to the Novara factory and to ANIC and concerning the agreements with IG Farben.

Some of these documents, reporting type of catalysts and production capacity of the Novara factory are summarized in the following.

In the letter of ANIC to Matthias Pier of IG Farben, dated March 20, 1944, some details are reported concerning the supply to IG Farben, of two catalysts (5058 and 6434) and an half-processed catalyst (Masse 0) obtained from wolframite, an iron manganese tungsten oxide {(Fe·Mn)WO₄} mineral. According to the letter content it is clear that the production of these catalysts was already practiced in Novara, likely for ANIC captive use.

In a later letter (March 30, 1944), ANIC Director Giuseppe Pastonesi required to the person in charge of the Reich Ministry of Armaments for Italy, to ensure the necessary protection of Novara plant. The attachments of this letter reported a description of the plant, including the capacity (10 t/month) and the information that this production was previously totally dedicated to the refineries of Bari and Livorno. Beside the material balance and the production costs of these catalysts were reported, suggesting that:

- Catalyst 5058: W sulfide WS_{2.15}, delivered to IG Farben as powder (also referred as *Schwarzpulver*, black powder) or granulated. Likely this powder catalyst could be used for hydrogenations in the liquid-phase or, after granulation for fixed bed operation in the vapor-phase;
- 2) Catalyst 6434: W sulfide WS_{2.15}, pelletized with Terrana activated with HF. This was likely used by IG Farben as splitting catalyst (*i.e.* as hydrocracking catalyst) for petroleum fractions and brown coal tar middle-oil, so to produce gasoline. In the ANIC refineries it was probably used

59

PAGINE DI STORIA

ROGES



Fig. 5 - Copy of the order of IG Farben, through Roges, to ANIC, for 10 t month of catalysts, dated July 21, 1944 [9]

for hydrogenation in the vapor-phase of the middle-distillate obtained from the hydrogenation in the liquid-phase of the Albanian oil;

 Masse 0 also referred as *Gelberde* (yellow earth) was H₂WO₄, to be utilized for the production of the W based catalysts.

These catalysts were supplied to the IG Farben factory of Stettin Pölitz, where there were hydrogenation plants for the liquefaction of coal, tar and petroleum residues. In this complex there was also a catalyst factory, no more in operation after the damaged caused by allied air raid^f.

Fig. 5 shows the order of IG Farben, through Roges, a trading company, of July 21, 1944. ANIC is required to supply 10 t/month of catalysts for the production of hydrocarbons.

Fig. 6 shows a table summarizing raw materials, chemicals, half-processed and final products of the ANIC Novara in the summer 1944.

[´]60

Finally it is interesting to point out as, in a letter of September 12, 1944 signed by Giuseppe Pastonesi, ANIC reminded IG Farben the issue on the wolfram return at the end of the war, according to the contract. Facing a war situation now compromised, few months before the defeat, the ANIC managers were concerning with the return of wolfram! Perhaps they did not know in detail about the war situation, or the faith in the victory was unshakable.

The ANIC and plants hydrogenation of Bari and Livorno

6

The fascist Italian government had high expectations on ANIC hydrogenation technology to support the war economy. During the Autarchic Exhibition of the Italian Mineral (Rome, 1938), ANIC published a monograph reporting data and pictures of its activities devoted to the production of fuels and lubricants [2].

It is interesting to note that even this monograph was considered of interest by the Allies, so to be microfilmed and recorded by the Technical Oil Mission [9].

This monograph reported statements according to the fascist propaganda: "Two powerful units were soon built up in Bari and Livorno refineries, very flexible and suitable to operate with different conditions, with a total annual capacity of 240,000 t of gasoline under normal conditions" [2].

Despite of these bombastic ads, the hydrogenation

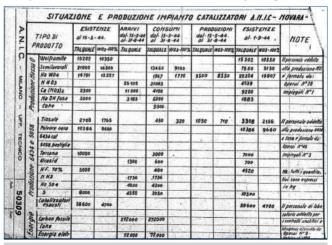


Fig. 6 - Copy of the table summarizing the production situation of the Novara factory in 1944

¹IG Farben had a catalyst factory in Stettin Pölitz, producing different catalysts including 5058, 6434. This plant was very badly damaged by air raids. The first raid was in August 1940 when the CO₂ removal plant was hit and the whole factory put out of action for 2 and half days. There was a second raid in October 1940 in which 30 tanks were destroyed and production was stopped for 2 and half weeks. No more raids occurred until May 1944 after which it rapidly became impossible to operate the hydrogen plant with any appreciable output ("Report On Investigations by Fuels and Lubricants Teams At The IG Farbenindustrie, AG, Works, Ludwigshafen and Oppau", U.S. Dept. of Interior, August 1946).



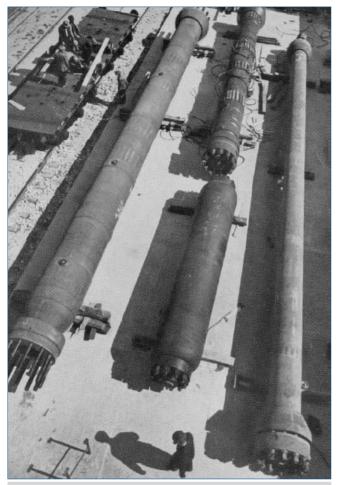


Fig. 7 - Reactors and separator to be installed at Bari refinery [2]

units of Bari and Livorno moved few limited steps. At the end of the war the refinery of Bari was maintained in operation by a joint venture between ANIC and the US Standard Oil, the owner of the Exxon oil company. The new company was named Stanic. The Stanic oil refinery of Bari was then operated until the end of 1970's. The refinery of Livorno was completely destroyed during the war. It was rebuilt by Stanic as conventional oil refinery, mainly devoted to lube oil production in 1950. It is still in operation by Eni [10].

The Bari hydrogenation unit was designed for a production of 120,000 t per year of gasoline, along with about 30,000 t per year of lubricating oils, paraffin and liquefied gases. Besides, 6,000 t per year of sulfur was by-produced.

The hydrogenation plant was also suited to produce synthetic gasoline from Italian brown coal, but it does not seem to have ever used this raw material. An official visit of the fascist hierarchs to the Bari refinery took place during the Fiera del Levante in September 1937. The true operation of the plants, however, began at the end of June 1938, when the tanker "Nautilus" docked at the pier of San Cataldo, with the first cargo of 2700 t of Albanian crude oil. The crude oil was first subjected to distillation; it was obtained a small amount of gasoline, together with a mixture of heavy hydrocarbons. The latter were then thermally cracked producing other light products including methane, which was that reformed with water to produce hydrogen, around 72 million cubic meters per year. This hydrogen was used for the catalytic hydrogenation of medium and heavy oils, at high pressure and temperature, so to obtain a high percentage of gasoline. The sulfur present in the crude oil was converted into hydrogen sulfide, and this was than oxidized to elemental sulfur, which represented a valuable by-product. Fig. 7 shows a hydrogenation reactors and the un-

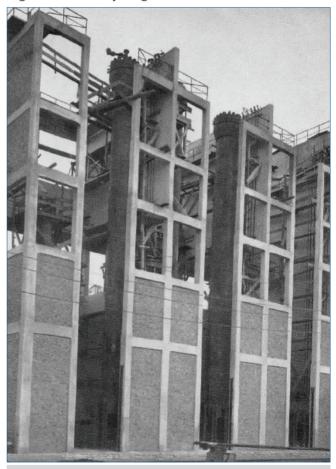


Fig. 8 - Hydrogenation reactors in operation at Bary refinery [2]

61

der pressure separator, to be mounted in the refinery of Bari. Fig. 8 shows the installed reactors.

These reactors were designed according to those used by the IG Farben, having a maximum diameter of 1.2 m and a height of 18 m (see Fig. 9) [11].

Conclusions

The Novara Research Laboratory of Montecatini in the 1930s developed a high-pressure hydrogenation process for the conversion of the asphaltic oil produced in Albania. The technology was based on a two hydrogenation stages: one in liquid-phase with dispersed catalyst (slurry) and one in vaporphase with pelletized catalyst (fixed bed), similarly to the Bergius-Pier process for coal liquefaction, already applied in Germany since 1927.

This new technology was industrially applied at the ANIC refineries of Bari and Livorno. It's interesting to point out how this catalytic hydrogenation technology had reached a high level of complexity for those years.

The catalysts utilized were produced at the ANIC Novara factory in both forms: powder and shaped (*i.e.* granulated or pelletized) $WS_{2.15}$. It seem that the powder was used for slurry reactors while the granules and pellets for the fixed-bed reactors. The W sulfide was shaped either as bulk or with a carrier (Terrana activated by HF).

Very recently increasing interest has been devoted to unsupported hydrotreating (HT) catalysts, having a Ni-Co/Mo oxides or Ni-Co/W oxides bulk composition. In fact, until 2000's the HT technologies have been based on bimetallic oxides (Ni-Mo, Co-Mo, Ni-W, Co-W) loaded on a carrier or copelleted with a ligand (in both cases preferentially Al₂O₂)^g. In 2003 a new HT catalyst has been introduced by ExxonMobil, Akzo Nobel and Nippon Ketjen. This new catalyst named Nebula has higher performances with respect to the conventional HT catalysts, mainly because a new carrier concept, which is not based on Al₂O₃ [12].

62

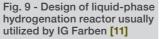
According to J.A.R. van Veen [8], the Nebula technology, is in certain respects a return to the Pier catalysts, in that the compositions are no longer something like 20%w metal-oxides and 80%w carrier, but rather more like 80%w metal-oxides and 20%w support (or structural promoter), *i.e.* almost that of a bulk catalyst.

As a matter of fact, after the introduction of Nebula catalyst series, numerous papers and patents have been reporting unsupported transition metal sulfides catalysts. Accordingly several unsupported materials have been identified with higher activity and/or selectivity than the traditional γ -Al₂O₃ supported Ni/Co-Mo/W catalysts **[8, 13]**. In practice, after more than 80 years, the knowhow gathered by Matthias Pier, Mario Giacomo Levi and Giacomo Fauser on the catalysts for high pressure hydrogenation of coal and heavy oils is still relevant for the modern refinery and industrial chemistry.

Epilogue (from hydrogenation of oil to that of vegetable oils to produce margarine)

The difficult post-war situation for the Italian company ANIC resulted in the agreement with the American Standard Oil, with the creation of Stanic, who took over the management of the two refineries of Livorno and Bari. In practice to ANIC only the small production units of Novara were left. In 1953 when the Eni (Ente Nazionale Idrocarburi) was founded, ANIC was incorporated in the new company. Than Eni started a program for the reorganization of ANIC, aimed to develop the petrochemical industry in Italy. In 1955 ANIC announced a project for the construction of a petrochemical complex in Ravenna and following the discovery of crude oil in 1957 in Gela, was started the construction of a petrochemical complex in Sicily.

ANIC continued to be active in Novara until the early Sixties, when the factory was sold to the Swiss company Helvetia. The hydrogenation know-



⁹Conventional HT catalysts are loaded into the reactor in the form of oxides of the active agents, but they act in the sulfided state when the operation is in progress. Therefore usually these catalysts are subjected to a pre-sulfurization procedure.

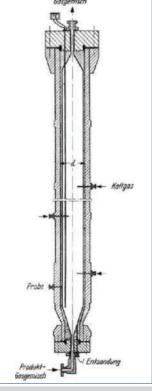






Fig. 10 - ANIC research laboratories (left) in Novara, in a picture of $1934\,$

how was so devoted to the production of margarine, named "Flavina", by catalytic hydrogenation of vegetable oils. Helvetia continued the production of margarine until the end of the 1970s'.

The picture in Fig. 10 shows on the left the ANIC Research Centre of Novara in 1934.

In 1941 when the construction of the new Institute of Chemistry, later named Istituto Guido Donegani^h, was completed (see Fig. 11), Giacomo Fauser moved with its collaborators in the new laboratories. Here he continued to work until 1967, the year of his retirement. After ammonia and catalytic hydrogenations, Fauser devoted his research on the production of synthesis gas from natural gas and hydrocarbon fractions, of nitric acid and urea, of methanol, of acetylene and ethylene. In 1971 there were around the world 370 industrial plants licensed with the name Fauser-Montecatini.

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Fig. 11 - Istituto Guido Donegani in a postcard of 1950's

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Giacome Fauser e l'idrogenzione catalitica del petrolio albanese

Negli anni Trenta, la Montecatini di Novara, sotto la direzione di Giacomo Fauser, sviluppò un processo per incrementare la resa in benzina e lubrificanti, a partire dal petrolio albanese. Il processo era basato su un'idrogenazione catalitica in due stadi e venne applicato industrialmente nelle raffinerie di Livorno e di Bari. L'articolo riassume gli aspetti storici e tecnici di questa vicenda.

63

^hGuido Donegani was the chairman of Montecatini SpA until 1945.