Extraction with Volatile Solvent

Solvent Extraction Method

↳ used to extract EOs from plant that cannot withstand the high heat used in steam distillation

→ Very delicate aromatics → Jasmine, Linden Blossoms etc. can not survive the process of distillation

→ To capture their magical aromas, a process of solvent extraction is used.

→ For some plant the intense heat damages them, so, that the essential oils are unusable therefore, they must be extracted by other means.

→ Solvent extraction needs complicated & expensive apparatus and team of well trained workers.

→ Mistake in operation can be costly, unavoidable loss of solvent, of which large quantities are used during the process. → important factor in calculating the flower oil.

* Extraction with volatile solvents

↳ first used by Robiquet in 1835 for flowers.
Solvent used ~~was~~ Petroleum ether (common & suitable solvent)

Principle of extraction →

↳ Charging of fresh flower into specially constru-cted extractors

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Extracted systematically at room temperature with a carefully purified solvent.

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~~Et~~ solvent penetrates the flowers, dissolves the natural flower perfume along with some waxes & albuminous & coloring matter.

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Subsequently solution is pumped into an evaporator and concentrated at a low temp.

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Concentrated flower oil is obtained after complete evaporation of solvent.

→ Temperature applied during entire process is kept at a minimum.

→ The extracted oil represent the natural perfume more truly as originally present in flowers

* Extracted flower oils → mostly more or less dark in color.

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because they contain more of natural plant pigments which are non-volatile.

(Steam distilled oil are mostly light in color & usually soluble in dilute alcohol)

Extracted oils require 95% alcohol for complete solution.

Advantage of the product of extraction process

→ Product has true natural odour.

Certain flowers eg. Jasmine, Tuberose, Jonquil, hyacinth, acacia, mimosa etc. do not yield their EOs on steam distillation & therefore are extracted with solvents.

Extraction With Volatile Solvent

first used by Robiquet (1835) for flowers.

Solvent → Petroleum ether → Most suitable solvent

Principle →

Charging of fresh flowers into specially constructed extractor

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Extracted systematically at room temperature, with a carefully purified solvent (eg petroleum ether).

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Solvent penetrates the flowers and dissolves the natural flower perfume together with some waxes, albuminous & coloring matter

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The soln. is subsequently pumped into an evaporator and concentrated at a low temperature.

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After solvent is completely driven off (remove) in vacuo, the concentrated flower oil is obtained

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Thus, temp. applied for entire process is kept minimum;

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& live steam as in the case of distillation, does not exert its action upon the delicate constituents of the flower oils.

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Compared with distilled oil, the extracted flower oils are more truly represent the natural perfume as originally present in the flowers.

→ Running expenses are quite high.
Solvent extraction needs complicated & expensive apparatus & crew of well trained workers.
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A mistake in operation can be costly, unavoidable loss of solvent, of which large quantities are employed during the process, is an imp. factor in the prize calculation of natural flower oils.

→ All extracted flower oils are mostly more or less dark in color.
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becoz they contain much of natural plant pigments which are non-volatile.
(Steam dist. oils are mostly light in color)
Mostly soluble in dilute alcohol., while extracted oils are soluble in 95% alcohol for complete soln.

Adv. → Their true-to-nature odour., In addition Certain types of flowers eg. jasmine, tuberose, Tonquil, hyacinth, acacia, mimosa & violet, do not yield volatile oil on steam dist. ∴ must be extracted with solvent.

Selection of Solvent →

Quality of solvent is an imp. factors.

Ideal solvent should possess several properties :-

(1) Completely & quickly dissolve all the odoriferous principles of the flowers, & as little as possible of such inert matter as waxes, pigments, albuminous compds etc. (solvent should be selective)

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(2) Should have low B.P to permit it being easily removed (distilled off), without resorting to higher temp°; yet the B.P should not be too low, as this would involved loss of large amt of solvent by evaporation

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(3) Solvent should not dissolve H_2O since H_2O present in the flowers would dissolve & accumulate in the solvent

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(4) Solvent must be chemically inert, i.e does not react with the constituent of the flower oil.

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(5) Solvent should have uniform B.P, i.e when evaporated should not yield any residue; Highest trace of High B.P compds upon evaporation of solvent, would accumulate & remain in the FOs & completely spoil its odour.

↓ Flower oil

(6) Solvent should be of low priced & if possible non flamable.

Petroleum ether, Benzen (Benzol), alcohol.

① Petroleum ether (PE)

→ Fractions, Boiling range 30°-70°, consist of saturated paraffins viz. mainly pentane & hexane because of complete inertness & complete volatility, suited for ED, chemical extraction.

have

Selective power of dissolving, yield prdt having little wax, albuminous & coloring matter but relatively more of the odoriferous compd.

PE must be free from sulphur & nitrogenous compds

② Benzene (Benzol)

It is a coal-tar product, made by treating & purifying coal-tar naphtha with H_2SO_4 & subsequently with NaOH.

fraction below 130° contain lower benzene hydrocarbon composed mainly of benzene, toluene, & other homologous compds

Industrial benzol. "benzol" often contains pyridine, carbon disulfide (CS_2), & thiophene which must be removed by treatment with conc. H_2SO_4 + H_2O + caustic soda soln

Further fractionation eliminates most of High B.P homologous compds. but complete purification is obtained only by repeated crystallization

Thus pure benzene, m.p 5.5°C, boiling is obtained, the Higher homologous remaining liquid & being separated by vacuum filteration or other methods

Alcohol →

Cannot be used for extraction of fresh flower becoz it dissolves H_2O contained in them & becomes increasingly more dilute.

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With some flowers (tuberose) alcohol develops a most disagreeable odor; from other (jaismine for eg.) it extracts dark, solid masses, which possess an odour similar to molasses;

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High-proof alcohol – in some instances dil. alcohol – is widely employed, however, for the extraction of dried plant materials, leaves, barks, roots & especially gums from which alcoholic tinctures are obtained, which find wide application in pharmacy & perfumery

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Concentrates of these tinctures (after removing alcohol in the vaccum still) forms oleoresins & resinoids, which are usually viscous, often almost solid, masses of dark color, representing conc. odoriferous principles + alcohol-soluble resins, coloring matter etc, contained in the original plant materials.

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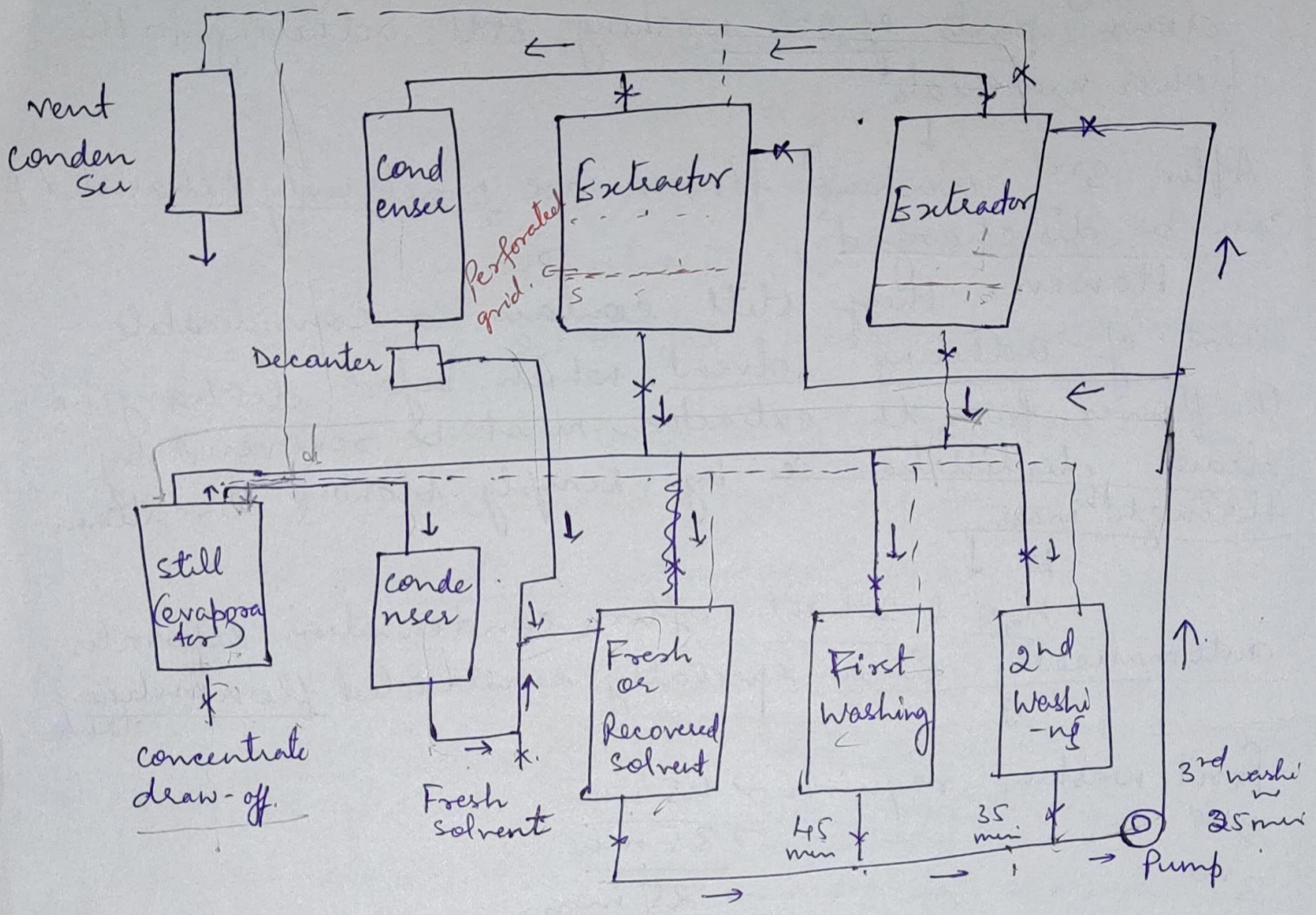
Resinoids of olibanum (Lobhan), myrrh (Ar. आवला), opopanax, benzoin (गुग्गुल) etc, are widely employed in perfumery & oleoresins, of vanilla, ginger root, capsicum, celery seed etc, in the flavoring of all kinds of food product & ~~Bev~~ Beverages.

Schematic diagram of an extraction system



Extractor & still → copper bcoz this metal retains its value after scrapping the apperatus & pliable copper can be hammered & repairs easily.

Extraction carried out methodically by successive washing when by each batch of flowers is treated 3 times with solvent.

3rd washing used as 2nd washing for the next flower batch, then as first washing & is finally pumped into the evaporator for concentration.

Solvent distilled off is used as fresh solvent for a 3rd washing which serves again as 2nd of the next flower batch etc. A 4th extraction yield in most cases at best only small quantity of waxes & other inert subs.

The actual flower oil is contained in I^st & 2nd washings. while the 3rd one serves merely to wash down parts of 2nd washing still adhering to the flower materials

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After 3rd washing flowers are practically exhausted & can be discharged.

However they still contain a considerable amt. of adhering solvent, which before discharging the flower from the extractor, must be recovered by steam distillation ie by simply blowing live steam through the mass

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H_2O & solvent after condensation separates automatically in a specially constructed florentine tank

first washing requires → 45 min
2nd ... → 35 min
3rd ... → 25 min

For drawing off soln. & pumping in the next washing 5 – 10 min must be allowed for each operation.

Conc. of soln → I^st the most conc. washing is filted off by fine screen & pumped into the so-called evaporator in which greater part of solvent is driven off. ie indirect heating by indirect steam blown in to a steam jacket beneath the still.