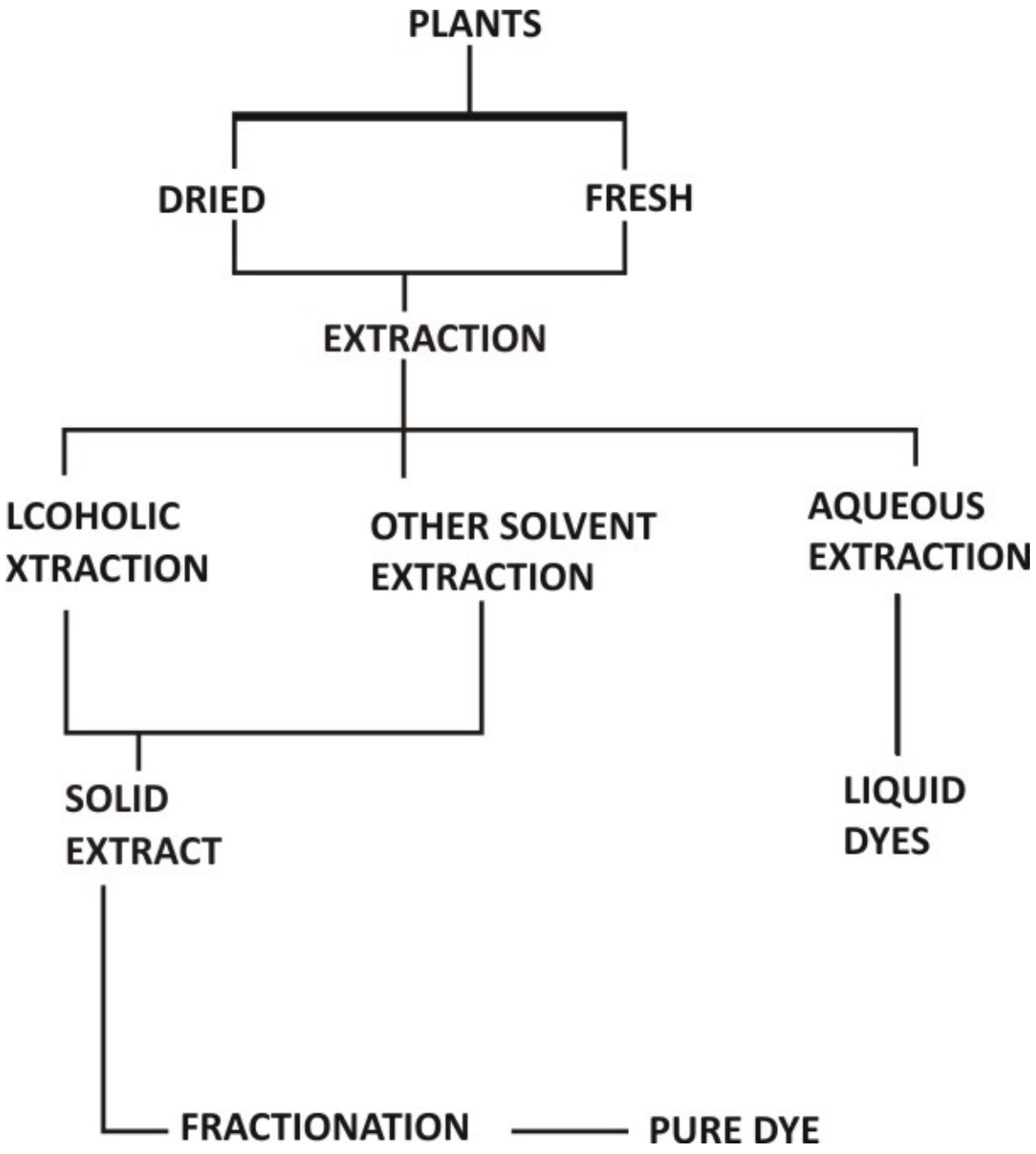
Methods of extraction of natural dyes

METHODS OF DYE EXTRACTION

Experimental trials were carried out in domestic gardens in collaboration with botanists mainly focusing on the best conditions for the growth of dye plants in regard to soil and climatic factors. Modern cultivation system for getting maximal dye yields including optimal seeding and harvesting time, optimal fertilization procedures were adapted. The utilizable plant parts were subjected to specific dehydration processes or the dyestuff was extracted as per the given strategy.


PLANTS
DRIED
FRESH
EXTRACTION
LCOHOLIC
XTRACTION
OTHER SOLVENT
EXTRACTION
AQUEOUS
EXTRACTION
SOLID
EXTRACT
LIQUID
DYES
FRACTIONATION
PURE DYE

Traditional method

- The traditional method used to extract the dyestuffs from all other plants mentioned earlier, where the plant material is added directly to the dye bath. This has been used by dyers for centuries and is still used by many dyers in north eastern states of India.

- **The disadvantages of this method are:**

- The plant material has to be separated from the textile

- It is not applicable to modern textile fabrication machines (pumps and spinerettes will be choked)

- Hard plant material such as madder roots or barks of Cassia, amla are dufficult to extract

- The low density of the dried material requires high processing volume

- Disadvantage has to be solved for use by modern mills. For industrial use the best method is to provide extracts. Aqueous extracts are not especially favorable for dye plants such as Parkia, Alkanet and Tulsi where we have used 50: 50 water : methanol extract for dyeing. The reason being that flavonoids, anthraquinones and aglycones are poorly soluble in water and therefore are extracted only partially.

Innovative Method for Extraction of dyes:

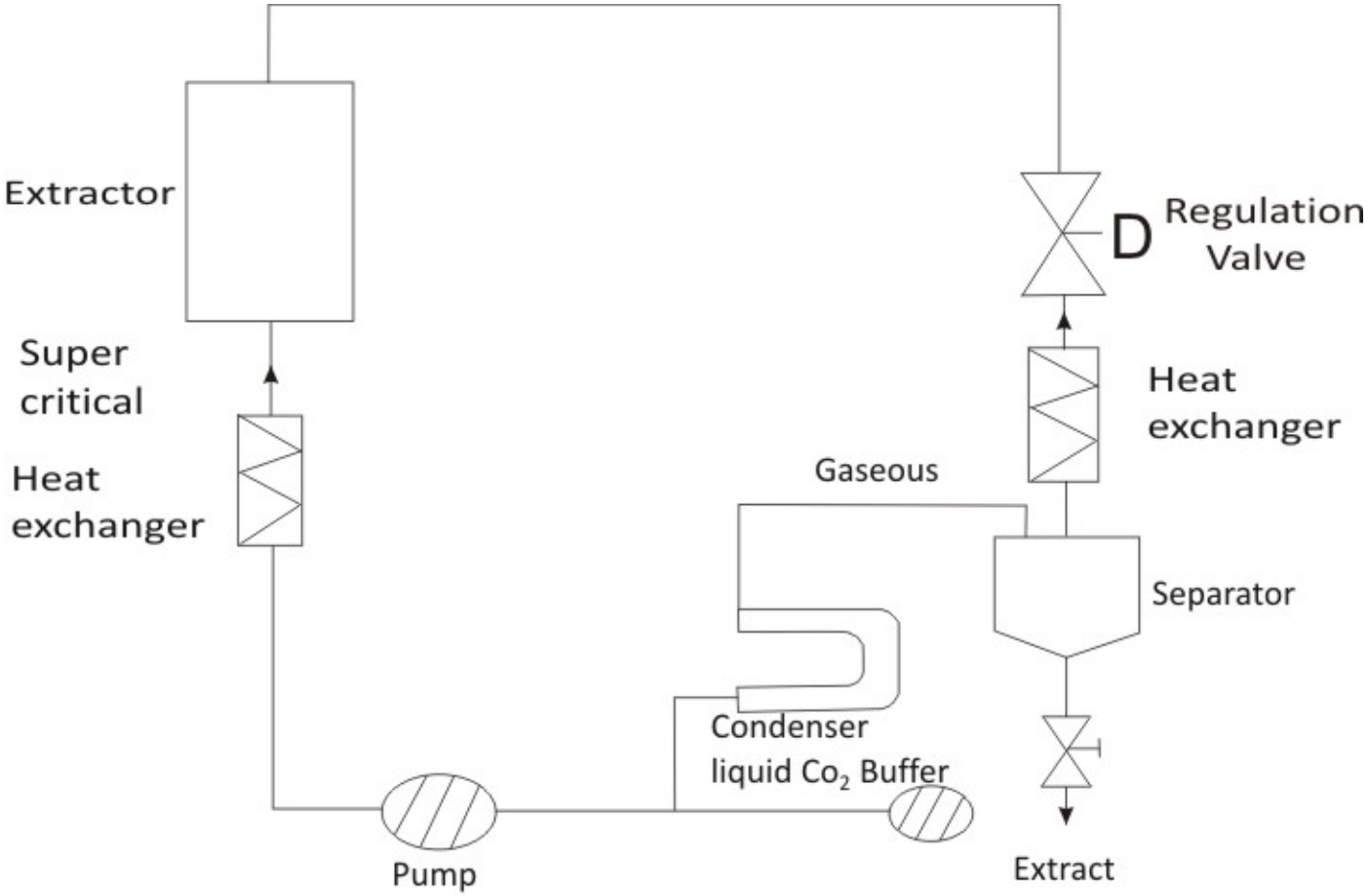
- Efficient extraction of the dye from the plant material is very important for standardization and optimization of vegetable dyes. Utilizing **a) Soxhlet**
- **b) Supercritical fluid extraction**
- **c) Subcritical water extraction and**
- **d) Sonicator methods**

SOXHLET

Soxhlet extraction

When a compound of low solubility needs to be extracted from a solid mixture a Soxhlet extraction can be carried out. The technique places a specialized piece of glassware in-between a flask and a condenser. The refluxing solvent repeatedly washes the solid extracting the desired compound into the flask. Soxhlet extraction was carried out for colorant identification. In this work dried plant parts were put into thistle of soxhlet extractor and methanol was used as solvent. Temperature of the instrument was maintained well under boiling point of the used solvent. Several cycles of solvent were run so as to extract all the compounds from plant parts.

SUPERCRITICAL EXTRACTOR

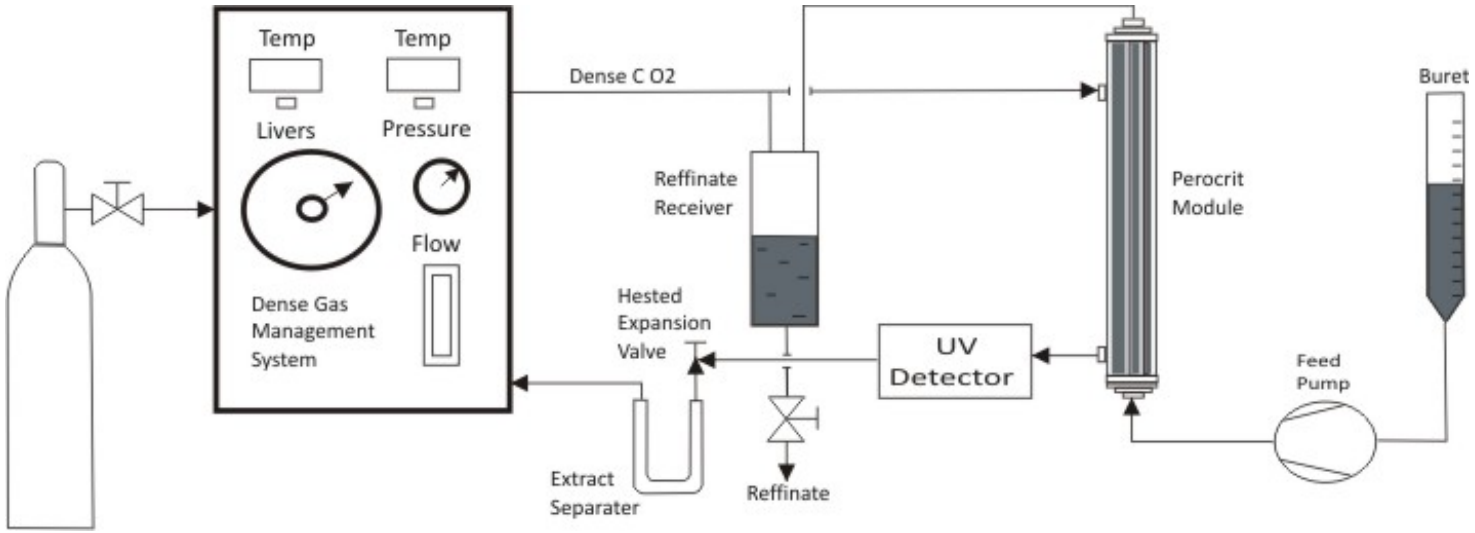


SUPERCRITICAL CO_2 EXTRACTOR

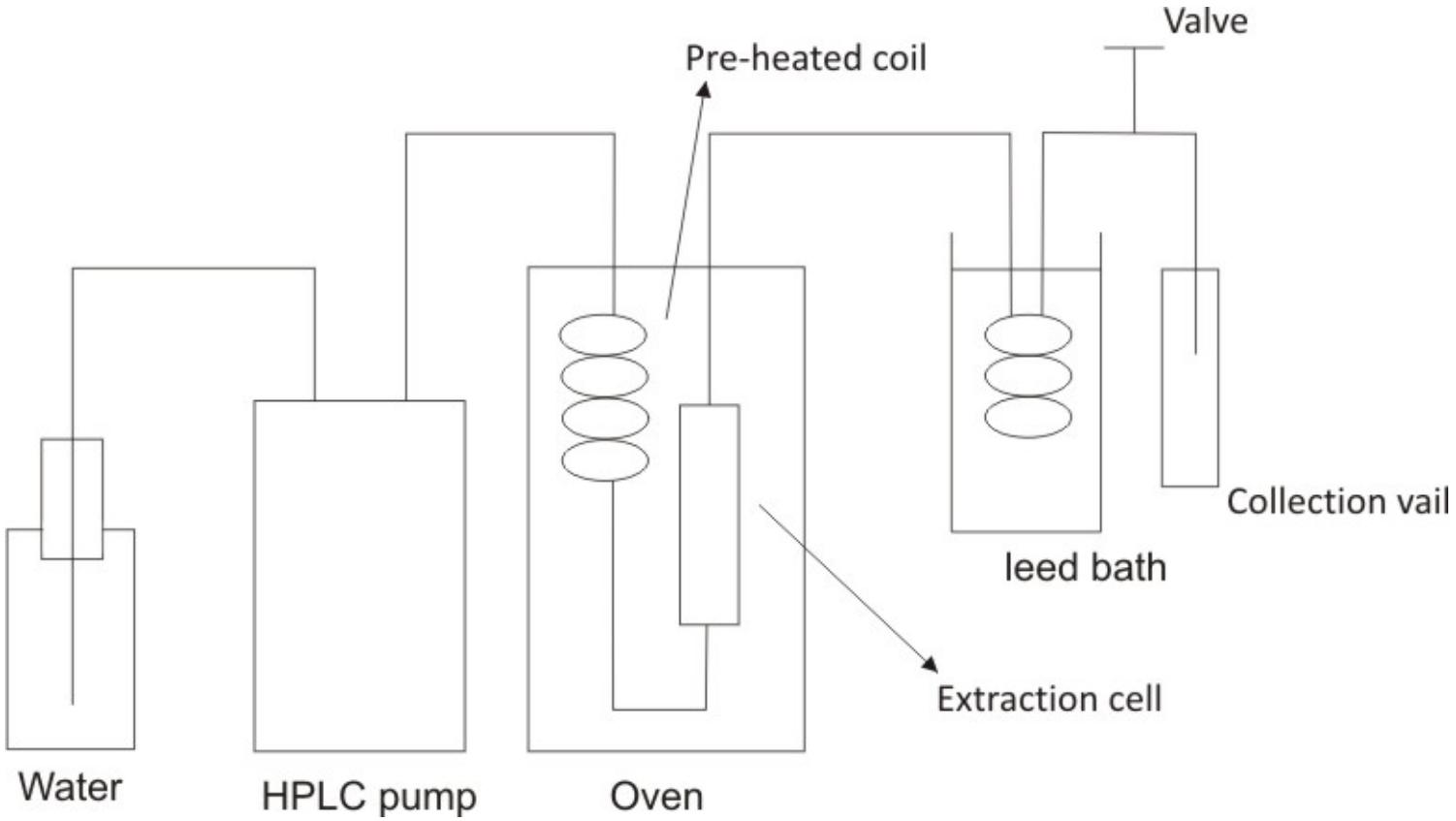
SCFE

- SCFE is a two step process, which uses a dense gas as solvent usually carbon dioxide above its critical temperature (31°C) and critical pressure (74 bar) for extraction. The natural product is powdered and charged into the extractor. Carbon dioxide is fed to the extractor through a high-pressure pump (100-350 bar). The extract charged carbon dioxide is sent to a separator (60-120 bar) via a pressure reduction value. At reduced temperature and pressure conditions the extract precipitates out in the separator. The extract free carbon dioxide stream is introduced several times for effective extraction of all the dye material from the natural product.

- Why SCFE is superior over the traditional solvent extraction of natural dyes? Firstly it uses a clean, safe, inexpensive, nonflammable, nontoxic, environmentally friendly, nonpolluting solvent-carbon dioxide (CO_2). Secondly, the energy costs associated with SCFE are lower than the conventional techniques.



Sub-critical Extractor



Graphical Subcritical Water Extraction

Subcritical Water Extraction

Subcritical Water Extraction was performed with some plants to extract natural colorant. The water was purged with nitrogen to remove dissolved oxygen prior to the extraction. Deoxygenated water was used in an HPLC pump programmed for a constant flow of 1–3 ml/min-1. A 10.4 ml extraction cell equipped with 0.5 m frit at the inlet and outlet was connected to a 1 m cooling loop (in ice water) outside of the oven. A pressure control valve was placed between the cooling loop and the collection vial. The extraction was carried out in efficient manner.

SONICATOR

Sonicator

Ultrasound-assisted extraction: UAE was carried out by mixing dried and ground sample in methanol or any solvent in a flask, which was then placed in an ultrasonic bath for 30 min. At the beginning, the temperature of extraction was 20-40°C and after one hour of extraction it becomes 60°C. The extraction was repeated two-three times and the extracts were collected.

Details of the method developed by us

Ultra Sonication-Assisted extraction: 20 g of flowers were mixed with 50 ml of each solvent (MeOH, EtOH, EtOAc, Ac) and water in a 100 ml conical flask. The ultrasound chamber (JULABO, USR3) having a frequency of 20 Hz using different sonic power (100–500 W) for different time intervals (15–120 min) was used. The power level was set at the maximum (level 9) and the temperature during the 1–2 hour extraction period was stabilized at 25-30°C during the extraction. For indirect sonication, the sample (conical) was immersed in an ultrasound chamber at the half height of the solution in the flask, operating at 20 kHz frequency. The temperature was controlled and maintained at below 30°C by periodical replacing water in the bath with cold one (15°C). The flask was taken out and cooled to room temperature by water. The flower extracts were filtered through Whatman No. 42 filter paper and the solution was collected. The residue was taken back and extracted again in the same conditions. The extracts of the twice-extraction were mixed. The ultrasound power actually delivered to the extracting liquid by the sonic bath was determined by the calorimetric method. This suggests that the Ultra Sonication-Assisted extraction process is more selective than the conventional one.

Extraction of dyes

- Two types of extraction were carried out for characterization of the colorant as well as analyzing the effect of newly found dye resources on cotton silk and wool. The extraction procedure followed namely

- **Alcoholic extraction**

  Dry leaves were finely crushed through a grinder and then subjected to soxhlet extraction, using methanol as a solvent. The cycle is repeated for three times at 60º C. Then the cooled extract was filtered through a filter paper and solvent was removed through a rotary evaporator.

- **Aqueous extraction**

  Respective plant parts were taken and poured in boiling water and then kept on water bath at 60^0C for about one hour so as to extract all the color from them.