

La química en la historia, para la enseñanza.

Anselme Payen

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Resumen

Anselme Payen (1795-1871) fue un científico y un hombre de la industria, un multifacético personaje que hizo importantes aportaciones en ambas áreas. Él desarrolló el proceso francés de fabricación de bórax a partir del ácido bórico y carbonato de sodio, la utilización del carbón animal como decolorante del azúcar de remolacha, un proceso mejorado de cámaras de plomo para producir ácido sulfúrico, el uso total de residuos animales, etc. Dedicó gran parte de su vida al estudio de la fisiología vegetal y dentro de sus muchos alcances en esta área podemos destacar el descubrimiento de la diastasa, la enzima de descomposición del almidón, la celulosa, la lignina, y el papel vital del nitrógeno en el desarrollo de los vegetales.

Abstract

Anselme Payen (1795-1871) was multifaceted scientist and industrialist who contributed significantly to both areas. He developed the French process for producing borax from boric acid and sodium carbonate, the utilization of carbon animal as a decolorizing matter for beet sugar, an improved lead-chamber process for manufacturing sulfuric acid, the total utilization of animal waste, etc. He devoted a large part of his life to the study of plant physiology and his many achievements in this area include the discovery of diastase, the enzyme for decomposing starch, cellulose, lignin, and the crucial role of nitrogen in vegetable development.

Life and career

Anselme Payen was born on January 6, 1795, one the six children of Marie-Françoise Jeanson de Courtenay and Jean-Baptiste-Pierre Payen. His brother died at an early age and one of his four sisters passed away at the age of fourteen.

Anselme's father studied at the Collège de Navarre in Paris, where he had as classmates such illustrious figures as Gaspar Monge (1746-1818),

Claude-Louis Berthollet (1748-1822), and Jean Antoine Chaptal (1756-1832). In spite of his showing great aptitudes for science and theological discussions, Jean-Baptiste's father decided that he should become a lawyer and for this purpose purchased for him the position of *sustitut du procureur du Roi* in Pairs. In 1792 Jean-Baptiste lost this position as a result of the revolutionary constitution of 1791, and on July 6 of that year used the compensation he had received to buy an ancient hunting house and some land in Grenelle, next to the banks of the Seine, then at the outskirts of Paris. There he established, one after the other, a bleach works, a calico printing factory, and a plant for the production of gelatin by digesting animal waste, especially bones; an industrial complex which would later be expanded to include manufacturing of sulfuric acid and hydrogen chloride, borax, sugar refining, etc. In the 1790s he was producing substantial amounts of sal ammoniac (ammonium chloride), which was then imported from Germany, from animal waste, soda, and hydrogen chloride, using the processes developed by Antoine Baumé (1728-1804) and Nicolas Le Blanc (1742-1806). He also started the production of sulfuric acid using the lead chamber process as well as the refining of crude sulfur imported from Sicily. After 1800 he began to refine tincal (crude borax) imported from the Far East, joined the Napoleonic beet sugar scheme (1811), and opened a boneblack factory (1818). All these factories were linked by one using the output of the other (Mertens, 2003).

It was in this atmosphere of science that Anselme was brought up. He was not enrolled in a school, his father was his only teacher until he could be sent away for more advanced education. This type of education exerted a tremendous influence on his character and made him a lone wolf, well informed on scientific matters, but rather unsociable and not looking for company, traits that persisted throughout his life. Afterwards his father sent him to Paris to study chemistry under Louis Nicolas Vauquelin (1763-1829) and Louis-Jacques Thénard (1777-1857), physics with Antoine-François Fourcroy (1750-1809), and mathematics with François Trémery. Vauquelin's course of applied chemistry took three years and was attended by well-educated persons, workshop overseers, and manufacturers. It was this course that Anselme attended between 1812

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and 1814. At Vauquelin's laboratory Anselme became acquainted with Pierre-Jean Robiquet (1780-1840) and Alphonse Chevalier (1793-1879). Robiquet and Payen were to be the main contributors of articles on chemistry and chemical technology to the *Dictionnaire Technologique* (Lejeune, 1822), launched in 1822. Chevalier and Payen were to publish jointly three successful treatises on reagents, hop, and the potato in the early 1820s (Mertens, 2003). After finishing his training with Vauquelin Anselme enrolled in Thenard's course of analytical and theoretical chemistry for advanced students "*L'Analyse et la Philosophie Chimique*" (Thenard, 1813) given at the *Collège de France*. Payen followed this course and probably became well acquainted with Thenard's *Traité de Chimie Élémentaire Théorique et Pratique* (Thenard, 1816), published between 1813 and 1816.

His father wanted Anselme to enter the *École Polytechnique* to avoid being recruited into the revolutionary army. The turmoil of the Hundred Days and the fall of Napoleon I released him of this problem. At the age of 21 his father put him in charge of the borax refinery and after his father passed away on February 1820, Anselme Payen, then 25 years old, had to assume full responsibility for the management of the many factories established by his father: sugar beet, sulfuric acid, hydrogen chloride, sulfur, gelatin, animal black, sal ammoniac, and a special laboratory for manufacturing the chemicals required by the pharmaceutical industry.

In 1821 Payen married Zélie-Charlotte-Mélanie Thomas, the daughter of a well-known businessman. They had five children, of which only one survived.

His first job as a teacher was at the *Société Philotechnique* and his first publication was a short treatise on chemistry, in collaboration with Chevalier (Payen and Chevalier, 1825).

In 1829 Payen began to teach chemistry at the *École Centrale des Arts et Manufactures*, acting as a substitute for Jean-Baptiste André Dumas (1800-1884) and in 1836 he was appointed professor at the same school to give his own course of industrial chemistry. This course was published in 1849 under the title *Précis de Chimie Industrielle* (Payen, 1849), followed by five new editions between 1851 and 1877. In 1839 he accepted a similar professorship of chemistry applied to the arts at the *Conservatoire Royal des Arts et Métiers*, occupying one of the five new chairs instituted by Louis Philippe (1773-1850). Payen occupied these two teaching posts until his death in 1871. At the age of 40 Payen decided to resign all his

administrative industrial activities and dedicate himself completely to scientific research. Many of his researches were related to industrial applications but they were always addressed towards vegetable physiology. He sold his factories to Théodore Jouet and by 1839 he could say that he had relinquished "all industrial management and commercial responsibility" (Mertens, 2003). As told by Girard (1871): "M. Payen avait eu cette fortune singulière d'être un industriel savant avant que d'être un savant industriel; il était né dans une manufacture de produits chimiques, et son enfance s'était écoulée au milieu de ces opérations industrielles don't l'étude devait être la préoccupation de toute sa vie" (Mr. Payen had the singular fortune of having been a learned manufacturer, before he became an applied scientist; he was born into an atmosphere of chemical products, and spent his youth in the midst of those industrial operations he was to study continuously all his life.)

His management of the sugar beet factory led Payen to become interested in agricultural chemistry and in the application of science in general to the improvement of agricultural practices. In a book published in 1828, together with Chevalier, entitled *Traité de la Pomme de Terre* (Payen and Chevalier, 1826) he described in considerable detail not only the preparation of various foods and feeding stuffs for human and animal consumption, including the production of sugar and syrup, but also the methods for the preparation of starch and alcohol from potatoes.

Payen was elected a member of the *Société Royale et Centrale d'Agriculture* (established in 1761 and now called the *Académie d'Agriculture de France*) on July 17 1833 and named its permanent secretary on March 4, 1845, a position he held for 26 years. He carried through a reorganization resulting in two divisions, a division of agricultural science (farming, forestry, stock breeding) and a division of applied science (physics and chemistry, natural history, mechanics and economics, all applied to agriculture). He acted as the editor of the monthly *Bulletin* between 1845 and 1871, and regularly contributed to the society's annual *Mémoires* (Mertens, 2003).

The *Académie de Médecine* elected him as associate member in 1868, in 1828 he was appointed Chevalier of the *Legion de Honneur* by Charles X, in 1847 Officier by Louis-Philippe, and in 1863, Commandeur by Napoleon III. He was a member of the *Société d'Encouragement Pour l'Industrie Nationale*, member of the *Comité de Salut Public et Hy-*

giene, President of the Société d'Horticulture de la Seine, and member and jury of six national industrial exhibition held between 1827 and 1850, as well as of four World Exhibitions held in London and Paris between 1851 and 1867. He was three times candidate for membership of the *Académie des Sciences*, section Rural Economy; he failed in 1838 [replacement of Henri-Alexander Tessier (1741-1837)], and in 1840 [replacement of Pierre Jean-François Turpin (1775-1840)], and succeeded in 1842, substituting Jean-Victor Audouin (1797-1841).

The Cellulose, Paper and Textile Division of the American Chemical Society gives every year the Anselme Payen Award to the best work in the field. This international prize, which includes a bronze medal and an honorarium of \$3,000, is given to honor and encourage outstanding professional contributions to the science and chemical technology of cellulose and its allied products. A small street in the 15th Arrondissement of Paris is named after him. At the Seventh Industrial Exhibition held at the Louvre in 1827 Payen was awarded a silver medal for the bituminous products he had exhibited.

Payen served for more than forty years as commander of a battalion of the National Guard of the town of Grenelle. His authority was enough to maintain order during the troubled years that followed the revolutions of 1830 and 1848.

Payen spent all his life in the same poor quarter of Paris, much respected by his working-class neighbours. During the famine that resulted from the siege of Paris he tried by all possible means to make various unusual materials edible. Payen did not miss one session of the Académie des Science and the Académie de Médecine. Suddenly, on May 9, 1871, when he had just entered the session of the Académie de Médecine, he lost his consciousness, having suffered an attack of apoplexy. For three days he was sunken in a deep torpor, making rare signs with his hands to his wife and daughter. He passed away on May 12, 1871, and was buried in the cemetery of Vaugirard. Few of his friends were able to attend his funeral because those were the days of the establishment of the Paris Commune and civil war raged on the streets of Paris. Jean Baptiste Huzard Sr. pronounced the funeral oration written by Michel Eugène Chevreul (1786-1889), a friend and fellow member of the *Société d'Encouragement Pour l'Industrie Nationale*. In his words, Chevreul put special emphasis on the fact that Payen had always combined the cultivation of science with the industrial know-how

he accumulated as a manufacturer of chemical products. Payen's wife survived him for two months.

Aimé Girard (1830-1898) succeeded Payen as professor of industrial chemistry at the *Conservatoire des Arts et Métiers*.

Scientific and industrial contributions

Payen was a multifaceted industrialist and scientist. The ample gamma of his activities is reflected in the near 200 papers he published in different journals (Anonymous, 1871). Here we will discuss only some of them.

Sal ammoniac (ammonium chloride)

Prior to 1760, sal ammoniac was imported from Egypt, where it was produced from the soot resulting from the burning of the excrements of various animals, in particular camels. On June 6/1767 the *Conseil du Roi* granted Antoine Baumé (1728-1804) a privilege to establish a manufacture of sal ammoniac at Gravelle, near Paris. Baumé's procedure consisted in decomposing by distillation, ammonium carbonate produced by the calcination of nitrogenous organic materials (woolen rags, horse hair, leather scraps, urine, and solid excrements), with chlorides of magnesium and calcium obtained from the salt marshes of Lorraine, in the presence of chalk that facilitated the reaction. The resulting mud was filtered, the filtrate left to crystallize and the solid ammonium chloride purified by sublimation. Later on Baumé started producing hydrogen chloride by the reaction of marine salt with sulfuric acid. Reaction of chalk with ammonium carbonate yielded ammonia, which was reacted with the hydrogen chloride to produce very pure ammonium chloride. Baumé's method would be afterwards utilized by Leblanc and Dizé in their sodium carbonate factory at Saint-Denis. Unfortunately, Baumé's partners in Gravelle refused to commercialize the new method because they judged it economically too hazardous. As result, Baumé departed from his associates and in 1787 discontinued the operation of his factory. According to Anselme Payen, the shareholders lost more than four hundred thousand francs. Many other manufacturers copied Baumé's procedure and as a consequence he was involved for many years in a bitter legal battle to have his rights recognized.

In 1802 Jean-Baptiste Payen was granted a concession to cut trees in the forest around Paris to use the wood for preparing ammonium chloride in his Grenelle usine. Ten years later, he and two other

manufacturers, Bernard-Nicolas Pluvinet and Nicolas Bourlier, reopened Baumé's business. Their activities were announced in the newspaper *Avant Coureur* as follows: "Les sieurs Payen et Pluvinet frères, propriétaires des deux fabriques de sels ammoniac blanc et gris, situées plaine de Grenelle et à Clichy la Garenne, préviennent le public qu'ils ont conjointement établi le dépôt general des produits de ces fabriques, rue de Croissant, No. 20, où ils feront la vente en gros" (Messrs. Payen and the Pluvinet brothers, owners of two plants for manufacturing white and gray ammonium chloride, located in the plain of Grenelle and at Clichy la Garenne, notify the public that they have established a general depot on street Croissant No. 20, for the wholesale of their products).

The partners introduced the use of new cylindrical cast iron retorts and improved the process to such an extent that commercial sal ammoniac had a better quality and a lower price. One probable reason for their success was the fact that by using the Leblanc process the sodium sulfate issuing from the sal ammoniac factory could be converted into a valuable by-product instead of throwing it away as a residue without value. In addition, the range of possible raw materials was greatly expanded beyond just bones: blood, flesh, horns, woolen rags, silk rags, horsehair, and leather scrapings (Mertens, 2003).

Soda developed rapidly from a subsidiary interest into one of Payen's main chemical products, and therefore he began to manufacture the sodium sulfate required for soda production by heating common salt and sulfuric acid. This was the process employed by Le Blanc and Jérôme Dizet (1764-1852) in their plant in Franciade (Saint-Denis), and declared the best available by the special commission appointed by the *Comité de Salut Public*, on January 27, 1794 (Lelièvre, 1794; Mertens, 2003).

Carbon black

In his memoirs on the subject Payen wrote (1822a,b) that not many people were aware of the important role played by *noir animal* (animal charcoal) in the fabrication and refinement of sugar. It was known that in contact with this substance, the sugar liquors were purified and decolorized. Without their help it would be impossible to obtain white crystalline sugar that satisfied our view and our taste. But few were aware of the fact that at the beginning of the European sugar industry *noir animal* was not in use at all. Chemistry knew the bleaching action of wood charcoal, but not

that of the carbon obtained by bone calcination. In 1809, for the first time, a smart refiner named Guillon, thought applying to the depuration of white syrups the decolorant action of wood charcoal. In this way he prepared white syrups of an agreeable taste that were immediately considered for consumption. Two years later, in 1811, Pierre Figuier of Montepellier, called for the first time the attention of scientists about the properties of animal carbon, properties which were clearly superior to those of vegetable carbon. At that time, Payen manufactured in Grenelle sal ammoniac by calcining different animal materials, particularly bones, in a closed vessel. In the calcination retorts there remained a residue of a calcarian substance mixed with divided carbon that retained the shape of the calcined bone. This residue, which was called *noir animal*, was the one that Payen thought to apply to the decoloration of sugars. The commercial success was not instant, the refiners were not eager to replace wood charcoal by the new product. Payen performed additional work with the collaboration of Derosne to try to overcome the resistance, and eventually his efforts were crowned with success: two factories were established in Paris to manufacture the new decolorizing agent and when their capacity became insufficient a third factory was established in Orleans, to be followed by others wherever sugar beet was being grown and processed (Girard, 1871).

In 1821 the *Société de Pharmacie* put up a reward for an answer to the questions as to how the decolorizing action of carbon should be explained and whether it was the physical state of carbon of animal origin that caused its greater decolorizing power. Three memoirs were rewarded: those written by Alexandre Bussy (1794-1882), Pierre-Antoine Elie Desfossés (1792-1865) and Anselme Payen (Mertens, 2003).

Payne's study went beyond the terms of the competition, he looked into the details of both boneblack production and the regeneration of used boneblack from sugar refineries. The editors of the *Annales de l'Industrie* got the right to publish the entire memoir, which treated of a widely used substance and was written by someone who manufactured great quantities of it on a regular basis at his establishment in Grenelle and who had done a lot of experiments (Mertens, 2003).

The first step of Payen's study consisted of the experimental proof of the substances that accompanied *noir animal*, such as calcium phosphate, were not

responsible for its decolorizing action. Payen found that there were actually two kinds of carbon, "black lead or graphite", which is lustrous (*charbon brilliant*), and a lustreless or amorphous variant (*charbon terne*). He proved that the latter species was the active one. He attributed this activity to "a kind of chemical division, a particular state of carbon determining its freedom to act energetically on coloring matter (Payen, 1822a). From numerous experiments with normal boneblack, normal charcoal, boneblack treated with hydrogen chloride, carbon from Prussian blue manufacturers, carbon from pyroligneous acid manufacturers, carbon from sugar mixed with calcium phosphate, etc., he concluded that the physical state (shape and state of aggregation) of these carbons was the one that determined their decolorizing power (Mertens, 2003).

Payen did not like the term physical state that the Société de Pharmacie had used to formulate one of its hypotheses. He preferred "chemical division": "It is possible to consider the physical state of animal charcoal as the essential cause of its action. But in my opinion it would be more to the point to look upon this peculiar state as a chemical division, because this division cannot be brought about by mechanical means". The state of the carbon and therefore its activity, its decolorizing power, depended on the way it was prepared in laboratory experiments or manufactured in chemical factories. Conversely, the activity of *noir animal* could be enhanced by a rational change of the mode of its production. All the improvements I successfully introduced into the preparation of boneblack in our factories as well as the additional improvements I intend to make are founded in this theory of the chemical division of active carbon (Payen, 1822a). Payen also developed an apparatus, which he called *decolorimeter*, for determining the decolorizing ability of various lots of animal charcoal.

Borax

Since old times borax has been an important raw material used particularly in the metal and ceramic industries, the first for soldering and brazing, the second as a flux to give certain glazes an appropriate fusibility. Up to the eighteenth century Europe imported crude borax (tincal) from India, China, Ceylon, Persia, Perú, and Tartary in the form of small crystals which would efflorescence in contact with air, by losing part of their crystallization water. In order to avoid this alteration, which reflected in a loss

of weight, the natives coated the crystals with a layer of fatty material that protected them completely. Most of tincal came from Tibet, from a series of Himalayan lakes (Teschu-Lumbu) located at an altitude of 13,000-15000 feet. The crude borax crystals from Tibetan lakes was transported in bags tied to sheep, which were driven by caravan over the Himalayas to the north of India. From there it reached Calcutta where Arabian, and afterwards European, traders picked it up.

The protective coating meant that borax had to be purified before its use. The pertinent process had been developed by the Venetians and kept secret for many years. Eventually it was transferred to Holland, where it became a monopoly. The Dutch secrets were revealed by Jacques-Christophe Valmont-de-Bomare (1731-1807) who visited the Dutch workshops and published the tincal secret in his *Dictionnaire Raisonné Universel d'Histoire Naturelle* (Valmont de-Bomare, 1775). According to Payen, it was the Lécuyer brothers who brought tincal refining to France in 1786.

The secrecy regarding the purification of borax is described very picturesquely by Pierre-Joseph Macquer (1718-1784) in his *Dictionnaire de Chymie* (Macquer, 1766): "As borax is not found in Europe, it is brought from the East Indies in a state which only requires a slight purification, which is given to it by the Dutch and Venetians who are the chief merchants of it. But is is not yet known whether this matter be a natural or an artificial substance, not whence, nor how it is obtained. Our ignorance concerning borax is certainly owing to the interest, which they who make a lucrative commerce of it, have to keep everything concerning its origin secret."

Borax started to be refined in France about 1805, but at the time of Payen the French refineries could only produce small and ill defined crystals that the commerce refused to accept, in spite of the fact that the price of refined borax in 1815 was about 7-8 francs per kilo. By the end of the eighteenth century boric acid was discovered in the *soffioni* in the region of Larderello in Tuscany. Here, geysers of water, called *soffioni*, *fumacchi*, *fumarole*, or *mofetti*. are released from the ground, at about 205°C and pressures up to 14 bar. The water contains about 0.4% of boric acid, which then crystallizes yielding up to 80% boric acid in the form of decahydrate, $H_3BO_3 \cdot 10H_2O$. Boric acid had been discovered in 1702 by Wilhelm Homberg (1652-1715), who had named it *narcotic salt of vitriol* or *sedative salt*. Tuscan boric

acid began to enter the European markets at the beginning of the nineteenth century (Payen, 1841b).

Payen's father joined the borax refining business in the beginning of the 1800s. When Anselme Payen took over the family business he considered, together with Chartier, the possibility of breaking the Dutch monopoly by preparing borax by the reaction between French sodium carbonate and Tuscan boric acid. The possibility of this reaction had been demonstrated on a laboratory scale by Hyacinthe Théodore Baron (1707-1787) in 1750 (Baron, 1750). In 1827 François-Jacques Lardereel (1789-1858) developed a new extraction method using the *soffioni* as a source of heat that enabled him to increase production ten-fold, and thus opened the way for Payen. Payen succeeded in producing two forms of sodium borate both in his research laboratory and, after scaling up the process, in his borax factory (Payen, 1828). In 1828 he discovered that the reason why artificial borax was of inferior quality than refined *tinkal* was its being a mixture of octahedral borax (pentahydrate) and prismatic borax (decahydrate), in variable proportions. For this reason the crystals resulting from the reaction of boric acid with soda were small and without solidity. On the other hand, pure prismatic borax crystals were voluminous and had the qualities preferred by commerce. After a decade of experimentation Payen discovered that by careful control of the operating variables, in particular the temperature and density of the solutions, it was possible to guide the process to produce either type of crystals. As explained below, he found that a sodium borate solution of a density of 30° Bé yielded pure octahedral borax between 79° and 56°, and that below 56°C prismatic borax began to be deposited on the octahedral crystals (Payen, 1841c).

This discovery is quite a feat, considering that at that time there was no knowledge of phase diagrams. It constitutes another proof of Payen's superior analytical qualities.

It did not take long until Payen's factory in Grenelle started offering to the commerce crystals more beautiful than those coming from the best Dutch refineries. In order to introduce in the market Payen had to fight the conservative attitude of the local users. His crystals were too good to be true, so it seemed "Le nouveau produit était tellement beau, que le commerce commença par le refuser. Pour obtenir qu'on acceptât le borax préparé artificiellement, M. Payen dut lui faire donner la teinte grisâtre qu'avait le borax hollandais; il dut aussi user les

angles de ses cristaux par un frottement sur les cribles produisant des effets semblables à ceux du transport; enfin il fut forcé d'imiter les emballages étrangers, c'est-à-dire d'employer des caisses en bois de Tremble ou Peuplier de Hollande, de donner à ces caisses une contenance de 60 kilogr. et de les doubler intérieurement de feuilles de papier bleu indigo" (The new product was so beautiful that initially commerce refused it. In order to have them accept the borax prepared artificially, Mr Payen had to impart to it the greyish hue of Dutch borax; he had also to wear down the angles of the crystals by rubbing them against a sieve, thereby producing effects similar to those resulting from transport; he had even to imitate foreign packing, that is to say, boxes made of aspen or Dutch poplar, with a capacity of 60 kg, and lined with sheets of indigo paper) (Barral, 1873).

The final result was that France won a new industry and the Dutch lost their monopoly. The price of borax rapidly dropped to less than one third and by 1871 it was already below 2.50 francs per kilo (Girard, 1871).

Payen described his fabrication process as follows (Payen, 1841a): "We have used as raw material boric acid from Tuscany, containing between 74 to 83% acid. The impurities present are water, ammonia sulfate, magnesium, calcium carbonate, alumina, iron chloride, ammonia chloride, and traces of hydrogen sulfide, clays, sand, sulfur, a yellow coloring matter, and a nitrogenous substance, soluble in alcohol. The sulfates and chlorides present lead to a substantial expense in soda, being transformed into sodium sulfate and carbonates of magnesium and calcium. The traces of alumina and iron oxide form with other insoluble substances a voluminous deposit that cannot be economically removed from the solution.

The preparation of crude borax involves reacting 1000 kg of boric acid with 1200 kg of crystalline sodium carbonate, in about 2000 kg of water. First the sodium carbonate is dissolved in a wooden tank lined with lead and heated with steam by direct injection. Once the carbonate has dissolved the temperature is raised slightly above 100° C and the boric acid is added slowly, in batches of 4-5 kg. The gases released (CO₂ and small amounts of ammonium carbonate) are absorbed in sulfuric acid to recover the ammonia released. Once all the boric acid has been added the temperature is brought to boiling, at about 105°C, whence the solution has a specific gravity of about 21° Bé (Baumé degrees are related to

the specific gravity G by the relation $^{\circ}\text{Bé} = 140/G - 130$). Heating is now stopped and the solution left to rest for about 10-12 hours, while a solid deposit is formed at the bottom of the vessel. The clear liquid is drained and left to crystallize in wood basins, where prismatic crystals are formed in a very slow and regular process. After 25 to 30 days, the temperature has dropped to around 27°C and crystallization is complete; the clear supernatant mother liquor is now led to the crystallizers and the solid removed. The mother liquor and the water used for washing the vessels are recycled. The crystals are removed to another vessel to be washed. The quality is still below that required by commerce. Further purification is achieved by re-dissolution and slow crystallization.

The principal problem of the refining operation is to obtain voluminous crystals; their volume depends on the mass being treated, the slowness of the cooling, and particularly, on the regularity of the cooling rate. The crude borax to be refined is introduced in a wooden vessel lined with lead and heated by steam. Water is added to dissolve the impure crystals, which are held in a basket is maintained slightly below the surface of the liquid. For every 100 parts of borax 5 parts of crystalline sodium carbonate are added. The solids are added in the same ratio until the solution has a specific gravity of 22°Bé at 100°C . The solution is left to rest to clarify it and then the hot clear liquid is siphoned to a large crystallizer, made of wood and lined with lead, and built in such a way to avoid vibrations. Depending on the external temperature the process lasts between 16 to 18 days. After this period of time the temperature has dropped to about $27-28^{\circ}\text{C}$. Care is taken not to let it go below this range, otherwise other substances may also crystallize. The mother liquor is separated from the crystals as fast as possible, and siphoned to crystallizers with a flat bottom, where small crystals appear. The large crystals of borax are dried and packaged.

Preparation of octahedral borax is similar, except that the solution is more concentrated, 30°Bé at 100°C . Crystallization of octahedral borax begins at 79°C and ends at 56°C . Again, the mother liquor is removed by siphoning.

Animal waste

In 1825 the *Société Centrale d'Agriculture* proposed a first award of 1000 francs and a second of 500 francs, for the best memoirs "destinés à faire connaître aux agriculteurs quel parti ils pourraient tirer des ani-

maux qui meurent dans les campagnes, soit de maladie, soit de vieillesse ou par accident" (to make known to farmers the benefit they could obtain from animals that die in their fields because of illness, old age, or accidentally). The program indicated that at that time the corpses of dead animals, after being skinned, were simply buried in the ground or left in the sun, to become the feed of prey birds, wandering dogs, or wolves. The Society was intent in ending these practices. But it was difficult to fight against ignorance and inveterate customs of negligence and laziness; there was a general belief that touching a dead animal would induce dangerous illnesses.

The contest remained opened for five years, Eventually, on April 18 1830, Payen's proposal was awarded the prize. His memoir of 136 pages, *Notice sur les Moyens d'Utiliser Toutes les Parties des Animaux Morts Dans les Campagnes* (Payen, 1830) addressed itself to every possible situation related to the complete use of dead animals, be it for industry or for making fertilizers intended for agriculture. In this manual suggestions were given regarding the fertilizing value of substances rich in nitrogenous compounds and of a procedure for evaluating the relative advantages of fertilizers. The latter was based on the decomposition of the sample to be tested at the red temperature, in a retort made of cast iron or clay. The gaseous products released were absorbed in diluted sulfuric acid, followed by determination of the amount of acid neutralized by an alkaline solution. While this method did not give him all the nitrogen in the form of ammonia, it may be considered a forerunner of the well-known Kjeldahl method.

The first part of Payen's manual was devoted to try to convince farmers that they had nothing to fear when they handled the corpses of cows or horses. To do so Payen quoted the ideas of the well-known veterinarian Jean-Baptiste Huzard (1755-1838), that there were a number of trades and industries where animal carcasses or parts of them were handled, and were not considered injurious. Among the examples mentioned were gut works, slaughterhouses, *poudrett* manufacture (feces turned into granular fertilizer), glue, gelatin, and sal ammoniac (manufactured from calcined animal parts).

Condensed versions of Payen's manual were published in French, German, and American journals.

The following chapters contained suggestions to the farmers on how to make full use of the dead animals by separating them into hides, hairs, hooves, fat, bones, meat, tendons, blood, and guts, and the

sale of these materials to tanners, rope makers, saddlers, gut workers, and to the manufacturers of combs, grease, candles, soap, boneblack, sal ammoniac, glue and granular fertilizers. Hooves could be processed into horn material ready to be used by manufacturers of buttons, combs and other domestic articles, goose feathers could be turned into quill pens, blood could be dried and transformed into a clarifying substance widely used by sugar refiners, etc. Materials such as bones and blood could be transformed into fertilizers on the spot and employed directly in the farmer's fields. According to Payen, cutting up a horse could earn the farmer an additional 60 francs or so. Oxen and cows were even more profitable (Mertens, 2003).

Payen's manual was very successful. Many industries, be of fertilizers made from animal residues, be of multiple products made from the horns, the bowels, blood, etc., were eventually built following the recommendations given in the book. A large amount of valuable products were then produced from materials previously considered a nuisance. Fifteen years later no animal carcasses were left to rot in the countryside any more (Mertens, 2003).

Natural rubber and gutta-percha

Payen investigated the characteristics of the vulcanization of natural rubber (caoutchouc) and the properties of the resulting product (Payen, 1852a; Le Bras, 1943). Vulcanization was performed by immersing a sheet of rubber, two or three millimeters thick, in a bath of melted sulfur at 120°C. Initially the liquid penetrated the pores faster than water or alcohol would do, and swelled the rubber. The pores became distended and the weight of the sample increased by 10 to 15%. Interesting enough, the properties of the organic material remained essentially the same, it could be shaped and soldered like the original material and solvents attacked it with the same energy. Increasing the temperature to 130-160°C, for about 30 to 40 minutes, caused the reaction to go faster and this time the aspect and the properties of rubber were modified substantially. The material acquired a yellow hue and could not be self-soldered, the elasticity decreased gradually until ultimately the rubber became hard and fragile. Longer reaction times increased the amount of sulfur absorbed and after 24 hours the original weight of the rubber had doubled. Regarding the chemical reaction, Payen considered it to be a substitution of hydrogen by sulfur: "Dès que la réaction du soufre commence, et pendant le

temps qu'elle s'effectue, c'est-à-dire pendant le temps où la température reste entre 135 et 145°, il se produit une combinaison du soufre avec une faible quantité d'hydrogène empruntée au caoutchouc, et, par conséquent, une formation continue d'acide sulfhydrique, dont la gomme peut absorber près d'un volume égal au sien... Un phénomène curieux résulte de ce fait, lorsqu'on agit par le procédé de vulcanisation au bain: au moment où, après cette vulcanisation, l'abaissement de température se produit, le soufre, en cristallisant met en liberté une partie de l'hydrogène sulfuré produit et ce gaz se dégage entre les cristaux en soulevant la masse à demi fluide" (From the moment a combination of sulfur has started, and during the time it takes place, that is to say, while the temperature remains between 135 and 145° C, a reaction between sulfur with a very small amount of hydrogen trapped in the rubber takes place, and consequently, a continuous formation of hydrogen sulfide, where the rubber can absorb approximately its own volume... As a result, when vulcanizing in a bath a curious phenomenon occurs as a result of this fact: after the temperature drops sulfur crystallizes and liberates a part of the hydrogen sulfide generated; this gas disengages between the crystals and lifts the semifluid mass).

Today we know that vulcanization consists in the bridging of the double bonds of isoprene with sulfur, this means that the disengagement of small bubbles of hydrogen sulfide observed by Payen was probably due to the rubber being impure with oils and resins.

It is not known if Payen noticed during vulcanization a physical phase during which the rubber absorbed sulfur, and a chemical phase in which the absorbed sulfur reacted with the rubber. Anyhow, did Payen notice the fixation of sulfur in the rubber because he remarked that: "Lorsque le terme convenable n'a été dépassée (c'est -à-dire lorsqu'on n'a pas dépassée le stade souple et élastique pour obtenir une substance dure et fragile), la matière organique recèle du soufre sous deux états différents: 1 à 2 centièmes sont retenus en combinaison intime; le surplus reste simplement interposé dans ses pores" (When the conditions had not been exceeded, that is when the flexible and elastic stage had not been overcome to yield a hard and fragile substance, the organic material held sulfur in two different states: one to two percent were retained in combined form and the rest was simply interposed in the pores). In other words, part of the sulfur had com-

bined and the rest was present as free sulfur. When left alone, the vulcanized sample eliminated the excess sulfur slowly; this process was greatly accelerated by treating the material with hot solutions of sodium or potassium hydroxide, carbon disulfide, turpentine, petroleum ether, or anhydrous ether.

Payen used a microscope to make a detailed study of the structure of rubber, which had been cut in very thin lamella. He observed the presence of “des pores très multiples, arrondis irrégulièrement, communiquant entre eux qui se dilatent même sous l'influence capillaire des liquides sans pouvoir dissolvent pour la substance elle-même” (multiple pores, arranged in irregular manner, communicating between them, and that expand under the capillary influence of liquids that do not dissolve the substance).

Another interesting observation related to the action of different solvents: “L'éther, la benzine, le sulfure de carbone et plusieurs mélanges entre eux ou avec d'autres liquides s'insinuent rapidement dans les pores du caoutchouc, le gonflent beaucoup et semblent le dissoudre, mais ce que, dans ce cas, on considère généralement comme un dissolution complète est, en réalité, le résultant d'une interposition de la partie dissoute dans la portion fortement gonflée, celle-ci ayant conservé les formes primitives amplifiées, et étant alors très facile à désagréger” (Ether, petroleum ether, carbon disulfide and other mixtures between them or with other liquids, penetrate easily in the pores of rubber, they strongly swell it and seem to dissolve it, but in this case, what is usually considered to be a total dissolution, is actually the interposition of the dissolved part in the portion well-swelled that keeps the original material amplified and is easy to separate).

Payen also reported that Gérard, at Grenelle, had observed a new phenomenon that allowed preparing rubber threads of high tenacity. When rubber threads, tensed to six times their initial length, were heated to 100°C, the extension became permanent. The process could be repeated until the final length was about 16 times the original one. The resulting thread had an extremely thin diameter, of smaller dimensions than ever observed.

Payen also studied the properties of gutta-percha (Payen, 1852b), extracted from the sap of *Isocandra percha*. Pure gutta-percha has a rose-brown color, becomes electrically charged by friction, and is a poor conductor of electricity and heat. At room temperature (25°C) it has a remarkable tenacity,

similar to that of rawhide, on heating it becomes softer and sensibly pasty around 48°C. At this temperature it can be laminated into thin lamina and stretched to form tubes. It is easily molded under pressure and temperature and can reproduce with high fidelity the polish and the fine details of the molds. It possesses a remarkable porosity, if it is dissolved in carbon disulfide and spread over a glass plate, the solvent evaporates and leaves a whitish lamina, which, under the microscope shows innumerable cavities. The air retained in the cavities of gutta-percha is responsible for its low density of 0.979.

Gutta-percha was not attacked by alkaline solutions diluted or concentrated, by ammonia, saline solutions, water loaded with carbon dioxide, vegetable acids, diluted mineral acids, and alcoholic solutions, such as beer, wine, and ciders. Concentrated sulfuric acid attacked it releasing substantial amounts of sulfur dioxide. It was also attacked by nitric acid and saturated solutions of hydrogen chloride. Organic solvents such as petroleum ether, turpentine, chloroform, and carbon disulfide dissolved it at room temperature.

Using extractive analysis Payen determined that the raw material could be separated into three fractions: 75-80% of pure gutta, 4-6% of a yellow resin (fluavile), and 14 to 16% of a white crystalline resin (cristalbane or albane), and then proceeded to determine the properties of each fraction. Gutta, the most abundant fraction, had all the properties of the raw material while the two resins were essentially inert.

Plant physiology

Between 1835 and 1842 Payen presented to the Académie des Sciences, seven memoirs related to the development and composition of plants and trees. They were of such quality that the Académie had them published in its *Recueil des Savants Étrangers* (a journal issued by the Académie for publishing important scientific contributions of non-members). They were complemented by two additional memoirs published in 1843 and 1846 with the collaboration of Charles François Brisseau de Mirbel (1776-1854) (Payen and Mirbel, 1843, 1846). His most important scientific studies were *Mémoire sur la Diastase* (Payen and Persoz, 1833) [in conjunction with Jean-François Persoz (1805-1868)], *Composition Élémentaire de l'Amidon* (1836) awarded a prize by the Académie des Sciences in 1840, *Mémoire sur la Composition du Tissu Propre des Plantes et du Ligneaux* (1838), *Manuel du Cours de Chimie Organique Appliquée Aux Arts*

Industrielles et Agricoles (1842-1843), and *Traité de la Distillation des Betteraves Considérée Comme Industrie Annexe des Fermes et des Sucrieries* (1855).

In this huge work Payen was one of the first to establish that the weft of tissue of plants has an identical composition in all the vegetable kingdom and that nitrogenous substances always accompany vegetable organisms since their birth. As stated by Joseph Decaisne (1807-1882), Payen advanced the famous observations of Hugo von Mohl (1805-1876) on the nature of primordial utricle. He realized that nitrogenous substances are essential ingredients for tissue development, and that cellulose, pure or united to immediate principles that do not contain nitrogen in their composition, constitutes the same substance in all vegetable tissues.

Payen dedicated himself to make a detailed study of these immediate principles, he was so successful that his name has become intimately associated to the history of amylaceous substances, to dextrin, to the discovery of diastase, the dextrin splitting enzyme, and to the study of cellulose and the incrusting substances (lignin) found on vegetable fibers.

In 1820 Payen became interested on the influence of nitrogen fertilizer in plant development and decided to study if this influence was exerted simultaneously in all the vegetables, or only in certain tissues. A few years later, Joseph-Louis Gay-Lussac (1778-1850) called the attention of physiologists about the presence of nitrogen in grains and in this important finding Payen found the confirmation of his views (Gay-Lussac and Thenard, 1810). He tried now to generalize his observations, to determine the composition of vegetable tissues at the moment or during their birth, and soon he recognized the crucial role played by nitrogen-containing substances on the nutrition and development of these tissues.

To Payen we owe the discovery that nitrogenous substances endowed with a quaternary composition similar to that present in animal products, are unevenly distributed in vegetable tissues. They accumulate in large quantities in young organs where the vital force of plants is concentrated, that is, in the nascent embryo, in the radicles and gemmula of the grains, in the spongium of the radicles, and in the central part of the undeveloped shoot. They are present in a very small amount in mature tissues; they slowly recede from the different organs as they become older, and begin to accumulate in the fruits, spores, and sporules. These facts permit an easy

interpretation of the role of nitrogenous substances: they are the essential agents of tissue development in the stage where the organs must provide for their own nutrition. Payen found these nitrogenous substances, were present, before that in any other discernable organization, in the cambium, that is, in this organic mucilage where the utricle tissue is born.

A following work was the study of the amylaceous material commonly known as fecula or starch. At that time it was not known the role it played or its chemical composition. It was not known if starch was a unique entity or if plants produced it in a large diversity. In a paper published in 1836 (Payen, 1836) Payen demonstrated that amylaceous substances extracted from roots, stems, and grains of different plants, may vary in their form, dimensions, and state of aggregation, but all contained the same chemical species, having always the same composition, be it that it comes from the tubercles of potatoes, from the grains of beet sugar, from the stems of cacti, or from rhizome of cane. Payen showed that the grains of amylaceous substances did not contain a liquid product and that each of them was formed by a series of minced tunics that intertwined one into the other and increased their cohesion with age. He established that starch is never found in rudimentary tissue but that it accumulates in developed organs where it constitutes a reserve of a material that can be assimilated and capable of contributing, under determined conditions, to the nutrition of the vegetable. In addition, Payen demonstrated that amylaceous material does not dissolve in water, unless transformed, that starch, even the lighter one, remains always a mixture of water and amylaceous granules, swelled, and distended to enormous proportions (Girard, 1871).

Payen is remembered mainly for his work on carbohydrates. In 1833 Payen and Persoz found that starch was hydrolyzed to sugar by a substance contained in malt, which they called *diastase* (the common name today is amylase and is known to be a mixture of extra cellular enzymes), a special substance rich in nitrogen. Diastase is generated in the grain during germination and because of its property of converting amylaceous substances into soluble substances, renders them soluble little by little and transports nutrient sugars to the nascent stem that consolidate it and form tissue. He studied every possible detail of the transformation of starch into dextrin and then into sugar by the action of diastase

and proved how starch, once rendered soluble, went from one tissue to another, as much as to accumulate again, as much as to bind in strong aggregation and participate in this form in the formation of cellular membranes in the tissue. He established the theory of beer fabrication, discovered the process actually used for obtaining dextrin, determined its composition, and showed it to be an isomer of starch.

For his discovery of diastase Payen is considered by many to be the father of biochemistry.

According to Payen, dextrin or sugar traverses the cellular frame already built, forms new tissues next to it and these cells or vessels become "les envelopes protectrices, les réservoirs et les conduits à l'aide desquels les corps animés qui les sécrètent et les façonnent se logent, puisent et charrient leurs aliments, déposent et isolent les matières excrétées" (the protecting envelopes, the reservoirs and conduits through the aide of which live bodies that secret and shape them strongly, and carry their food, depositing and isolating the excreted materials) (Girard, 1871).

This is the fundamental contribution of Payen's work. In 1839 the Académie awarded him the prize for experimental physiology for his works on starch. Payen's work shows that the cell tissue has the same composition as starch, and that it is the same in ovules and fruits such as cucumbers, pith, and the hardest woods, and that in trees these celluloses are agglomerated with a special matter that is the lignin proper. Payen established neatly the distinction between the isomer tissue with starch and lignin. The first one resists the attack of the many agents that attack the other one in a very vigorous manner.

In another piece of work Payen showed that cellulose was part of all vegetable tissues, celluloses, fibers, and vessels or traicheids. Injected with nitrogen material and silica, cellulose formed the epidermis of stems and leaves, solidified by an organic matter richer in carbon, which he called *incrusting substance* (today lignin) it formed the fibers of trees. Cellulose had always the same composition, although its state of aggregation could be different (Girard, 1871). Payen said: "It is understood, that amorphous mineral substances injected within the vegetable membranes, where they constitute an integral part of tissues or nitrogenous compounds, or circulating dissolved in the circulating liquids (sap), serve directly for the development of the living vegetable. Now, several physiologists have asked the question of what can be the role of the crystalline

mineral substances secreted in certain special cells, specially after having observed that once the leaves that have reached their final development fall, without the concretions having changed in volume or aspect. They have concluded that these crystalline mineral concretions represent simple excretions. My viewpoint is that these concretions have a double benefit. During the life of the vegetable the crystalline entities eliminate from circulation many mineral substances absorbed in excess from the soil and that, afterwards, may hurt the normal development of the plant. Plants may choose the mineral elements, which are more useful or favorable in larger proportions for the development of certain species. It is equally true that this capability has its limits beyond which these substances became noxious. Gathered in crystalline form in the special organisms, they cease their detrimental action. At the time of the fall of the leaves, or at the death of the plant, their agglomeration kept in reserve, accomplish a useful task, in restituting to the ground the mineral substances taken during the life of the plant from the deep layers of the soil. Once developed and having acquired all their volume, remain without change until the leaves that hold them fall" (Girard, 1871).

Until 1838 wood was believed to be a simple carbohydrate material, containing varying proportions of carbon according to the species, but with the ratio of hydrogen to oxygen fixed in the proportion of water. The slight excess of hydrogen found experimentally in certain species, particularly the hard woods had not been explained. Gay-Lussac and Thenard had analyzed oak and beech trees and found that ligneous matter was formed by 53% carbon and 47% water. Dumas recognized that Gay-Lussac and Thenard had been too hasty in their conclusion (Payen, 1839a,b). Payen was the first to attempt the separation of wood into its components and to show that wood was not a single proximate compound but was composed of at least two very distinct chemical parts. He treated wood with nitric acid and obtained a fibrous substance, which was relatively resistant to this reagent. Payen called this substance *cellulose*, a term that he coined and introduced in chemical literature. He chose this name because cellulose was the basic constituent of the cell wall of plants. He showed that cellulose had the same elemental analysis as starch and was apparently isomeric with it. He found that irrespective of its origin, cellulose had always the same chemical composition, which could be represented by the formula

$C_6H_{10}O_5$. Payen also found that in isolating cellulose he had to remove a substance or group of substances, which had a higher percentage of carbon than cellulose. Payen called these substances incrusting materials, les *matières encrustantes* (lignin), and considered that the cellulose was mechanically incrustated or impregnated by them (Payen, 1939c). Payen found that tree contained 54% carbon, 6.2% hydrogen, and 39.9% oxygen, that is, it more hydrogen than that needed for converting its oxygen into water (which corresponds to the now accepted empirical formula $C_6H_{10}O_5$). His results indicated that lignin corresponded to a class of compounds different from the starch that accompanied it. Payen dissolved the latter matter with solutions of nitric acid and sodium hydroxide and isolated the material. The residue gave 44% carbon and 56% water, while the tree had the above analysis. The gross composition of the incrusting matter was $C_{3.5}H_{2.4}O_{10}$. It had about 1% more hydrogen than was required to form water with its oxygen, it was attacked by nitric acid releasing shining vapors, and concentrated sulfuric acid and hydrogen chloride colored it strongly. These properties differentiated lignin clearly from cellulose, in addition to the empirical formula (Payen, 1839a,b).

In order to obtain the purest possible cellulose, Payen subjected treated cotton fiber with hydrogen chloride. After washing and drying, the fibers could be readily pulverized (Payen, 1839a,b). He went on to test if the white matter of the ligneous material, cellulose, having the same elementary composition of dextrin, had a similar power for rotating polarized light. To do so he prepared a solution of cellulose by slow heating in concentrated sulfuric acid. The resulting dextrin solution was limpid and not only it rotated light but the rotatory power per unit mass was in the same in value and direction as that of dextrin. Hence, in the three substances cellulose, starch, and dextrin having different properties, the same body was present in different aggregation states (Phillips, 1940; Reid, 1940).

The Académie des Sciences appointed Antoine Louis Brongniart (1742-1804), Théophile-Jules Pelouze (1807-1867), and Jean-Baptiste André Dumas (1800-1884), to examine and verify the results of Payen's work. Dumas report confirmed these results in all aspects. He says "Wood is formed of cells identical with elder pitch in composition and more or less filled with a material richer in carbon and hydrogen, which nitric acid dissolves" (Payen,

1839c). After commenting on the identical composition of cellulose, starch, and dextrin, and noting that Payen had converted cellulose into the later by means of concentrated sulfuric acid, Dumas made the following discerning statement: "Here we have, then, three substances which have properties very characteristic and very distinct in the eyes of chemists: cellulose, starch, and dextrin, in which one is disposed, however, to see the same substance in different stages of aggregation." ■

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- 2) Un índice alfabético de los 837 artículos que han aparecido en la revista desde el volumen 1 número 0 (julio de 1989) hasta el volumen 15 número 4 (octubre de 2004).
- 3) Los 61 números de la revista completos, incluidas desde sus portadas hasta todas sus páginas.

4) El Acrobat Reader 7.0 listo para instalar en su máquina con sistema operativo Microsoft Windows XP.

La presente colección de *Educación Química* ha sido preparada de manera que permite buscar CUALQUIERA de las palabras (tema o autor, por ejemplo) que esté contenida en todos y cada uno de los ejemplares de la revista publicados de 1989 a 2004. El resultado es impresionante, pues aparecen TODAS las veces que aparecen las palabras buscadas en TODAS las revistas de 1989 a 2004.

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