Monographs for the Million

Hydro-Distillation of Oils From Aromatic Herbs

By E. F. K. (Tim) Denny, The Bridestowe Estate,
Lilydale, Tasmania, Australia

Steam distillation for extracting essential oils from plant materials is a very simple operation. In fact, it is so simple that, for a long time, no one thought the way that the steam liberates the oil was worth investigating. So, until recently, this was very poorly understood and distillers were unaware of much inefficiency and waste built into stills and their operating procedures.

Because saturated equilibrium between a liquid surface and the overlying vapor space occurs almost instantly, it had always been assumed that oil-bearing herb merely had to be enveloped in an "atmosphere" of steam for any exposed oil to vaporize automatically in seeking to saturate the steam in the vapor space. Some of the observed evidence seemed to support this belief and it was generally accepted.
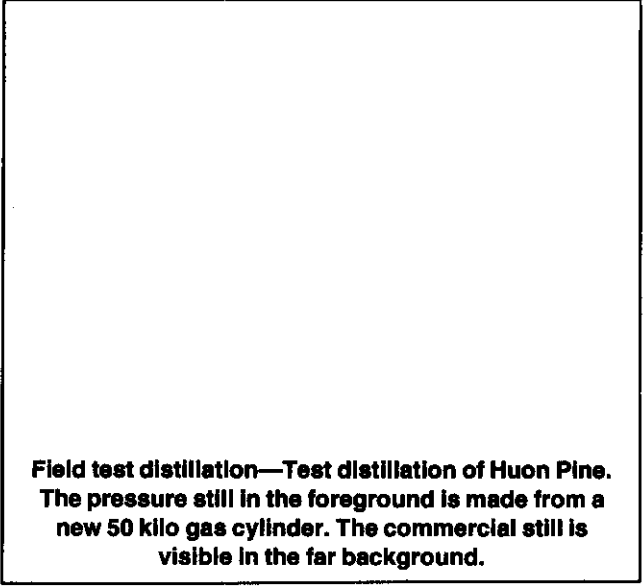
Investigation of the actual method by which the steam gathers up the oil, reveals that the factors involved are very different from what was previously assumed. Latent heats, and not the relative vapor pressures of steam and oil, are the dominant consideration. Understanding this, we now can improve our operating practices, and design our equipment with a scientific precision that previously was impossible.

Basic principles

As I said, this steam distillation is a very simple operation. Oil bearing herb is packed into large vat-like stills. Steam is then passed through the charge of plant material, and this causes the oil to vaporize. When the mixture of steam and oil vapor emerges from the still, it is caught and passed through a condenser. In the liquid phase,

Field test distillation—A simple still made from clean oil drums in use for determining the parameters of basil and lemongrass.

Field test distillation—Test distillation of Huon Pine. The pressure still in the foreground is made from a new 50 kilo gas cylinder. The commercial still is visible in the far background.

the oil separates spontaneously from the water.

In effect, we are making the oil leave the surface of the herb by boiling it in the presence of water and converting it into vapor. If we are to understand how this is done, we must remember two simple principles from our early school days. First, to convert any liquid into a vapor we have *to apply energy in the form of heat.* This is called the latent heat of vaporization. Conversely, if any vapor is to be converted back into liquid, its energy must be reduced and the latent heat surrendered and given up.

Second, all compounds with determinable boiling points emit fast-moving vapor molecules from their surfaces. These vapors exert a pressure on their surroundings which increases with rising temperature. If heat is applied to a liquid *until its temperature attains the point where its* vapor pressure becomes equal to the surrounding pressure, the temperature will rise no further. If heat continues to be applied, the liquid will take up this energy as latent heat and will vaporize at *the appropriate rate that liquid is then said to boil* and the temperature at which this occurs is the boiling point under the prevailing pressure.

If two mutually insoluble compounds, like oil and water, are present in the same vapor space, each will exert the *pressure appropriate to the* concentration of its own molecules, as though the other were not there. The total pressure exerted by the mixed vapor then becomes the sum of the partial pressures exerted by each constituent present. *It follows that a liquid mixture will boil* when its temperature is raised to the point where the combined vapor pressure of its components becomes equal to the surrounding pressure.

The basic principle of this hydro-distillation, therefore, consists in bringing the two mutually insoluble liquids into contact at a temperature very close to the boiling point of the more volatile of the two, the water. Then the addition of a very small amount of vapor pressure from the oil will make the combined pressure equal to the surrounding pressure. The mixture then will boil and the oil will vaporize into the passing steam as fast as its requirement of latent heat can be applied.

It is the rate of application of this latent heat that governs the speed and efficiency of the distillation. It depends on two things. The first is the magnitude of the temperature gradient leading heat from the general vapor space to the oil's point of evaporation on the leaf. The second is the effective size of the heat transfer area onto which steam particles can usefully condense and surrender their latent heat to vaporize the oil.

The temperature gradient

The oil cannot vaporize unless it receives latent heat, and inside a field still this heat can come only from condensing steam. Therefore, the temperature of the steam in the general vapor space must be higher than that at which oil is boiling in the presence of water on the surface of the herb. Otherwise there could be no temperature gradient to lead heat from the steam to the vaporizing oil. It follows that the oil's concentration and the share of the total pressure that it exerts in the general vapor, must be significantly *less than the saturation level at which it boils* with water on the leaf. This ensures that the steam's temperature in the general distillate vapor cannot fall below that at which it exerts the total ambient pressure in the still, less only a *meager contribution from the unsaturated oil.* Obviously, this temperature must be higher than that at the point of evaporation on the leaf surface where, by definition, saturation conditions must prevail and the oil exerts its full potential vapor pressure. *This gives us the temperature gradient* we require.

For every oil and set of conditions there is a balance point for the maximum attainable oil concentration in the general vapor space. Any *further enrichment of that vapor would lower its* temperature and reduce the gradient. This would retard the rate that heat is delivered to the oil and reduce its rate of evaporation until the normal balance situation was restored.

This automatic balance operates throughout the period of flow. But it only shows the ultimate limit to the oil's proportion of the distillate at the start of very efficient distillations. With oils of average volatility, it is not practical to make *changes that will greatly affect the magnitude of* the temperature gradient. This makes it important to promote the best natural gradient we can, by ensuring that all the other conditions for heat transference are fully favorable.
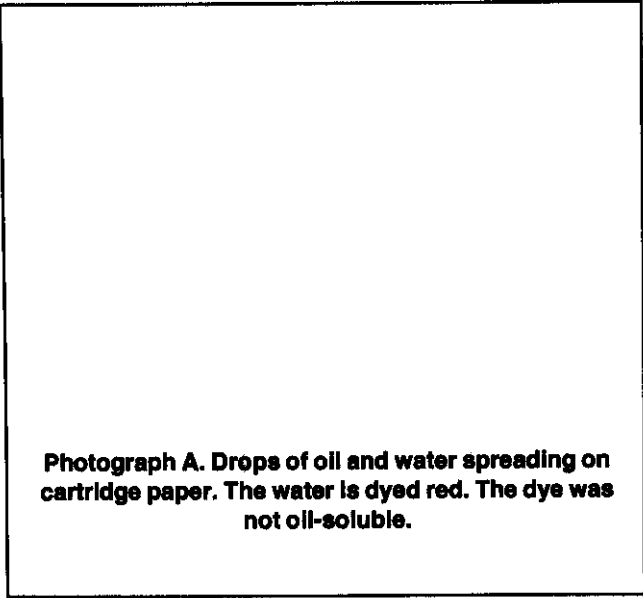
On the other hand, with the more difficult high boiling oils like Huon Pine, very little oil is returned by distillation under atmospheric pressure. This is because the temperature gradient is then very small indeed. Frequently it is as little as 0.05°C. If the oil does not decompose at the higher temperature involved, it is both acceptable and necessary to use considerable gauge pressure in the still. It can be shown that this may increase the magnitude of the temperature gradient by as much as one quarter of a degree.

Of course, this very modest gain is insignificant for oils of average volatility. But it amounts to a very large factor of increase in the gradient to these less volatile oils and in the rate that they receive heat and boil away. The distillate then is enriched by the same factor and the ratio of oil to water in the condensate is improved by a far larger proportion than can be explained by the increased density of the steam. Later we shall discuss the effect of operating pressure on the density of the steam.

Heat transfer area

Now let us consider a simple distillation like that of mint, where the oil is in little blisters on the surface of the leaf. At the start of the process, steam will condense and moisten all the plant surfaces in surrendering its latent heat to raise the temperature. The blisters burst and form small patches of surface oil. Around each of these there will be an interface where oil and water are in contact (see photographs A, B and C). When the temperature reaches approximately 98°C, the additive properties of the two vapor pressures will cause the mixture of oil and water at this interface to boil. Oil and steam vapor must now rise from the entire perimeter of the oil patch so that every homogeneous oil area now will be covered by saturated vapors in equilibrium with its surface.
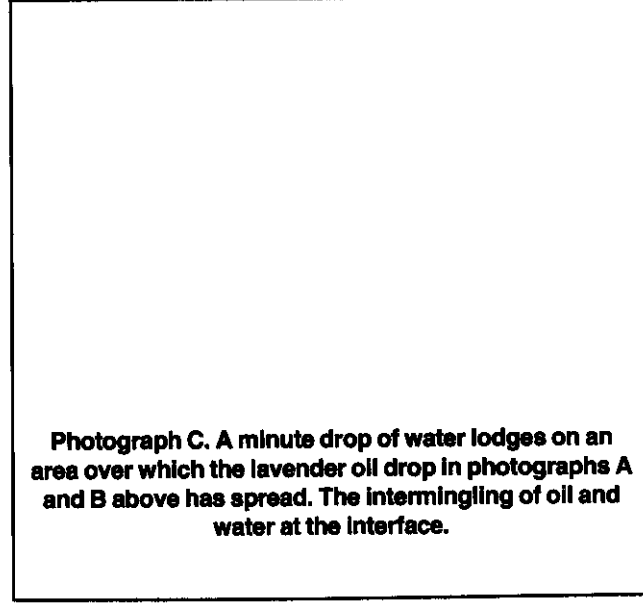
Steam cannot condense into the homogeneous oil surfaces, but its particles will readily condense into water surfaces in immediate contact with the oil's perimeter. In doing this, they surrender the latent heat required to vaporize the oil. The oil can boil away only from the circumference of each surface patch, and the time required to exhaust the oil is that taken to reduce these radii to nil. The rate that oil will vaporize obviously depends on the speed with which the steam flow delivers fresh steam to the interface to replace that which has already given up its latent heat to the oil. The speed of oil distillation is, therefore, related to the speed of the steam inside the still. The actual relation is always direct but it is not always a simple one.
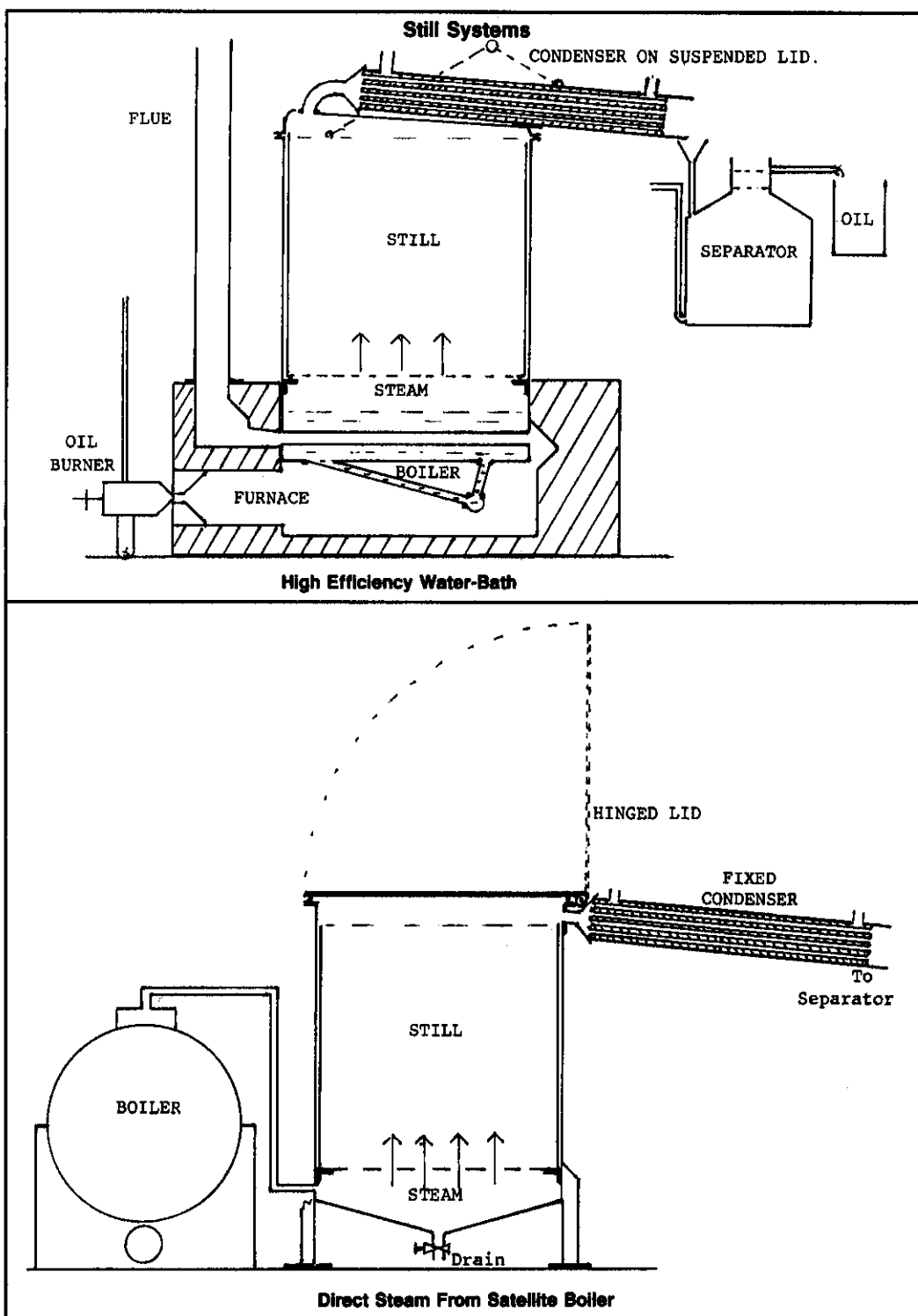
Photograph A. Drops of oil and water spreading on cartridge paper. The water is dyed red. The dye was not oil-soluble.

Photograph B. The spreading areas of oil and water meet and the two liquids intermingle.

Photograph C. A minute drop of water lodges on an area over which the lavender oil drop in photographs A and B above has spread. The intermingling of oil and water at the interface.

Still Systems

CONDENSER ON SUSPENDED LID.

FLUE

STILL

SEPARATOR

OIL

STEAM

OIL
BURNER

BOILER

FURNACE

High Efficiency Water-Bath

HINGED LID

FIXED
CONDENSER

STILL

To
Separator

BOILER

STEAM

Drain

Direct Steam From Satellite Boiler

The oil's rate of evaporation also is directly proportional to the size of the water area in immediate contact with surface oil into which the steam particles can usefully condense. Clearly, this heat transferring area will be increased greatly if the oil and water intermingle at the interface instead of forming only a thin line of contact around the oil's perimeter. It is found that these two liquids do, in fact, form a heterogeneous mixture where they meet on absorptive surfaces. It is, therefore important to ensure that the herb surfaces are at least slightly absorptive before the material is loaded in the still. In many cases, this will call for the herb to be partially dried in the sun until the leaves are nearly brittle.

However, there is little point in going to the

trouble of drying the crop if there is not enough water present on the leaf throughout the distillation to maintain contact with the receding perimeters of oil. The initial water which condensed to heat the charge, does not follow up the shrinking patch of surface oil, and all the water that condenses to surrender the necessary latent heat, will replace no more than one-fifth of the volume of the oil that it removes. It is essential that moist steam deliver cloud particles which lodge on the herb surfaces and bridge this widening gap between the original surface water and the receding perimeters of oil. This is the only way that the heterogeneous mixture at the interface can be maintained and the heat transfer area kept at the maximum attainable at all stages of the distillation.

But if steam is generated under high pressure in a satellite boiler, it will expand and dry out its moisture content as it enters any atmospheric pressure still. It may no longer carry enough water particles to sustain the contact between oil and water at the interface. Then all but the early part of the distillation will take far more steam and time than necessary. For the majority of atmospheric pressure distillations, the steam should be generated under relatively low gauge pressure.

Conversely, when the steam is generated in the same vessel as the load of herb, it can carry too many cloud particles and cause flooding of the herb surfaces. No residual absorptive capacity is left to help the oil and water to mix together at the interface and this creates a condition tending to that of inadequately wilted herb. These water bath systems are among the best when they are carefully designed. But there must be an adequate interval between the turbulent surface of the boiling water and the bottom of the plant charge. This space makes room for an efficient scheme of baffles to protect the charge from water splashes and to trap any excess moisture in the steam.

In short, the moisture content of the steam must be adapted to the absorptive property of the herb. It must keep the surface moist, but it must not overload the herb's absorptive capacity by flooding it.

Effect of gauge pressure in the still

The effective size of the heat transfer area will be increased if we can keep a greater mass of steam in contact with it during the distillation. We can do this by operating with gauge pressure in the still. The mass of steam in contact with a given surface area is proportional to the cube root of the steam's density squared. As an example of

what happens, let us raise the pressure in the still from one atmosphere absolute to two. The cube root of the steam's density squared increases by a factor close to 1.5. Therefore, one-and-a-half times as much steam is in contact with the heat transfer area and the oil receives its latent heat one-and-a-half times as quickly without any increase in the rate of flow. Then only two-thirds as much steam has to pass to recover a given yield of oil.

Obviously, the potential savings of fuel and time are considerable, and there may be more subtle benefits in certain cases where the use of pressure can reduce the amount of oil lost in the discarded water. However, pressure stills are expensive and relatively limited in size. This restricts their use to special oils and situations. For ordinary crops the big box stills, loaded automatically by the harvester with up to six tonnes of herb per charge, will process more material at a lower total cost.

Design factors

For each plant material there are two parameters which define its behavior in the still. The first is the time required for a given flow of steam to exhaust the oil from a single piece of herb at the bottom of the charge. The second is virtually the increment to this time because of each centimeter of charge height. Both these parameters have numerical values with definite relations to the speed of the steam and the oil content of the herb. They are very easily determined from small test distillations of the plant material, and for most herbs, these can be carried out using very simple and inexpensive equipment made from clean oil drums. When the two parameters have been determined for the conditions in which it may be proposed to distill commercial quantities of the tested herb. Then the distilling times and the daily handling capacity of any size still, steamed at any rate of flow, can be calculated very accurately before turning to the drawing board.

In ordinary design work, the height of the still is chosen to make efficient usage of the steam. The diameter will be designed to give the operator a convenient unit to handle. Then the boiler is selected to provide a distillate flow that will allow the required number of charges to be processed in the day. Then, as in so many other fields, all that is required is that the user should, as the last resort, read the instructions.