Production and Use of Nicotine

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Tobacco is produced the world over. Fifty-four countries grow about 7 billion pounds of it annually on 7¾ million acres. It is smoked in cigarettes, cigars, and pipes. It is chewed in its natural leaf state or, ground to a powder to which various materials have been added, as snuff. Or it may be treated with a mixture of sugars, honey, licorice, and other flavors, and oil and formed into plugs, ropes, or pellets for chewing or smoking. A few people snuff it into the nostrils to clear the head. Some call it a filthy weed, but the fact is that tobacco has tremendous economic significance.

Varieties, soil, cultural practices, and curing methods develop characteristic properties of the leaf. Curing methods include natural air-curing, sun-curing, fire-curing (in which smoke is used to add flavor and aroma), and flue-curing (in which heat produces a bright yellow leaf). Perique is an unusual tobacco which is processed entirely by the farmer, who uses an anaerobic fermentation to produce it. In parts of India and Pakistan bulk fermentation of the green leaf is part of the curing process.

The species *Nicotiana tabacum* supplies most of the world's tobacco, and it is the only species grown in the United States for tobacco purposes. The species *Nicotiana rustica,* which was being used by the North American Indians when the first white explorers reached the continent, is now grown extensively in India, Pakistan, and the Soviet Union, and to a lesser extent in Arabia, Persia, Syria, Abyssinia, Poland, and Hungary. The total production of *N. rustica* is estimated to be 750 million pounds from 700,000 acres. It is used for smoking, chewing, snuff, and nicotine extraction.

The alkaloid nicotine is an important insecticide. It is used universally to protect plants and animals against pests and parasites. It is especially valuable as a contact insecticide for the control of aphids, scales, and leafhoppers. In some of its fixed forms it is a stomach poison for chewing insects. Its effective control of scab and ticks has made sheep husbandry possible in many parts of the world.

The United States production of this alkaloid is approximately 1,350,000 pounds a year. We usually export about 120,000 pounds, although in 1949 we shipped more than 400,000 pounds abroad. The annual production in other countries (except Soviet Russia, for which we have no figures) is more than 500,000 pounds of alkaloid. Nicotine products are made in England, France, Algeria, Italy, Germany, Hungary, Switzerland, Japan, and Soviet Russia. More recently, small developments have been reported under way in Australia, Brazil, Rhodesia, and South Africa. The government tobacco monopolies of Turkey and Thailand have announced that nicotine-extraction plants will be part of projected tobacco factories.

Most of the nicotine comes from stems or midribs and other wastes and dusts discarded in the manufacture of tobacco products from the leaves of *Nicotiana tabacum*. Because our consumption of tobacco has been increasing steadily, an increasing amount of factory byproducts is going to the nicotine extractor. At the same time the demand for nicotine has kept pace with the available supplies. The net amount of nicotine now available, however, is not in proportion to the increased tonnage of tobacco byproducts. Improved agronomic practices have reduced the nicotine content of our domestic cigarette tobaccos. Furthermore, increased freight charges have tended to limit the area to which byproducts can be profitably shipped for extraction.

At times the prices of low-grade leaf tobaccos of the dark fired and heavy air-cured types permitted their use for nicotine production. That use has not been possible since 1939, when support prices established values for the low grades above their nicotine values. Two subsidized diversion programs in 1943 and 1944, however, used low and medium grades of surplus leaf to obtain enough nicotine for food production for the war effort. For those reasons, the suggestion has been made to grow *Nicotiana rustica,* a species that has a high content of nicotine, solely for the alkaloid.

NICOTINE, $C_{10}H_{14}N_2$, in its pure state is a colorless liquid with a faint characteristic odor. Its vapor pressure at 68° F. is only 0.08 millimeter of mercury. The pure alkaloid volatilizes readily when exposed to the air, although part of it may turn into a dark resinous material.

Nicotine is a basic compound, which forms salts with acids. The simple salts do not readily crystallize, most of them being hygroscopic. Others, however, such as benzoate, oxalate, salicylate, and tartrate, are crystalline solids and are stable under atmospheric conditions. Nearly all of them are readily soluble in water. There are several water-insoluble nicotine compounds, like the combinations with the bentonites, some of the zeolites, tannic acid, peat, and certain resins. The bentonite and tannate compounds have found commercial application.

A rather comprehensive series of more complex metal double salts has been prepared by combining the metal and the nicotine salts of the same acid. Another series of nicotinammino compounds has been prepared by combining the alkaloid with a metal salt of the selected acid. Many of these are insoluble in water, and so far have been of interest chiefly in fundamental insecticide studies. A few that contain copper or cadmium show fungicidal properties. Nicotine readily combines with certain of the dye acids to form water-insoluble compounds, but their practical application is yet to be determined.

Nicotine can be oxidized rather readily with certain oxidizing agents to nicotinic acid and nicotinonitrile, the nitrile of nicotine acid, which have become important as antipellagra vitamins.

TOBACCO AND ITS EXTRACTS were used for the control of insects long before it was known that nicotine was the toxic agent. The first reference to the use of a tobacco extract for spraying plants was in 1690. English and continental gardeners early recognized the value of tobacco from the American colonies. According to present standards it must have been strong tobacco. In a letter dated January 20, 1734, Peter Collison of London suggested to his American correspondent, John Bartram, the Philadelphia botanist, the use of tobacco leaves to protect letters and packages containing seeds and plants being shipped to him. In 1746 he advised Bartram to use a water extract of tobacco for the control of the plum curculio on nectarine trees.

Tobacco dusts and extracts were recommended for the control of plant lice in France in 1763. In 1773 Richard Weston developed a hand bellows for fumigating insects with tobacco smoke. The first American reference was in 1814 by Peter W. Yates of Albany, who used tobacco water against sucking insects. William Cobbett, in England, in 1829 recommended tobacco extract for the control of the woolly aphid. Thomas Fessenden in 1832 included tobacco in a list of insect repellents and insecticides.

By 1884 tobacco was described as one of the three most valuable insecticides in general use, the other two being white hellebore and soap. Tobacco was then used as a dust or water extract, or the leaves and stems were burned as a smudge for fumigating greenhouses. Specially prepared papers impregnated with tobacco extracts were extensively used. Special burners were developed for burning the tobacco or nicotine preparations and to blow the heavy smoke into the greenhouses or about the infested plants. Tobacco washes, prepared by gardeners and horticulturists from specially prepared tobacco leaf and stem materials, were in common use. They preceded the tobacco extracts soon to be manufactured commercially.

Commercial tobacco extracts found extensive use for the control of sheep ticks and scabies. In the early years, the extracts were often prepared from leaf tobacco, tobacco dust, or tobacco stems at the site of the dipping vats. Later, commercial extracts were offered in Europe, Australia, New Zealand, South America, and the United States. The first manufactured products were simple water extracts that contained 1 to 10 percent nicotine. In 1895 a product containing 40 percent free nicotine was offered to the trade, followed in 1898 with one containing 80 percent free alkaloid.

In 1910 a product containing 40 percent nicotine as the sulfate appeared. It marked the beginning of the modern nicotine business, as it made possible transportation in gallon cans instead of casks or large hogsheads, previously required to carry the weaker extracts or the raw leaf and stems to supply the same amounts of active alkaloid. The 40-percent nicotine sulfate offered fewer hazards in handling in sheep dips and for other applications than the pure alkaloid, and it proved more effective because it also contained other distillation products of tobacco with adhesive properties.

Today nicotine still has its widest use in agriculture as a contact insecticide for the control of important groups of sucking insects, such as aphids and scales. For these insects, it is used as a dilute spray, or as a dust, or it is vaporized in smoke or other aerosol form. In those forms, the alkaloid is readily available, and its lethal action is immediate upon contact with an insect. A similar action, but of more extended duration, follows the use of nicotine sulfate alone, as in the control of poultry lice and mites. Here the alkaloid is released more slowly over a period of 24 to 48 hours. In a third form—the so-called fixed nicotine combination—the alkaloid is very slowly given off over a relatively long period, often up to 30 days. The fixed forms include nicotine bentonite, bentonite fused with sulfur combined with nicotine, and nicotine tannate. Materials of this type act as a stomach poison to leaf-chewing insects.

The development of fixed nicotines, like nicotine tannate and nicotine bentonite, marked an advance in insecticide chemistry, and greatly broadened the field of nicotine usage. Before their discoveries, nicotine was used almost entirely for quick action as a contact insecticide in a nascent form. The use of nicotine sulfate on poultry roosts, however, takes advantage of rather slow volatility. In the fixed forms, nicotine is an excellent and effective stomach poison for certain insects of economic importance. Other combinations may have wider applications. Moreover, nicotine is unique among insecticides in that it is relatively safe, because it ultimately disappears, even in the fixed forms, and leaves no residues that might be dangerous to consumers of food products sprayed with it.

A nicotine bentonite preparation also is used widely to control internal poultry parasites. A mixture of nicotine sulfate and copper sulfate gives effective control of some internal parasites of sheep.

The pure alkaloid, offered in concentrations of 50, 80, 95, and 98 percent, is adapted for direct fumigation, especially in greenhouses, but also in fields and orchards. This form has been replaced in part by smoke aerosols, where the nicotine is impregnated on paper, on tobacco dust, or on a similar slow-burning material so as to make a smudge quickly. Special burners have been developed for making the smudges. A combination of nicotine and DDT in a tobacco base makes an effective smudge for freeing an area of mosquitoes.

Another type of smudge, or vaporized nicotine fumigant, is offered in a mixture containing 80 percent nicotine alkaloid and 18 percent oil. The mixture, when dispensed by a heating device, has proved effective in treating large acreages. Nicotine has also been successfully used in the bomb-type aerosols, in which materials of low boiling point furnish the impelling power.

Nicotine-bearing dusts are available in a wide range of alkaloid contents. Some are prepared from the natural leaf tobacco and contain 0.45 to 1.0 percent nicotine. When prepared from air-cured tobaccos, they are alkaline in nature and slowly give up their nicotine on exposure to the air. When they are prepared from flue-cured tobacco, which is acid in nature, the nicotine is less readily given off. The acid-type material is adapted for use in controlling some internal parasites of poultry and sheep.

Concentrated tobacco dusts that carry 10 or 20 percent nicotine are available. They are used as a dust base and are diluted to the desired nicotine content with tobacco dust of very low nicotine content, with pyrophyllite, or with certain talcs or clays. When they are reduced to 4 percent or less of nicotine they are easily dustable and effective.

Nicotine is compatible with most of the other insecticides and standard fungicides used today. Because of its versatility, a large part of its commercial and home-garden usage (when several insects and plant pathogens are to be controlled in one application) is in combination sprays and dusts.

Statistics on the use of nicotine in any one year or on any one crop are difficult to compile because of the year-to-year variations in infestations of insects it can control. When used as a contact insecticide, nicotine acts quickly, and the commercial grower usually does not apply it until an infestation is present. On the other hand, insecticides for chewing insects are often applied as a regular routine, and usually ahead of an expected insect emergence. Thus, when the more stable forms of fixed nicotine are used (for example, for the codling moth on apple or the European corn borer on sweet corn) their application is on a more or less regular schedule, ahead of the insect's appearance.

Some of the newly developed synthetic organic substances that threaten the position of nicotine as an insecticide for use on certain crops carry hazards in their application and in their residues. Their permanent position is not yet clear, despite their insecticidal efficiency. The demand for nicotine continues to be as strong as ever.

An estimate of the major uses of nicotine in American agriculture for an average year is shown in the table on page 772. The variation in the probable insect infestation may raise or lower the quantities indicated for any one crop from year to year.

Tobacco sauces for flavoring chewing and smoking tobaccos were, strangely enough, the first manufactured tobacco extracts employed in agriculture. They were extensively used in Europe, especially in Germany and Scandinavia. Such tobacco extracts were manufactured in Europe and the United States. Sometimes they were produced from the finest American tobaccos. The extracts were often of special formulation as to type and grades of tobacco and were designed for particular usages. Their nicotine content varied from 1 to 10 percent, depending on formulations and degree of concentration.

One reason for the rapidly growing market that developed for nicotine products was that entomologists and veterinarians who joined agricultural pest-control agencies relied more and more on the products in the fight against increasing infestations. They suggested that the sources of tobacco then available be improved. Tobacco solutions that once were poured away after having served their purpose in tobacco factories were reclaimed and sold for insect control. The effective control of sheep mites and ticks with nicotine dips created a large market for nicotine products. Outbreaks of the parasites during the 1890's brought about Federal regulations in the United States and made sheep dipping mandatory for interstate shipments. Important markets for nicotine had also been developed in other sheep-growing areas. The chemist and chemical engineer were brought into the tobacco business in order to manufacture concentrated products more efficiently.

The expanding demand for nicotine caused the development of methods for recovering the alkaloid from tobacco extracts. The alkaloid was removed in whole or in part from such extracts, which thereby improved in quality, because nicotine was neither desirable nor required in flavoring extracts. Continued progress was made in purifying and concentrating the insecticide product.

Before 1880, tobacco flavoring sauces and home-made tobacco extracts were in general use. In 1881 extracts carrying 2 to 3 percent nicotine were offered. The alkaloid content was increased to 6 and 10 percent by 1885. A 40-percent solution of free alkaloid appeared in 1895; an 80-percent solution, primarily for greenhouse use, was put out in 1898. Nicotine sulfate appeared in 1900, first with 10 percent alkaloid and later with 40 percent. It has since become the standard nicotine solution of the agricultural trade.

Nicotine tannate appeared in 1929 as the first fixed nicotine compound. It was followed in 1933 by nicotine bentonite. The alkaloid of 99.9 percent purity was offered for commercial use in 1940.

Because of a reduced demand in Europe for strong-flavored tobaccos, the once important flavoring-extract business has greatly declined. On the other hand, the demand for nicotine has continued to increase, and extraction units of substantial capacities have developed.

NICOTINE AND OTHER ALKALOIDS can be removed from tobacco by dry distillation, by water or hydrocarbon solvents, and by steam distillation. In the United States most of the extraction is by steam distillation in a continuous or batch process. The alkaloid is recovered by liquid-liquid extraction, or by scrubbing the alkaloid from the vapors with sulfuric acid. Ion-exchange reactions have been suggested for recovering nicotine from solutions.

The stems and other materials are usually cut or ground to pass a ¼-inch screen. A lime slurry is added to free the alkaloids and to impart from 22 to 35 percent of moisture to the material.

Continuous extraction is done in a rotary steam-tube drier. The drier may be 50 to 55 feet long and 6 feet in diameter. It is usually equipped with two rows of heating tubes with steam at a pressure of 100 pounds per square inch, gage, equivalent to 338° F. The revolving movement keeps the material in constant motion as it passes down the slightly inclined drier. In the first part of the drier the tobacco material is maintained on a moist basis by steam. Heat from the tubes of the drier drives off the moisture, carrying with it the alkaloids and ammonia vapors. The rate of flow of the tobacco is so adjusted by the operator that the recovery of the original nicotine content is practically complete within an hour; the moisture of the extracted material as it leaves the drier is about 5 percent. The dried residue, containing important amounts of nitrogen and potash, is used as a fertilizer material.

Instead of a continuous extracting system, a batch method may be used. It employs rotary driers, which hold about 4 tons of tobacco material to which a lime and water slurry has been added. Stripping steam at 300° F. or higher is fed into the driers. The material stays in the driers for 2 to 3½ hours at 300° F. or higher. Under those conditions, about 95 percent of the contained nicotine is extracted. At the completion of the run, a vacuum is drawn on the drier, which removes some of the fine dust and obnoxious vapors that otherwise would saturate the atmosphere in the drier building.

In either system, the vapors pass first through a dust collector. They may then be sent through a sulfuric acid scrubbing tower to recover the nicotine as sulfate, or they may be condensed and the nicotine alkaloid removed by liquid-liquid extraction.

Where an acid-recovery system is used at substantially atmospheric pressure, the vapors are maintained at a temperature above 212° F., so that the steam in the vapors does not condense in the recovery system, thus diluting the acid solution. The acid towers are arranged in series to allow one of them to be cut out when the product has reached maximum concentration.

The concentrated product can seldom be brought to the usually desired alkaloid content of 40 percent in the recovery system. It is cooled and the excess ammonium sulfate is crystallized out, the nicotine sulfate layer being decanted. This layer is then brought up to the desired strength, usually about 40.25 percent nicotine content, by adding sufficient pure alkaloid to react with the free sulfuric acid. Another method of concentrating the nicotine sulfate solution is to draw it off from the towers at around 10 percent concentration, and evaporate this to 40 percent nicotine content in an open kettle or under vacuum.

In a third method, the vapors from the still are condensed and the nicotine is recovered by a liquid-liquid extraction process. Advantage is taken of the solubility of nicotine in some of the hydrocarbons, such as hot kerosene. In practice, the warm condensate from the drier (temperature about 180° F.) is passed through a packed column, in which the kerosene or other solvent passes countercurrently. These columns are high enough to allow complete removal of the nicotine from the condensate. The heated kerosene enters the extracting column at the bottom, and the condensate with the nicotine enters at the top. Such columns are usually packed with porcelain rings or saddles for nearly their entire length in order to expose as much surface as possible to the action of the solvent. The warm kerosene absorbs the alkaloid from the water solution as it meets the solvent. The condensate, now free of nicotine, goes to waste, carrying with it whatever ammonia it might contain. The recovery of the ammonia in this system usually costs more than it is worth.

The kerosene-nicotine solution flows to a series of tanks where it is mixed successively with 25-percent solutions of sulfuric acid. The mixing can be effected with a centrifugal pump system ahead of the tanks. The sulfuric acid reacts with the nicotine and removes it completely from the kerosene. The kerosene is returned to storage to be used again in the system.

When the nicotine sulfate solution in the first tank contains approximately 40 percent alkaloid, it is centrifuged to remove any tarry and resinous material. It is then made up accurately to 40.25 percent alkaloid content. The solution is adjusted with alkali to pH 6 for safety in transport in metal containers.

The direct acid recovery in a tower system is probably the more economical method for recovering and concentrating nicotine vapors to the sulfate. Its advantage is that it is a direct, continuous operation and involves the minimum of product handling. The distilled nicotine vapors are accompanied by evolved ammonia, which encumber the system with ammonium sulfate. The amount of ammonia varies with the kind of tobacco material used.

BECAUSE OF THE CONTINUED strong demand for nicotine, the increased prices of low grades of leaf tobacco, and increased freight rates for moving the raw material, experiments have been under way for several years to find an environment in the United States where high nicotine-producing strains of *Nicotiana rustica* might be profitably grown.

Extract plants in the growing areas might put out the finished product, like 40-percent nicotine sulfate, or a semifinished product of perhaps 10 percent nicotine content which can be shipped to a central plant for purification and concentration. In the absence of available low-grade leaf tobacco, the American production of nicotine now is confined to large plant operations, where the supplies of raw material consumed are thousands of tons annually. Such raw material now is of relatively low nicotine content—from 0.3 to 1 percent. In order to use high-nicotine material like *N. rustica,* which has 6 to 10 percent nicotine content in the leaves and a correspondingly high ammonia content, special methods were developed at the Eastern Regional Research Laboratory. These, however, do not differ in essential detail from those used in processing tobacco-factory byproducts. They are designed for small factory operations which utilize the whole plant (stalks and leaves) and which might process about 2,000 tons of high-quality raw material annually.

Yields of 125 to 325 pounds of nicotine per acre have been obtained with *N. rustica.* Even the lower yield should give the producer a profit. Because of the relatively high income per acre now obtained in most tobacco-growing areas, as compared with that which might be expected from a nicotine crop, it is believed that the growing of *N. rustica* will find interested growers only outside of the established tobacco-growing areas, and in such areas that might use a cash crop of medium value.

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WHILE I was traveling in the interior of the province of Kuangtung in the south of China, I felt concern that only the land in the valleys right along the streams was cultivated, while thousands of acres of hill lands were left to grow in grass and weeds. I realized that rice farmers could not profitably grow rice on hill lands, but I remarked to my interpreter that no doubt in the past these sections had been covered with subtropical forests. I asked him if trees would not grow again.

"Yes," he replied, "they will grow well, but there are not enough soldiers."

His answer puzzled me, and I asked what soldiers had to do with planting trees.

"Nothing," he said, "but if we did not put a soldier beside each tree guarding it day and night, the people would cut them down for firewood."—*Hubert Maness, Agricultural Attaché, American Embassy, Montevideo, Uruguay.*