The extraction of clove oil from cloves by steam distillation

Theory

Boiling of a liquid in a vessel open to the atmosphere occurs when the total vapour pressure reaches atmospheric pressure. When two or more immiscible liquids are heated, the total vapour pressure over these liquids is equal to the sum of the individual vapour pressures. This allows substances to be distilled that, if heated on their own to higher temperatures, might suffer partial decomposition or charring. In this experiment the initial products of the steam distillation will be clove oil and water.

Procedure

NB: Wear your safety glasses.

Set up the Quickfit apparatus as in Fig. 1. **Make sure your set-up has a safety opening to the atmosphere as in the diagram.**

Note the mass of the cloves, and place them in the pear-shaped flask. Cover with a little warm water (about 5 cm^3).

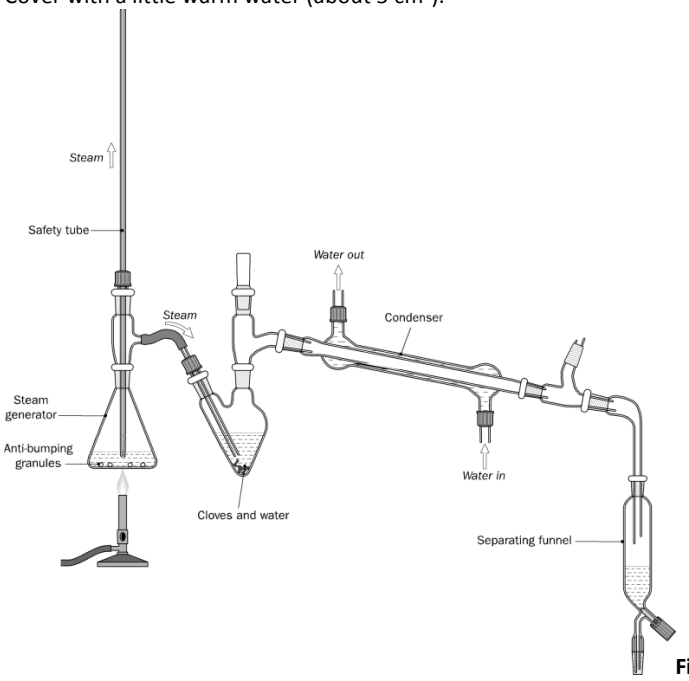


Fig.1

Place an adequate supply of water in the steam generator, connect it to the rest of the apparatus and set it to boil. If Quickfit apparatus is used for steam generation make sure that you use anti-bumping granules in the steam generator.

If the level of the boiling water in the steam generator falls too low, the system will not work smoothly. Remove the heat, carefully loosen the safety valve, and top up the steam generator with **hot** water. Reconnect everything and resume heating.

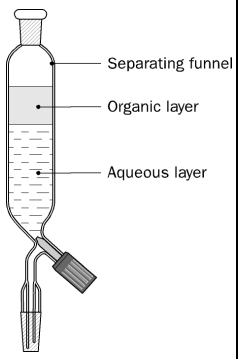
Collect the distillate. It should have a pale milky appearance. Using the dropping funnel as a receiver at this point will facilitate the next stage of the separation.

After 20 to 30 minutes disconnect the steam generator to avoid the possibility of suck-back problems and turn off the heat under it. You will have probably collected between 40 and 50 cm^3 of distillate. Note the smell of the distillate.

Separation of clove oil from water

The procedure is as follows:

Disconnect the dropping funnel from the rest of the apparatus. Add about 8 cm^3 of cyclohexane to the distillate in the dropping funnel. Stopper the dropping funnel and shake the mixture. Release any pressure build-up carefully after each shake by inverting the dropping funnel while holding the stopper and slowly opening and shutting the tap.



Run the lower aqueous layer off. Collect the top layer - this contains the clove oil and the cyclohexane.

Dry the organic layer by shaking with anhydrous sodium sulfate in a conical flask. If possible, allow to stand overnight, before removing the solid by filtration or decanting.

Separate the more volatile cyclohexane from the clove oil by placing the mixture in a small beaker (whose mass is known) on a water-bath in a fume cupboard. The cyclohexane evaporates, leaving the clove oil behind. (Note that if the cyclohexane is to be distilled off it will boil at 81 ^{0}C.) Prepare the students for a very low yield of clove oil.

Note the smell of the clove oil. Do not allow the clove oil to come in contact with your skin.

student questions

Explain why the clove oil could not be distilled directly from the cloves.

Clove oil contains component molecules whose boiling points are quite high. Heating them to their boiling point would be destructive.

Explain the principles by which steam distillation works.

Some organic compounds are immiscible with water. Usually these compounds have a low vapour pressure. After mixing them with water, however, the mixture will distil when the sum of the two vapour pressures reaches atmospheric pressure. It follows, then, that this must happen below the boiling point of water. This process is known as steam distillation.

Suggest a reason why the clove oil is much more soluble in non-polar solvents (than in water).

The component molecules found in clove oil are often relatively non-polar and will therefore be more soluble in the non-polar solvent than in water.