Plant equipment for the production of essential oils and aroma chemicals

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Aromatic substances have been extracted from plants for use in medicines, perfumes, and cosmetics since the early history of man. The techniques evolved over the centuries for their isolation have kept pace with man's technological development. It is probable that collection of aromatic gums and balsams as exudates was initally used. Extractive techniques such as "enfleurage" and pressing of oils from source material followed. The development of steam distillation substantially widened the range of plant essential oils which would be processed. The market for these oils and recognition of their properties as related to species and conditions of growing led to the cultivation of economic crops solely for harvesting these essential oils. In many cases the relatively short critical period for harvesting and the need for rapid workup of the plant material required the use of field stills for maximum yield. This is particularly true for leaf and flower oils which are subject to rapid deterioration. Those oils derived from wood, bark, and seeds can be isolated at processing sites other than the collection sites either in the country or origin (to take advantage of lower labor costs) or in countries of major use where there is more modern and efficient apparatus.

Steam distillation equipment in the fields was fairly primitive originally and ranged in capacity from one drum or less to 500 gallons. The distillation could be either a water distillation, a dry steam distillation using false bottom equipment or a grid support, or a water and steam distillation; the type of distillation affects the quality and yield of the product. The stills could be fixed in the ground or mobile, requiring only a source of fuel and cooling water.

In the mint fields of the United States and in the production of clary sage oil, a series of Cobb stills are employed. Trucks and trailer bodies transport the plant material to the stills. The still lid is placed over the top of the trailer body and low pressure steam lines are attached at the bottom of the body (see photo). After the distillation process, the truck and trailers are detached from the steam lines and the spent contents are redistributed on the fields for their fertilization value. In the case of clary sage, an intermediate extraction is introduced to recover the valuable "sclareol" after distillation of the essential oil.

The growth in demand, the rising cost of the

Cobb stills used in dry sage and mint processing.

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essential oil, and the increasing knowledge of the composition of oils have led to efforts to synthesize aromatic compounds in order to minimize dependence on laborious and capricious agriculture production. This has brought about considerable expansion in chemical manufacturing and a technical explosion in synthetic chemical ingenuity. The need for a diversity of chemical entities has resulted in the use of the most modern manufacturing techniques and advanced chemical engineering knowledge.

In the essential oil industry, chemical reactions can be run on a relatively large scale using equipment of 4-5,000 gallons capacity and made of stainless steel or glass-lined construction, depending on corrosion conditions and the nature of the chemical reactants. Large volume production of a half million pounds or more per year of a synthetic aromatic would generally necessitate equipment designed for the efficient chemical processing needs of that product. Hence, the materials of construction, equipment layout, flow of raw materials, control of reaction conditions by cooling or heating, agitation, workup, and isolation of the reaction product for final purification are some of the factors which are brought into equipment and process design.

Most chemical operations are continuous batch procedures where the product moves through the process during various stages of its synthesis. Yet, at all times, all stages of the chemical synthesis may be worked on simultaneously. In some chemical procedures, such as chlorination and nitration, or in distillation, continuous operations can be done more efficiently. Continuous distillation is a very attractive and efficient procedure for purification if the number of components to be separated is minimal and their boiling points are sufficiently separated. The number of distillation columns is one less than the total number of components in the system and requires a complete study of the heat input, boil up rate, number of theoretical plates required for separation, reflux ratio, and the height and diameter of the column required.

Synthetic aromatics and the isolation of the components of essential oils require exacting purification procedures to insure that rigid chemical and physical specifications and, more importantly, organoleptic standards are met. Hence, fractional distillation under vacuum becomes the heart of the synthetic aromatic plant. The industry has taken advantage of the latest in distillation column design to insure maximum separation of components with minimum back pressure to prevent heat buildup in the still pot. Commonly, columns of 20 to 30 theoretical plates are adequate for most aromatics although 60 and even 100 theoretical plate columns are known in the industry. Column designs range from bubble cap columns to packed columns using porcelain interlox saddles, stainless steel

Pall rings, vertical annular rings, sieve trays, mesh packing such as Goodloe or Koch-Sulzer, and some proprietary packing.

Purification of solids by crystallization has included the use of rate controlled refrigeration to insure proper seeding and crystal growth for maximum purity. Programmed centrifuges provide optimum cycles for centrifugation, washing of crystals to remove mother liquor, spin drying, and discharging of the product to the drying area. Solids have been dried in tray dryers with currents of warm air, in rotating vacuum twin V cone dryers, or in fluidized bed driers. Some solids of moderate melting point from 50° to 80°C are purified by vacuum distillation and flaking on a drum dryer.

Autoclaves or pressure vessels are commonly used for catalytic hydrogenations, oxidations, alkylations, and condensation reactions. They range in size from 200-4,000 gallons in capacity and in operating pressures from 50 psi to 2,000 psi depending on the needs of the reaction.

The equipment generally used in the synthetic chemical area of the essential oil industry is multi-purpose since, with minimum modifications, the same equipment may be used for the manufacture of many products. The tonnage of any one product would not justify the use of a large efficient manufacturing unit; hence, a product line must be developed and production schedule set up to maintain maximum utilization of the equipment at minimal cost. The wide diversity of chemical reactions used in the industry reads like an organic chemistry textbook and demands great flexibility in the approach to synthesis, equipment, and processing.

Natural flavoring materials are obtained by extraction of fruits to obtain fruit concentrates. Then the by-product essence is recovered by distillation in order to restore proper flavoring values. Spices may be steam distilled to recover spice oils or extracted with solvent to remove all flavoring values as oleoresins. The oleoresin extracts are distilled to remove solvent which is then recycled, reducing the solvent in the oleorsin to the minimum levels required by the FDA (30ppm). The oleoresin is generally a tenfold or more concentrate of the original spice. Vanilla extract is an aqueous alcoholic extract of a prescribed strength of vanilla beans. The extract and beans are separated in a continuous centrifuge and the beans are returned for a second extract.

The conversion of liquid flavors and perfumes to their solid forms is most widely accomplished by microencapsulation or spray drying. Spray drying produces a solid product from a solution or a slurry almost instantaneously. This is accomplished by reducing the liquid to a fine spray, mixing it with a hot gas, and separating the dried powder from the gas. The gas supplies the heat for the evaporation and carries off the moisture in an elapsed time of five to twenty seconds.

The liquid flavor is emulsified with gum arabic, gelatin, or dextrins at 20% by weight of the carrier in a 35% solution of the carrier. After pasteurization, the emulsion is atomized into the spray drier either through a centrifugal atomizer spinning at 12,000 rpm or through a nozzle under several thousand pounds of pressure. The fog-like mist is rapidly dried to particles ranging in size from 10 to 250 microns by contact with filtered air heated electrically or with gas to 450°F. The finished product is collected at the bottom of the cone drier which ranges in diameter from 8 to 20 feet, is separated from fines in a cyclone collector, and delivered by a conveyer to a packing and weighing area. Five thousand cfm of air heated to 450°F will remove fifteen pounds of water with an exit air temperature of 210°F. Short exposure time and relatively low heat of the solid particle when dried (about 150°F) allow very little flavor deterioration. A centrifugal atomizer gives a more uniform spray and a nozzle tends to yield larger particles.

The need of the industry to manufacture trace components in essential oils which play a significant role in creative perfumery and flavors has been met by the rise of several small companies devoted to a particular technology which lends itself to small scale production of these expensive and valuable compounds. The basic work of isolation and structural elaboration has been carried out by government laboratories and the larger companies which are well staffed with trained people and such equipment as GC, NMR, GC-MS, and liquid chromotography. Patent positions are often obtained based on the synthesis or use of these compounds.

The synthesis of cis-3-hexenol and its derivatives has produced a family of compounds which includes esters, unsaturated aldehydes, acetals, and lactones of unusual power and high cost. The usage levels of these materials is so low that their manufacture can be accomplished in large laboratory glassware. The discovery of widespread occurrence of the pyrazines, a thermal interaction product of sugars and amino acids, has been demonstrated in coffee, chocolate, roasted nuts, meats, and vegetables. A greater number of alkylated pyrazines having other functional groups have been synthesized and patented for use in flavors and perfumes. Aromatics not usually associated with useful odors are drawing on other structural forms such as sulfur and nitrogen compounds, including thiols, sulfides, thiazoles, pyridins, and pyrroles. The flood of information from the literature and the widespread use of advanced analytical instruments have stimulated the organic chemist to synthesize not only these products occurring in nature but many other useful and interesting homologues.