Purification of a substance with water vapour distillation

Aims of the experiment

- Getting to know water vapour distillation as a separation method.
- Understanding water as a carrier for water vapour distillation.
- Understanding the temperature dependence of substances volatile in water vapour.
- Observing the formation of a two-phase system more closely.

Principles

The process of water vapour distillation takes advantage of the fact that a non-volatile component also dissolves slightly in water vapour and can be carried off during distillation.

In this experiment, a citrus fruit peel is added to the water solvent for the distillation. The mixture is then heated until it boils. Water vapour forms, which acts as a carrier and releases the fragrances and flavours from the peel. However, these essential oils are only slightly volatile in water vapour and thus only constitute a small percentage of the vapour. Most of the gas phase consists of water. Therefore, in order to retain a large amount of the fragrances and flavours, a large quantity of water must be distilled. Due to the large amount of water, the temperature profile needs not be recorded, since the boiling temperature is approx. 100 °C.

The water vapour cools down in the distillation bridge and two phases are formed in the collection flask. The water and the oil separate from each other, since the solubility of the citrus peel oil in water is dependent on the temperature. Here, the oil is hydrophobic, i.e. „water-hating“, since it cannot interact well with water. In order to achieve as small a contact area with the water as possible, small oil droplets form on the surface of the water. Furthermore, the oil floats on top of the water, since it has a lower density than water.

The advantage of this separation method is that the extraction of essential oils and the separation occur simultaneously. During distillation, only one component that is volatile in water vapour is separated in water and not two different substances. Water vapour distillation is also very substance-friendly and selective, since there are only very few substances that are volatile in water.

Fig. 1: Experiment apparatus for water vapour distillation.

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Risk assessment

Even though citrus peel oil is a natural product, it is suspected that some of its components are carcinogenic. For this reason, the here prepared oil should not be used.

Equipment and chemicals

2 Adhesive magnetic board, 500 mm.....666 4659
1 Holder, magnetic, size 2, 11...14 mm.....666 4662
2 Holder, magnetic, size 3, 18...22 mm.....666 4663
1 Heating mantle, 250 ml 666 6522
1 Distillation bridge after Claisen.....665 338
1 Round-bottom flask, 100 ml, ST 19/26.....664 300
1 Round-bottom flask, 250 ml, ST 19/26.....664 301
2 Joint clip, plastic, ST 19/26, set of 10.....665 391ET10
1 Panel frame C50, two-level, for CPS.....666 425
1 Chemical thermometer, -10...+220 °C/1 K 666 161
1 Laboratory stand 16 cm x 13 cm 300 76
2 PVC tubing, 7 mm diam., 1 m 604 501
4 Hose clamp 8...12 mm 604 460
1 Silicone gaskets, GL 18/8, set of 10.....667 295
1 Boiling stones.....661 091
1 Stopcock grease, 60 g.....661 082

Additionally required: .....
one lemon or orange (unwaxed as far as possible)

Set-up and preparation of the experiment

Preparations for the distillation

Making up the base mix: The citrus fruit peel is added to approx. 150 ml of water in the 250-ml flask without the white part. Then some boiling stones are added to the flask.

Set-up of equipment

For the water vapour distillation, a distillation bridge is fixed to the magnetic board using three clamps as shown in Figure 1. Two of these clamps are fixed to the bridge itself and the last one to the side of the distillation flask. The water hoses are connected to the distillation bridge in reverse flow. Then the chemical thermometer is inserted in the GL screw connector. For this, the GL cap is unscrewed, the thermometer inserted and the cap screwed back on. This creates a tight connection. The heating mantle with laboratory stand is assembled and connected. Then the receiver flask and the collection flask are greased and clamped to the apparatus using a joint clip. Finally, the heating mantle is positioned under the distillation flask.

Note: The thermometer should be effectively sealed using a gasket. If the temperature profile of the distillation is also to be recorded, a temperature sensor can be used instead of the thermometer. Furthermore, the water connections should be tested before starting the distillation.

Performing the experiment

1. The heating mantle is switched on and the distillation starts.

Note: If a temperature sensor is used instead of a thermometer, the measurement should be started when the heating mantle is switched on.

2. The distillation is carried out until a sufficient amount of water is distilled with essential oil and small oil droplets are visible.

Observation

By heating, some of the contents of the citrus peel are released and the solution turns yellow or orange depending on the colour of the peel. When the solution is heated further, the liquid phase passes into the gas phase. In the process, the highly volatile fragrances and flavours of the citrus peel are carried off, since they are soluble in the hot water vapour. When the hot vapour meets the cold glass of the flask, it condenses and small drops form.

Through further heating, more and more vapour forms, which condenses on the glass of the apparatus and flows back into the flask as drops. The hot vapour heats up the glass and thus rises in the apparatus.

After a while, the vapour reaches the thermometer and heats it up. The temperature of the thermometer rises to boiling temperature. The hot vapour is then cooled abruptly in the distillation bridge and condenses. The condensation flows to the collection flask and is collected there.

The water solubility of the citrus peel oil depends on the temperature. This means that the water and the citrus peel oil separate from each other in the distillation unit. Two phases are thus created in the cooled distillate. The fragrances and flavours of the citrus peel oil also have a characteristic odour, which can also be detected in the collection flask.

Result

Water vapour distillation shows how essential oils are extracted from solid components, e.g. citrus peels. It can also be observed that the water solubility of essential oils is dependent on temperature.

The fragrances and flavours are carried off by the water vapour. If the water vapour cools in the distillation bridge, two phases are created in the collection flask. The lower is the water phase and the citrus peel oil is in the upper phase. The essential oils of the citrus peel can thus be easily separated from the water solvent. However, the upper phase here is often very small, since only a small amount of the oil is volatile in water vapour and mainly water is distilled. The fragrances and flavours of the citrus peel can also be detected by means of an odour test.

Cleaning and disposal

The solutions created can be poured down the drain with a large amount of water and the remaining citrus peel disposed of in the general waste.