Continuous Steam Distillation of Essential Oils

By G. R. Boucard and R. W. Serth, Texarome Inc., Leakey, Texas

A continuous process for steam distillation of essential oils was described in a previous article.[1] Due to the continued widespread interest in this subject, we'll provide further insight into the details of the process, as well as equipment specifications and cost estimates for distillation plants of various sizes.

The process consists of a totally insulated pneumatic conveying system using superheated steam as a carrier gas and arranged in such a way as to provide a two-stage, counter-current flow of the gas and the solid phase. During the transport, which is made to last 30 seconds, the oil goes into the vapor phase and exits the system with the steam after filtration. Following total condensation of the gas stream, the oil is separated from the water condensate in a gravity separator. The water phase is treated and recycled to the boiler. The dry pulverized stream of spent solids is blown directly into the boiler biomass furnace to generate steam and electric power for the process. All the components and the design characteristics of the system follow the laws of pneumatic conveying, which affords a quick and convenient way of scaling the system up and down.

Theory

The basic concept of our continuous essential oil distillation process evolved from the following considerations, which were based on prior laboratory research by the authors:[2]

Step 1. If one can find a way to put finely ground aromatic material (any essential oil plant tissue) in continuous contact with low pressure (2 psig) superheated steam and maintain the contact for 30 seconds in a well dispersed two-phase system, the oil will vaporize and travel from the plant tissue (solid phase) into the steam environment (gas phase).

Step 2. If one can find a way to perform step 1 twice, or to carry out the contacting in a counter-current fashion, then the plant tissue will be virtually exhausted of its oil content, and a maximum oil yield will be obtained.

Step 3. Having achieved step 1 and step 2, it is now necessary to find a way to end the contacting and to cleanly and totally separate the gas phase (containing the steam and all the oil) from the solid phase (exhausted plant tissue of any essential-oil bearing plant).

Step 4. This conventional step consists of piping the filtered gas phase (oil and steam) into any type of water-cooled or air-cooled condenser to obtain a mixture of oil and water condensate, which then can be separated in a gravity decanter, also known as a Florentine flask.

It sounds simple and it is simple. The concept is illustrated in Figure 1, which shows a complete *stage* encircled with a curved line and a *tray* encircled with a dotted line. Also shown are the *reboiler*, the *condenser* and the *reflux*. In this case, the two phases are the *liquid* and *vapor* phases (l and v). The *feed* point is shown for continuous operation. Finally, the *distillate* and the *bottoms* are shown as the two end products.

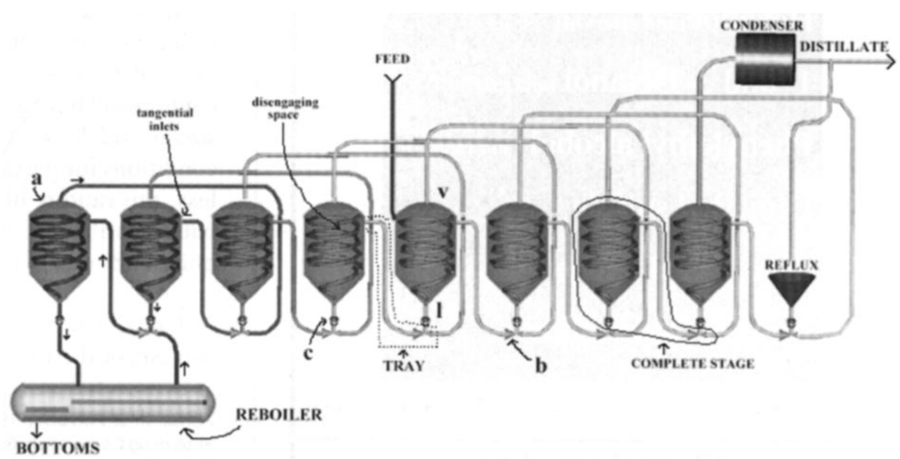

Figure 1. Boucard/Serth concept of a General Mass Transfer Cycle shown on an eight-stage distillation "column" (a = cyclonic vessel, b = venturi device, c = rotary valve)

 0272-2666/98/0002-0001$04.00/0—

We should add that the process described herein is just the "drying" and "steam distillation" application of what we like to call a General Mass Transfer Cycle. As can be seen in Figure 1, the configuration of the general apparatus lends itself to a number of continuous mass transfer tasks, such as multicomponent distillation (vapor/liquid), gas absorption (gas/solid, gas/liquid), leaching (liquid/solid), drying (gas/liquid), extraction (liquid/liquid), partial pressure distillation (gas/liquid and gas/liquid/solid), evaporation (gas/liquid) and even crystallization, desorption and ion exchange.

The distinction of this GMTC concept is that, using cyclonic separators or hydroclones, it accommodates all phase combinations (vapor/liquid, gas/liquid, liquid/liquid, solid/liquid, gas/solid) and is, at least in theory, applicable to most mass transfer tasks. (See **Other Applications for Continuous Distillation**.)

Other Applications for Continuous Distillation

For the benefit of those outside the field of essential oil distillation, it should be mentioned that the continuous distillation process described here, a process that falls under the broad classification of "thermal desorption" processes, is immediately adaptable to stripping away any volatile organic or inorganic compound with a boiling point of up to 1,000°F from any solid matrix suitable for pneumatic conveying.

An Essential Oil Application

The physical laws underlying the process itself obviously fall under the domain of chemical engineering, but the actual apparatus obeys the laws of mechanical engineering, in particular those governing pneumatic conveying. If the distiller is already familiar with pneumatic conveying this will be easy to understand. If not, consider this brief definition:

> A sufficiently strong flow of air (or any dry gas) generated by a compressor or a blower, is able to pick up a certain amount of finely ground solids (or liquid droplets) and blow them through a pipe from point A to point B over long distances. This is generally called pneumatic conveying (Greek *pneuma*, breath).

Our process basically is a pneumatic conveying system that uses superheated steam instead of air as a carrier gas. We found that steam conveys just as well as air, provided it is not allowed to condense. One of the key requirements of this system is that it must be totally and thoroughly insulated. Since steam coming from a boiler is already under considerable pressure, no blower or compressor is needed. In fact, a pressure regulator must be used to bring the steam pressure down to 2 psig, a standard pressure in pneumatic conveying.

Calculations and practice have shown us that a ratio of 1:1 (one pound of steam/hr for one pound of dry [10%] distillation material/hr) provides enough heat to vaporize the oil. It so happens that this amount of superheated steam at 425°F and 1 to 2 psig constitutes enough gas volume to convey the material through the system pneumatically at the standard conveying velocity of approximately 5,000 fpm, given pipes of appropriate size.

It must be noted here that ground-up materials with a moisture content greater than 10% (up to 50%) can be processed just as well, so long as they remain a free-flowing solid with an angle of repose of 60° or less. The only difference is that the wetter material may require a higher ratio of superheated steam, since the higher moisture content will have a tendency to quench (de-superheat) the steam and lower the system's temperature below ideal conditions for partial pressure steam stripping. Nevertheless, the ratio is likely to be lower than in conventional batch distillation, which for purposes of comparison requires ratios of 3:1 for cedar, 12:1 for vetiver and 20:1 for amyris.

Our apparatus, just like any pneumatic conveying system, consists of the following components:

Perfumer & Flavorist (ISSN 0272-266) is published bi-monthly by Allured Publishing Corporation, 362 S. Schmale Road, Carol Stream, IL 60188-2787. Subscriptions: USA and Canada US$125.00 one year; all other countries US$165.00 one year shipped by air. Periodical postage paid at Carol Stream, Illinois and at additional mailing offices. **Postmaster:** Send address changes to ***Perfumer & Flavorist***, 362 S. Schmale Road, Carol Stream, IL 60188-2787, USA.

- A blower (actually a boiler in this case)
- Conveying pipes
- Cyclones
- Rotary valves (airlocks)
- A dust filter

Aside from the special way of running the conveying pipes, there is one more key element that must be added to the conventional pneumatic conveying system to make it distill essential oils. That element is residence time cyclones.

Residence Time Cyclones

Chemists know that all reactions take time to complete, from very short reaction times for explosions to long reaction times for fermentation. But physical separations also take time, from a "flash" drying to a week-long sedimentation. Our continuous distillation process is a "flash drying" or "flash distillation" of sorts. However, laboratory tests conducted under different operating conditions showed that the flash occurs in a steep curve that, at a temperature of 400°F, plateaus after a period of 30 seconds given a particle size of 35 mesh or less (for wood).[2] Distillation materials with the oil on the surface of the plant tissue, such as certain leaves and flowers, may require shorter "flash times." At any rate, a standard pneumatic conveying system, designed for the purpose of conveying only, does not provide sufficient residence time for the flash distillation to take place in most cases, and certainly not in the case of cedarwood, which was our aromatic raw material. For that reason, we had to find a practical way to "make time" inside the system.

Two intensive variables affect the required residence time for flash distillation: temperature and particle size.

Temperatures higher than 450°F will cause charring and partial pyrolysis of the biomass and the decomposition of the essential oil. Temperatures of less than 400°F will significantly lengthen the required residence time for equal yield. Hence, we found 400–425°F to be the ideal operating temperature for flash essential oil distillation.

Particle sizes larger than 35 mesh will drastically increase the required residence time. Particle sizes smaller than 35 mesh will shorten the residence time and increase the yield at equal temperature, but they are very hard to achieve economically with a heavily lignified biomass such as wood. Dry leaves, seeds and roots are better candidates for this system than wood because they pulverize easily.

We found that 30 seconds was a good distillation time in our continuous process facility. But how does one achieve this much residence time in a system where the material travels at 5,000 fpm (83 feet per second), and still make a 100 ton/day plant fit on a 40-foot trailer? We had made the earlier observation that material entering a cyclone spends a significant amount of time within the device. In fact, some material stays spinning around inside the cyclone even after we cut off the feed of in-going material, due to the updraft created by the vortex, and only falls out after we stop the air flow from the blower.

Using a standard high-efficiency cyclone design with

Table I. Cost estimates ($) for fabrication of Texarome-type continuous distillation facilities of three different capacities

Item*	200 tons per day	25 tons per day	6.25 tons per day
Metering bin and hammermill	100,000	25,000	15,000
Boiler and piping	60,000	20,000	7,500
SS pipes/elbows/flanges	25,000	10,000	3,000
SS cyclones plenum	55,000	35,000	17,500
Filters and SS housing	35,000	8,000	3,000
Rotary valves	60,000	25,000	10,000
Cooling	40,000**	10,000***	3,000***
SS condenser surface	5,000	2,000	500
SS oil receiver	20,000	2,000	500
Electric system	50,000	10,000	4,000
Total	450,000	147,000	64,000

* Engineering specifications available from authors
** *Pump and tower/pond*
*** Air cooled

plexiglass windows, we studied the flow pattern and residence time of various particle sizes in a cyclone. By direct measurement, we established that biomass solids of 35 mesh and under with a moisure content of 10% and a density of 15–20 lb/ft^3 entering a standard high efficiency cyclone at the standard conveying velocity of 5,000 fpm will reside in the cylcone for at least 0.5 second for each linear foot of cyclone height.

Hence, one can see that, whereas a linear foot of standard pneumatic conveying pipe in a system of any size provides only 0.072 seconds of residence time, a small 6 ft cyclonic vessel provides 3 seconds, and a larger 12 foot cyclone provides 6 seconds. Obviously, the larger the system, the smaller the number of residence time cyclones required. Or, put in business terms, it pays to go big. Our 1,000 lb/hr apparatus has a total of 9 cyclones 6 feet tall. Larger systems may require a total of only 4 or 5 cyclones. By comparison, *it would take half a mile of pipe to achieve* a 30-second residence time.

Cost Considerations

The apparatus we use consists simply of a series of pipes connected to small cyclonic vessels and three rotary valves. Pipes and empty vessels are inexpensive. Rotary valves are relatively expensive unless they are built by a local machinist. The novelty of the design is a rather convoluted way of running the pipes and interconnecting the vessels in order to achieve a two-stage, counter-current flow of the phases (gas/solid), with subsequent separation of the phases, while maintaining a co-current flow for solids transport. Furthermore, the residence time cyclones are used to provide the contacting time needed for the mass transfer to take place.

Our apparatus faithfully incorporates the three main requirements of the multistage mass transfer process (counter-current flow, phase contacting and phase separation) and is a serious piece of chemical processing hardware. However, any small welding shop can fabricate and assemble such an apparatus using carbon steel or stainless steel, and deliver an essential oil distillation plant of considerable throughput in a very short time for a fraction of the cost of a conventional batch plant with the same capacity. A number of practical considerations will determine the design parameters of any particular continuous distillation facility. We won't mention them all here, except to say that the two most important are the desired capacity of the facility (expressed in tons/day of raw material) and the bulk density and moisture content of the raw material.

Since the continuous facility runs around the clock in a fully automated mode, let's take a nominal throughput of 200 tons/day divided by 24 hours, which is 8.3 tons/hour or 16,666 lbs/hour. At a yield of 2.5% , such a plant would produce 417 lbs of cedarwood oil per hour, 10,000 lbs/day, or 22.7 drums/day (55 gal drums/440 lbs net). Table I gives an idea of what it *should* cost to have a small local engineering construction firm handle the turnkey fabrication of a cedarwood oil continuous distillation facility of several

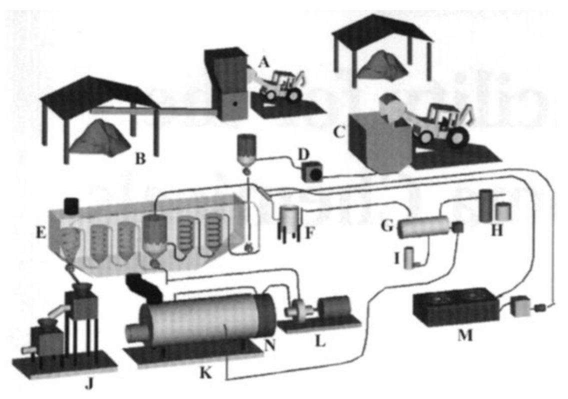

Figure 2. Layout of a continuous distillation facility

A = wood chipper
B = chip storage
C = metering bin
D = hammermill and cyclone
E = continuous distillation unit
F = oil separator
G = condensate tank and boiler feed pump
H = soft water make up
I = chemical feeder
J = furnace
K = boiler
L = TG set
M = air cooling and pump
N = steam superheater

different capacities. Figure 2 shows a flow sheet for a complete facility of any capacity.

Typically, doubling the raw cost of material, labor and shop overhead, as listed in the table, should constitute a fair turnkey price, either for the whole facility or the components. However, this may not include the start-up and "debugging," since the builders are not likely to be familiar with this particular technology, or even with essential oil distillation in general. Thus, for our example plant processing 200 tons/day of cedarwood, the total turnkey cost would be about $830,000.

Finally, it is customary to expect to pay a premium over and above the fabrication price for proprietary technology. This premium usually is factored into the total project in the form of up-front cash, royalties, licensing fees, stock ownership or other form of payment for the transfer of the technology from the developer to the end user.

References

Address correspondence to G. R. Boucard, Texarome Inc., P.O. Box 157, Leakey, TX 78873 USA.

1. GR Boucard and RW Serth, *Perfum Flavor* **16**(2) 1-8 (1991)
2. RW Serth, GR Boucard and BS Ainsworth, *Continuous Partial Pressure Distillation of Essential Oil-bearing Materials: Part I. Bench-scale Tests*, College of Engineering Technical Report, Kingsville, Texas: Texas A&M University (1984)