The Beginnings of Chemical Engineering

THE DESIGN OF PLATINUM BOILERS FOR SULPHURIC ACID CONCENTRATION

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The beginnings of the development of chemical engineering as a science and technique appear about the middle of the last century and it is interesting to explore the circumstances that brought them about. There seems little doubt that the real start took place in the sulphuric acid industry, when the invention by Roebuck and Garbett of the chamber process in 1746 set many problems in construction, the choice of materials, the control of temperatures, the transfer of heat, the flow of highly corrosive liquids and so on. The earliest use of the term "chemical engineer" so far noticed is in the first edition (1839) of Ure's *Dictionary of Arts, Manufactures and Mines* in the article on sulphuric acid (page 1220) where there is the statement: "provision should therefore be made against this event by the chemical engineer".

Little record of the earliest achievements in this field has come down to us until we reach the time when the platinum boiler was introduced for the concentration of the relatively dilute acid yielded by the chamber process, and had to compete very strongly with the glass retorts already in established use. The high cost of the metal was, of course, a great handicap, and forced a development of the highest possible efficiency with the thinnest possible vessel walls, and this in turn compelled scientific attention to a whole series of fundamental principles of chemical engineering that previously had been subject merely to rule-of-thumb. The story is instructive and can be reconstructed from the literature and from the records of the manufacturers.

From the painstaking researches of the late Mr. L. F. Gilbert, there seems no doubt that the first boiler for this purpose was made in 1805 by that great pioneer in platinum, W. H. Wollaston, for one Philip Sandman, a small-scale manufacturer of vitriol in Southwark, and after this, between 1809 and 1818 Wollaston went on to make fifteen more such vessels. Mr. Gilbert reports that they were plain boiling-down pots "usually truncated cones, each narrowing slightly to a bottom that was convex externally". They held from 24 to 47 gallons each and were heated from the bottom in sandbaths. A charge of weak acid was put in and then boiled down to the right gravity in a discontinuous operation, being poured or siphoned out and the operation repeated. In some cases a platinum siphon was provided to help the discharge.

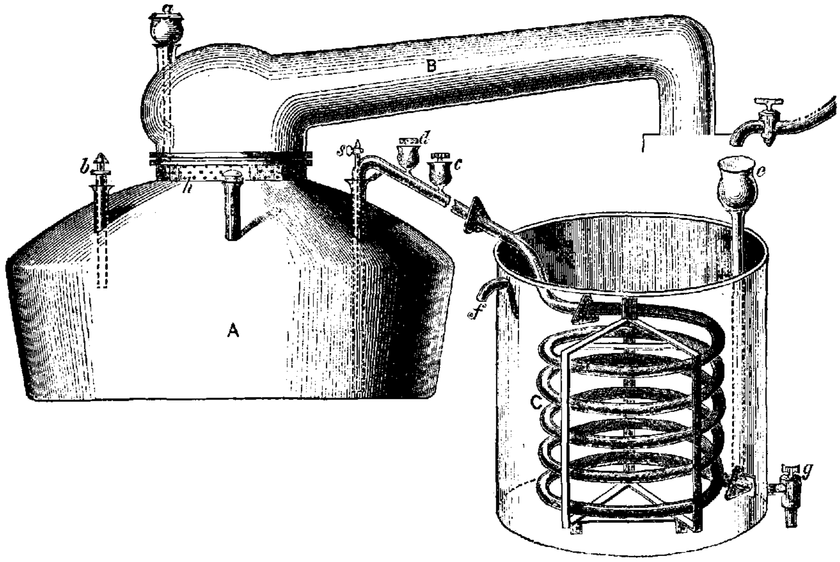
As is well known, Wollaston kept this work in his own hands and after about 1820 it ceased. One of the reasons for this was no doubt Wollaston's increasing preoccupation with his other scientific work but another was undoubtedly competition from France. There the refining of platinum had been set up on a commercial scale by J. R. Bréant, backed by the scientific knowledge of Vauquelin, and the refinery was served by a first-class fabricator named Couturier. He at once entered into the boiler business and his "alembics" became famous. The trouble about Wollaston was that his ingots of metal were small and therefore his sheets were small. These he soldered together by means of fine gold and the joints so made did not take kindly to the discontinuous process of heating and cooling that has been outlined. Consequently leakage and fume and waste of acid were considerable. Bréant, on the other hand, set himself to produce large ingots from which he obtained single sheets up to four feet square. This, of course, reduced the number of joints considerably, but Couturier was not content with that. Before very long he had achieved an autogenous platinum joint by forge-welding. As the use for them of the term "alembic" indicates, the boilers were now provided with a retort-head with a swan-neck passing to the condenser and they were heated in seatings made in a hot flue.

Continuous Operation

The technique of boiler making in platinum was still further improved in the eighteen-forties in Paris by Adrien Quennessen, who exhibited at the London Exhibition of 1851 a very fine specimen of 30-gallon size "made in one piece without seam or solder". This was a challenge to the English who had been out of the business since 1820 and it was responded to by George Matthey, the active young junior partner of Johnson and Matthey, a firm that had been working quietly in platinum for many years but had so far produced no remarkable developments. He realised that if platinum boilers were to make progress and to win the battle with glass, which the conservative acid manufacturers of England favoured strongly, they had got to offer a continuous operation and one economic in metal, in time and in fuel. The French boilers differed very little from those of Wollaston. The process was still always discontinuous and the only improvement had been the introduction by Bréant of a water-cooled siphon. The heating, too, was effected most inefficiently by bottom heat on to a deep volume of liquor, and it was to this point that Matthey gave his first attention in collaboration with "an eminent chemical engineer" named William Petrie of Charlton in Kent. Where this man gained his experience and how he obtained his eminence we have no idea, but the evidence of his work shows that there was some solid basis of fact in it. The first fruit of the collaboration was shown at the Paris Exhibition of 1855. This boiler had a much larger bottom surface for exposure to the heat and consequently worked with a much less depth of acid (actually two inches only). Heat transfer was further improved by heating "by means of direct radiation from the surface of a clear fire, as distinguished from heating by a draught through flues in part out of sight of the fire". Moreover the boiler was arranged for continuous operation by direct feed and withdrawal by siphon. It was lined with gold to lessen the effects of corrosion on the platinum ; its diameter was about 33 inches, its height to the neck 20 inches ; its capacity 40 gallons ; and its output 34 cwt. of strong acid per day of continuous working.

Design Considerations

By July 1859 considerable further advances had been made in boiler design, as is shown in a letter from Johnson and Matthey to their American agent in which they tell him that in designing these vessels, the most attention must be paid to the area of the bottom and sides rather than to volume. "The gallons of liquid per hour which the vessel will rectify or distil depends on the number of square feet of heat-absorbing surface of the vessel, while the bulk of liquid contained at one time is quite unimportant if the system of continuous running in and out at the same time be adopted." They suggest that the liquid should be worked through about three vessels in succession connected by siphons, as this prevents any mixture of unrectified liquor with that which is running out in a rectified



A platinum boiler exhibited by Johnson Matthey at the Paris exhibition in 1867. This had a capacity of 8 tons per day

state. "By having the bottom of the vessel supported on iron bars made smooth to fit well against the metal, the new form can have it wholly exposed to the full heat of the fire, and this system of boiling is far more advantageous than when the heat is applied to an equal surface placed more vertically, as the sides of the vessel are, for the bubbles clear themselves more rapidly from the bottom, while in ascending from the sides they obstruct the heat besides not heating the rest of the liquor by passing so completely through it." The circular bottom of this boiler had a diameter of 24 inches, and the conical sides sloped inward to give greater mechanical strength than the former outward slope, to 8 inches at the head.

The next model was shown at the London Exhibition of 1862. The chief new feature was that the rectified acid left the boiler by a tube passing through the side from a separate compartment in the centre, and passed to an arrangement for cooling it by preheating the cold feed. This vessel produced more than two tons of strong acid per day, and was the last type to be lined with gold. This change represented a considerable economy, since gold then cost three times as much as platinum and was permitted by improvements in the quality of the latter, as well as by the discovery by Pelouze that a small addition of ammonium sulphate produced enough sulphur dioxide to destroy the traces of nitrous compounds which caused most of the corrosion.

After 1861 an entirely new factor was introduced into the construction of the boilers by George Matthey's invention of the fusion welding of platinum, which made possible fully autogenous, and therefore much stronger, joints.

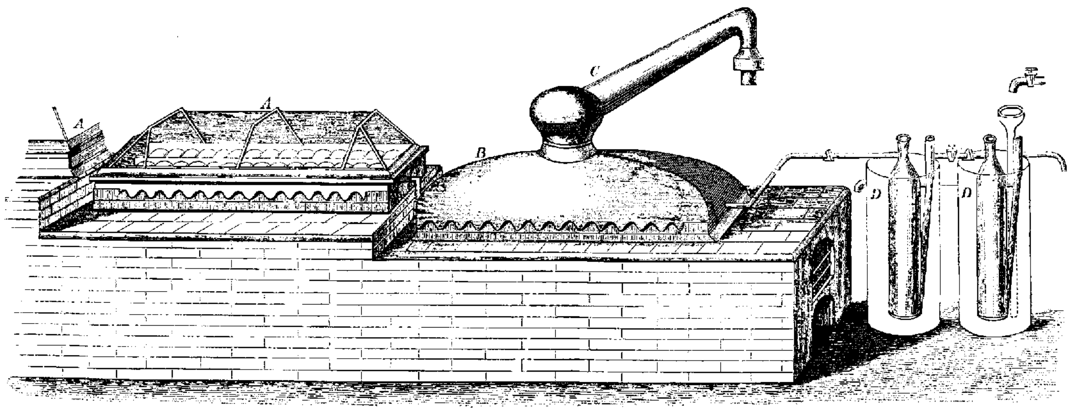
As time went on the capacity of the boiler installations increased and in August 1865 a quotation was made for one to rectify five tons per day. Improvements made about that time include the feeding of the cold acid through a sort of collar perforated with small holes, through which it fell into the hot acid in the form of rain instead of in a single stream, which prevented "bumping" and consequent strain on the vessel. Also the swan-neck now sloped upwards to the condenser instead of downwards, so that splashes were returned. In the Paris Exhibition of 1867 a boiler was shown which yielded 8 tons per day.

The next advance consisted in providing the boiler with a corrugated bottom (Prentice's patent), which gave greater strength and greater surface for heat transmission.

In 1876 came the final development in design, according to the patent of M. Gustav Delplace of Namur, Belgium. This provided a vessel of an elongated square or oval shape, with a very flat structure, into which Prentice's corrugated bottom was often incorporated. The flat design resulted in considerable economy in the weight of platinum used, and in working with a very thin layer of acid and correspondingly increased evaporating power. For a make of four to six tons of acid per day the still measured three feet by one foot six inches and normally two were worked in series "to provide a degree of concentration higher than any before obtained commercially in platinum apparatus worked continuously." From this time onwards there was little change in the general principles of the design of the boilers, and they remained a cardinal feature of Johnson Matthey's work for the rest of the century and into the early years of the next one.

It must not be thought from the above that Johnson Matthey had a monopoly in the supply of these boilers. From the beginning there was keen competition from France, where the successors of Bréant and Couturier, and also Quennessen himself, came together in a partnership for which the most convenient name is Desmoutis Quennessen (it varied as third partners came and went). This carried on the work of its constituent members and at first was more successful in converting the Continental acid manufacturers to the use of platinum than the British firm was with those of England. The French were early in the introduction of continuous working and the "rain" feed, and they also had a system of internal partitions which prolonged the flow of the acid through the still and prevented the mixture of incoming and outgoing liquid.

In 1863 competition was further stimulated by the introduction in Germany by Faure and Kessler of a system of evaporation in two large flat platinum dishes arranged in cascade. This gave a very good performance and forced the English and French firms to improve their products. In the struggle the former proved the most responsive, and the application of their study of the principles of chemical engineering proved a decisive factor in their leadership in the boiler field.



A Delplace boiler, built by Johnson Matthey in 1876 and incorporating the corrugated bottom devised by Prentice