QUALITY CONTROL OF CRUDE DRUGS

Chemical Evaluation

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CHEMICAL EVALUATION

- It comprises of different chemical tests and assays.
- Isolation, purification and identification of active constituents are chemical methods of evaluation.
  - **Resin**: Sulphated ash, acid value
  - **Balsam**: Acid, saponification and ester values
  - **Volatile oil**: Acetyl and ester values
- Preliminary phytochemical investigation is a part of chemical evaluation.
- Qualitative chemical tests are useful in detection of adulteration.
QUALITATIVE CHEMICAL TESTS

- Tests for Alkaloids

**Dragendorffs test** (Potassium bismuth iodide solution): Orange red colour precipitate

**Mayer’s test** (Potassium mercuric iodide reagent): Cream colour precipitate

**Wagner’s test** (Iodine-potassium iodine): Reddish-brown precipitate

**Hager’s reagent** (Saturated solution of iodine): Yellow precipitate
Tests for tannins

Ferric chloride reagent
A 5% w/v solution of ferric chloride in 90% alcohol was prepared. Few drops of this solution were added to a little of the drug filtrate. If dark green or deep blue colour is obtained, tannins are present.

Lead acetate test
A 10% w/v solution of basic lead acetate in distilled water was added to the test filtrate. If precipitate is obtained, tannins are present.
Tests for flavonoids (Shinoda test)
A small quantity to test residue was dissolved in 5 ml ethanol (95% v/v) and reacted with few drops of concentrated hydrochloric acid and 0.5 g of magnesium metal. The pink, crimson or magenta colour is developed within a minute or two, if flavonoids are present.

Tests for amino acids

Ninhydrin test
The Ninhydrin reagent is 0.1% w/v solution of ninhydrine in n-butanol. A little of this reagent was added to the test extract. A violet or purple colour is developed if amino acids are present.
Tests for proteins

Biuret test:
A few mg of the residue was taken in water and 1ml of 4% sodium hydroxide solution was added to it. A drop of 1% solution of copper sulphate followed this. **Violet or pink** colour is formed if proteins are present.

Xanthoproteic test
A little residue was taken with 2 ml of water and 0.5 ml of concentrated nitric acid was added to it. **Yellow colour** is obtained if proteins are present.
Tests for sugars

Fehling’s solution test

The Fehling’s solution was prepared as follows:

Solution A:
- Copper sulphate - 34.64 g.
- Sulphuric acid - 0.5 ml
- Distilled water to - 500 ml

Sodium B:
- Sodium potassium tartarate - 176 g
- Sodium hydroxide - 77 g
- Distilled water to - 500 ml

The two solutions were mixed in equal volumes immediately before use. Drug is treated with mixture and warmed. If a red precipitate of cuprous oxide is obtained, reducing sugars are present.
THIN LAYER CHROMATOGRAPHY (TLC)

- Based on adsorption chromatography
- Adsorbent: Silica gel G (3 mm thick)
- Activation: Heating the plate at 105°C for 30 minutes in an oven
- Solvent system: Depend on the nature of chemical constituents.
- For volatile oil
  Toluene: ethyl acetate (97:3)

\[ Rf = \frac{\text{Distance travelled by component}}{\text{Solvent front}} \]
High performance thin layer chromatography (HPTLC)

HPTLC plate: silica gel G F$_{254}$

Sample, 10 mg/ml; Standard, 1 mg/ml;
Mobile phase: toluene - ethyl acetate, (90:10)
Scanning: 313 nm

Imperatorin standard from Sigma Aldrich, New Delhi, India.

HPTLC chromatogram of
(A) Standard imperatorin,
(B) Methanolic extract NJ,
(C) Methanolic extract of RD,
(D) Methanolic extract of AP,
(E) Methanolic extract of TA and
(F) Safoof-e-Muhazzil

**Imperatorin content in the samples and formulation**

<table>
<thead>
<tr>
<th>Test samples</th>
<th>Imperatorin content (Mean ± SD, % w/w)</th>
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<tbody>
<tr>
<td><em>N. jatamansi</em> DC. (NJ)</td>
<td>1.58 ± 0.03</td>
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<tr>
<td><em>Rosa damascena</em> Mill. (RD)</td>
<td>0.44 ± 0.03</td>
</tr>
<tr>
<td><em>Apium graveolens</em> L. (AG)</td>
<td>3.05 ± 0.02</td>
</tr>
<tr>
<td><em>Trachyspermum ammi</em> L. (TA)</td>
<td>1.23 ± 0.01</td>
</tr>
<tr>
<td>Safoof-e-Muhazzil</td>
<td>2.16 ± 0.02</td>
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</tbody>
</table>
OTHER ANALYTICAL TECHNIQUES

- High Performance Liquid Chromatography, HPLC
- Gas Liquid Chromatography, GLC
- Column Chromatography, CC
- Ultra-violet and Visible spectrophotometry
- Infra-Red Spectroscopy, IR
- Nuclear Magnetic Resonance Spectroscopy, NMR
- Mass Spectroscopy
HEAVY METAL ANALYSIS

- The medicinal plant materials can be contaminated with Arsenic, Lead, Mercury, and Cadmium. As these components even in trace amounts are dangerous, they have to be removed from the herbal drugs. The heavy metals are analyzed by using technique of Atomic Absorption Spectrophotometry.

- Limit for Arsenic (As): 10 mg/ kg (= 10.0 ppm)
- Limit for Cadmium (Cd): 0.3 mg/ kg (= 0.3 ppm)
- Limit for Lead (Pb): 10 mg/ kg (= 10.0 ppm)
- Limit for Mercury (Hg): 1 mg/ kg (= 1.0 ppm)
**DETERMINATION OF AFLATOXINS**

- Aflatoxins are the mycotoxin from *Aspergillus flavus* and *Aspergillus parasiticus* having the chemical formula $\text{C}_{17}\text{H}_{12}\text{O}_6$. This is predicted to cause hepatic carcinoma in human beings. The plant species may be contaminated with this toxin.

- The test for aflatoxins as prescribed by WHO for the herbal drugs is designed to detect the presence of $\text{B}_1$, $\text{B}_2$, $\text{G}_1$, and $\text{G}_2$. The method used to detect is presence is simple TLC developments. The presence of aflatoxins is detected by blue fluorescence spot detected in UV light at 365 nm.
PESTICIDES RESIDUES

- Pesticides are simple substances or mixtures used to eliminate undesirable vegetable and animal life in agricultural and urban ecosystems.
- Pesticides can be classified according to their chemical composition, function and mode of action in organisms.
  - Chlorinated hydrocarbons and related pesticides: Aldrin, BHC, DDT, DDD, DDE, Endrin, Lindane etc.
  - Chlorinated phenoxyalkanoic acid herbicides: 2,4-D & 2,4,5-T.
  - Organophosphorous pesticides: Malathion, Carbophenothion, Parathion etc.
  - Carbonate insecticides: Carbaryl (Carbaril).
- Chromatography (mostly column and gas) is recommended as the principal method for the determination of pesticides residues.
Thank You