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## X-RAY DIFFRACTION STUDY OF A PANCHAVAKTRA RAS

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### ABSTRACT

#### Keywords:

A Herbo-Mineral Preparation,  
Three Samples of Panchavaktra Ras,  
X-Ray Diffraction Study

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Analytical monitoring of Pharmaceutical products is necessary to ensure its safety and efficacy throughout all phases of the drug. A systematic approach should be adapted to the presentation and evaluation of stable information, which should include, as necessary, physical, chemical, biological and microbiological test characteristics. Indian System of Medicine (ISM) frequently uses metal/mineral drugs. It is mandatory to standardize the preparatory procedures. For the Pharmaceutical standardization, three batches of the Panchavaktra ras were prepared and analytical study of final product carried out. Panchavaktra ras consist equal parts of Parada (Mercury), Gandhaka (Sulphur), Tankana (Borax) Pippali (*Piper longum* L.) and Marica (*Piper nigrum* L.), and Mardhana (Grinding) was done carefully with sufficient quantity of juice of Datura metal leaves for 24 hours and a final product was obtained in the form of Vati (tablet) form. It is one of the formulations mentioned in Amavata (Rheumatoid Arthritis) disease. The final products were subjected to Powder X-Ray Diffraction (XRD) studies and values of XRD peaks of particular Panchavaktra ras were observed. This study revealed that high peaks of HgS (Metacinnabar), free S (Sulfur), Chabazite (Ca- exchanged), dehydrated Ca (structure- Rhombohedral) in the final products. The structural and chemical characterization of the HgS (Metacinnabar) found as cubic, free S (Sulphur) as Orthorhombic in all the samples. The 50% strongest peaks of HgS were present at 2-Theta scale between 26-31, 43-44, 51-55, 70-72 degrees. This paper points out the importance of XRD, as a standard tool for further studies and research of Herbo-mineral formulations.

**INTRODUCTION:** Khalviya rasayana (mortar pestle apparatus used medicinal preparations) is the one among different preparations, Khalviya method is a basic procedure applicable to all Rasaoushadis (Mercurial medicines) before they are subjected to any specific procedure. It is a simple process to convert crude drugs i.e. macro to micro level and also gives specific Samskara (stages of processing) to a drug. Panchavaktra ras is, most commonly used by various Acharyas and studied in various diseases.

In the name of Panchavaktra ras, various formulations are available in ayurvedic texts. This formula is taken from well known Andhra Telugu book named Basavarajeeyam <sup>1</sup>, 6<sup>th</sup> chapter (Vataroga nidana lakshana chikitsadhya) in the treatment of Amavata (Rheumatoid Arthritis) disease.

Panchavaktra ras consists of equal parts of

1. Parada (Mercury-Hg)

2. Gandhaka (Sulphur-S)
3. Tankana (Borax- $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ )
4. Pippali (*Piper longum* Linn.)
5. Marica (*Piper nigrum* Linn.)
6. Bhavana (maceration) with the juice of Krishna dhatura leaves (*Datura metel* Linn.)

All these ingredients were procured from the local market of Vijayawada, Andhra Pradesh, India and all the herbal and mineral material were thoroughly screened by experts of rasashastra, Dr. N.R.S. Govt. Ayurvedic College, Vijayawada, based on the Grahya lakshanas (to be taken or accepted characters) mentioned in the classics.

Kajjali is prepared initially with one part of Parada and Gandhaka then added to one part of each Tankana, Pippali and Maricha, Bhavana (Impregnation or Maceration) given with Krishna Dhatura patra swarasa

(Juice of Black Datura metal leaves). Standardization of the pharmaceutical preparation of Panchavaktra Ras by preparing in three seasons and analytical study of three samples were carried out.

## MATERIALS & METHODS:

### 1. Purification of the ingredients:

- a. **Purification of Parada (Mercury)**<sup>2</sup>: Parada is taken with equal quantity of Nagavalli svarasa (Juice of betel leaf), Ardraka svarasa (Ginger juice) and Triakshara (Yava, Sarja, Tankana) in a clean Khalva yantra and rubbed for eight hours per day for three days. The obtained material is washed and poured out with the help of lukewarm water for several times until we get clean and clear Parada (Procedure can be seen in figure 1).



FIGURE 1: PURIFICATION OF PARADA (MERCURY)

**b. Purification of Gandhaka (Sulphur)**<sup>3</sup>: A sharava (earthen vessel) with wide mouth is taken and filled with 2 litres of Gokshira (Cow's milk) and 150 ml of Goghrita (Cow's ghee) then the mouth of vessel is covered by a cloth and tied by iron wire. The coarse powdered Gandhaka spread upon the cloth and a earthen vessel is covered on it. The joint being closed with Multanimitti (Fuller's earth.) smeared cloth for at least 3 times and allow it to dry in sunlight. This vessel is kept inside a pit (1.5") beneath the surface of soil in such a way, so as to keep the brim of the vessel on the level of the surface of ground. The empty space around vessel is filled by loose soil. Cow dung cakes are kept on earthen vessel and set on fire. The Sulphur, after melting by fire flows down in to the ghee mixed milk vessel through cloth. After Svangasita (self cooling), the vessel is taken out carefully from pit and Sandhibandhan (sealing) is opened. The granules of Gandhaka collected at the bottom of vessel are washed with hot water (Procedure can be seen in figure 2).



**FIGURE 2: PURIFICATION OF GANDHAKA (SULPHUR)**

**c. Purification of Tankana (Borax)**<sup>4</sup>: Raw Tankana is taken in a clean and dry Khalva yantra and pounded well to prepare powder. This powder is taken in to a Sharava then it is heated in Mandagni (Mild heat), followed by Tivragni (maximum heat), until all the water content in the Tankana is completely evaporated. Finally

Tankana is obtained as a white coloured puffy light substance (Procedure can be seen in figure 3).



**FIGURE 3: PURIFICATION OF TANKANA (BORAX)**

**d. Pippali and Marica:** These materials are taken in an earthen plate separately and frying was done on mild fire, and then made powder 80# sieve.

**2. Preparation of Panchavakra ras:** In the first Kajjali (black sulphide of mercury) prepared with the equal parts of purified Parada and Gandhaka in khalva yantra (mortar pestle apparatus) until the whole material turn into black, very fine like collyrium and the dazzling particles of mercury completely disappeared<sup>5</sup>. Then, the other ingredients were mixed thoroughly in specified ratio (1 part each) and grind in the Khalva yantra with the juice of Krishna dhatura leaves, to obtain a homogeneous blend.

The blended mass was dried in shade, then added starch, binding agents and lubricants (quantity mentioned in **table 1**) according to the drug quantity and made tablets through the punch machine fitted with suitable die. The rolled tablets were dried in a tray-dryer at a temperature not exceeding 60°C. It was packed in a tightly closed glass containers for further use. The final product of Panchavakra ras was found to be a dark gray coloured (Procedure can be seen in **figure 4**).

**TABLE 1: PREPARATION AND OBSERVATION OF PANCHAVAKTRA RAS**

| Ingredients  | Expt-1        | Expt-2        | Expt-3       |
|--|---------------|---------------|--------------|
| Shodhita Parada  | 300 g.        | 250 g.        | 200 g.       |
| ShodhitaGandhaka   | 300 g.        | 250 g.        | 200 g.       |
| ShodhitaTankan   | 300 g.        | 250 g.        | 200 g.       |
| Pippali powder   | 300 g.        | 250 g.        | 200 g.       |
| Maricha powder   | 300 g.        | 250 g.        | 200 g.       |
| Total Qty. of drug before Bhavana                                | 1500 g.       | 1250 g.       | 1000 g.      |
| Qty. of Dhattura patra Swarasa used for Bhavana                  | 2000 ml.      | 1600 ml.      | 1245 ml.     |
| Total Qty. of drug after Bhavana                                 | 2520 g.       | 2100 mg.      | 1700 g.      |
| Total Qty. of drug after drying                                  | 1925 g.       | 1600 g.       | 1300 g.      |
| Quantity of Starch added   | 50 g. (2.6 %) | 121 g. (7.6%) | 98 g. (7.6%) |
| Quantity of binding agents added                                 | 192 g. (10%)  | 64 g. (4 %)   | 65 g. (5 %)  |
| Quantity of Lubricants added (magnesium stearate, Talc, Aerosol) | 77 g. (4%)    | 64 g. (4 %)   | 52 g. (4%)   |
| Total weight of Drug mass  | 2245 g.       | 1849 g.       | 1515 g.      |
| Weight of the tablet   | 150 mg.       | 150 mg.       | 150 mg.      |
| Total no of Tablets  | 15000         | 12000         | 10000        |
| Date of commencement   | 13-11-2006    | 26-02-2007    | 25-02-2008   |
| Date of completion   | 17-11-2006    | 01-03-2007    | 31-02-2008   |

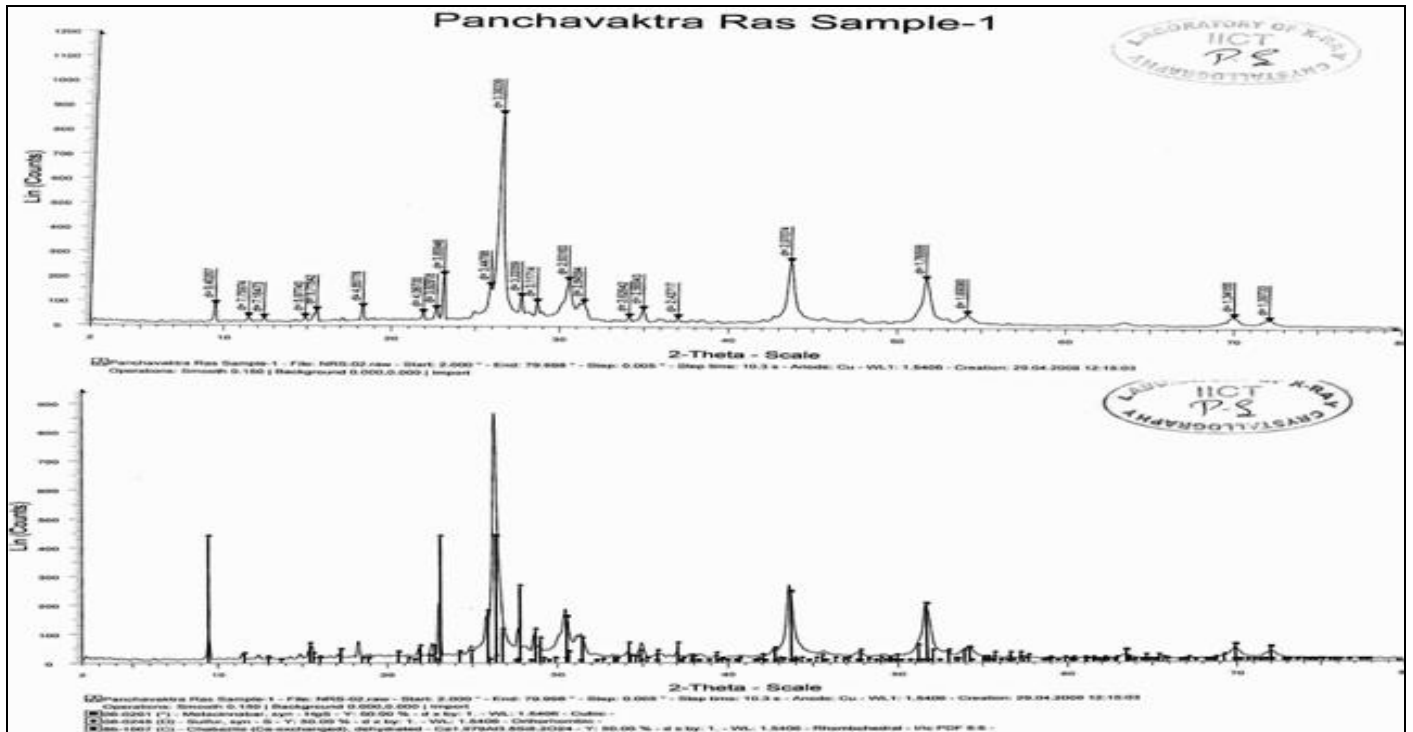
**FIGURE 4: PREPARATION OF PANCHAVAKTRA RAS**

3. **Powder X-Ray diffraction (XRD) Study:** Powder X-Ray diffraction (XRD) studies were carried out in the department of Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology (IICT), Hyderabad. X-ray diffraction (XRD) is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactured materials <sup>6</sup>. X-Ray Diffraction study is a powerful procedure for detecting the presence of various phases in a given sample. The basic principle of the phase analysis using powder XRD technique lies the presence of diffraction peaks corresponding to various inter planar ( $d_{hkl}$ ) spacings which are characteristics of a given material. The relative intensities of various

peaks occurring at different 'd' spacings are also different for different phases.

**Preparation of Sample:** The sample preparation for the analysis was done using standard XRD procedure. The sample was powdered to 100 mesh size using mortar and pestle, to get a homogenous powder mixture. The powder was then spread onto a double-side tape with a spatula, and then placed on a cavity with plastic holder. Care was taken to fill the powder uniformly in to the mount. It was exposed to x-ray beam of intensity 35KV and 20MA. All the peaks were recorded on the chart, and the corresponding  $2\theta$  values were calculated. Results are summarized in **figure 5, 6 and 7 and Table 2, 3 and 4.**

**Panchavaktra ras 1:**



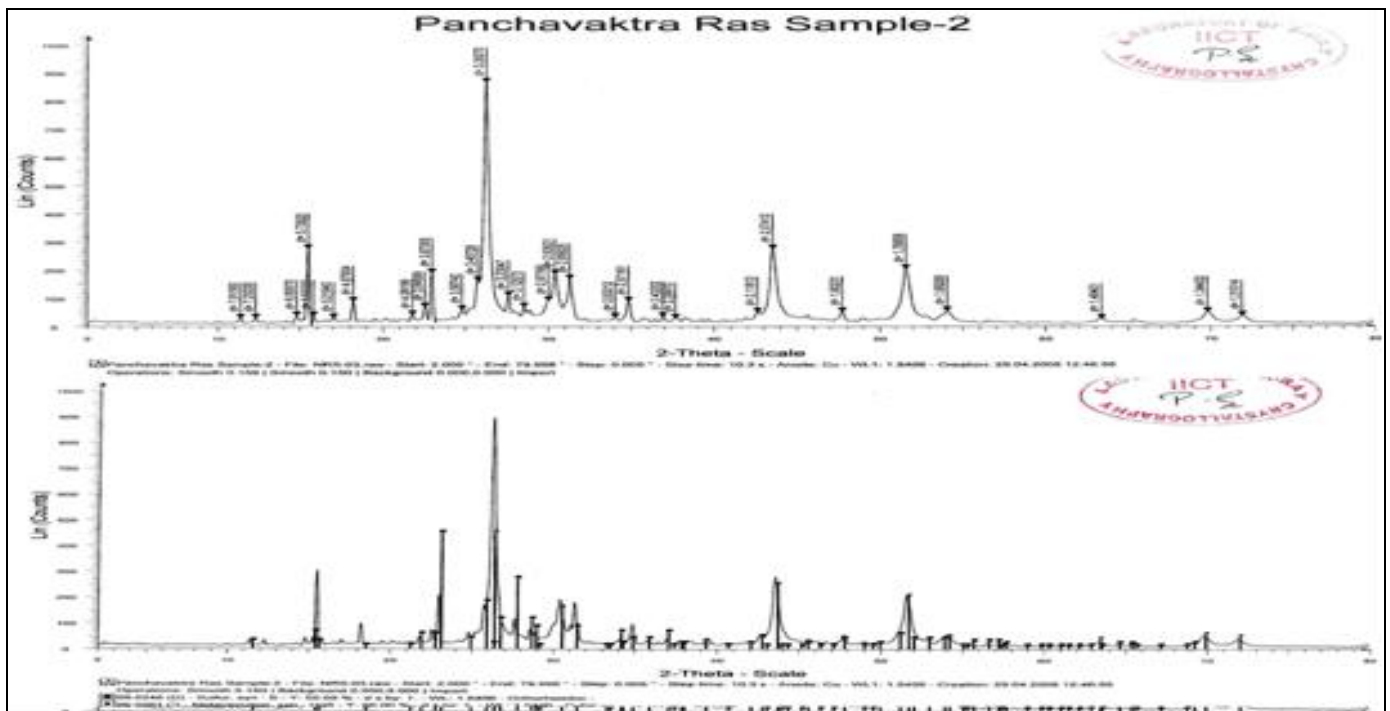
**FIGURE 5: X-RAY DIFFRACTION OF THE 1ST SAMPLE OF PANCHAVAKTRA RAS**

- ⇒ HgS (Metacinnabar)
- ◆ ⇒ S (Sulphur)
- ⇒ Chabazite (Ca- exchanged), dehydrated Ca, Al, Si

**TABLE 2: SHOWING 2θ (°) VALUE OF STRONGEST PEAKS OF 1ST SAMPLE OF PANCHAVAKTRA RAS**

| Panchavaktra ras | 2θ value                       |
|------------------|--------------------------------|
| HgS              | 26.4, 43.8, 51.7, 30.6, 70, 72 |
| Free S           | 23.1, 27.8, 25.9, 28.8         |
| Chabazite        | 9.5, 17.2, 20.8, 24.2, 30.7    |

**Panchavaktra ras 2:**



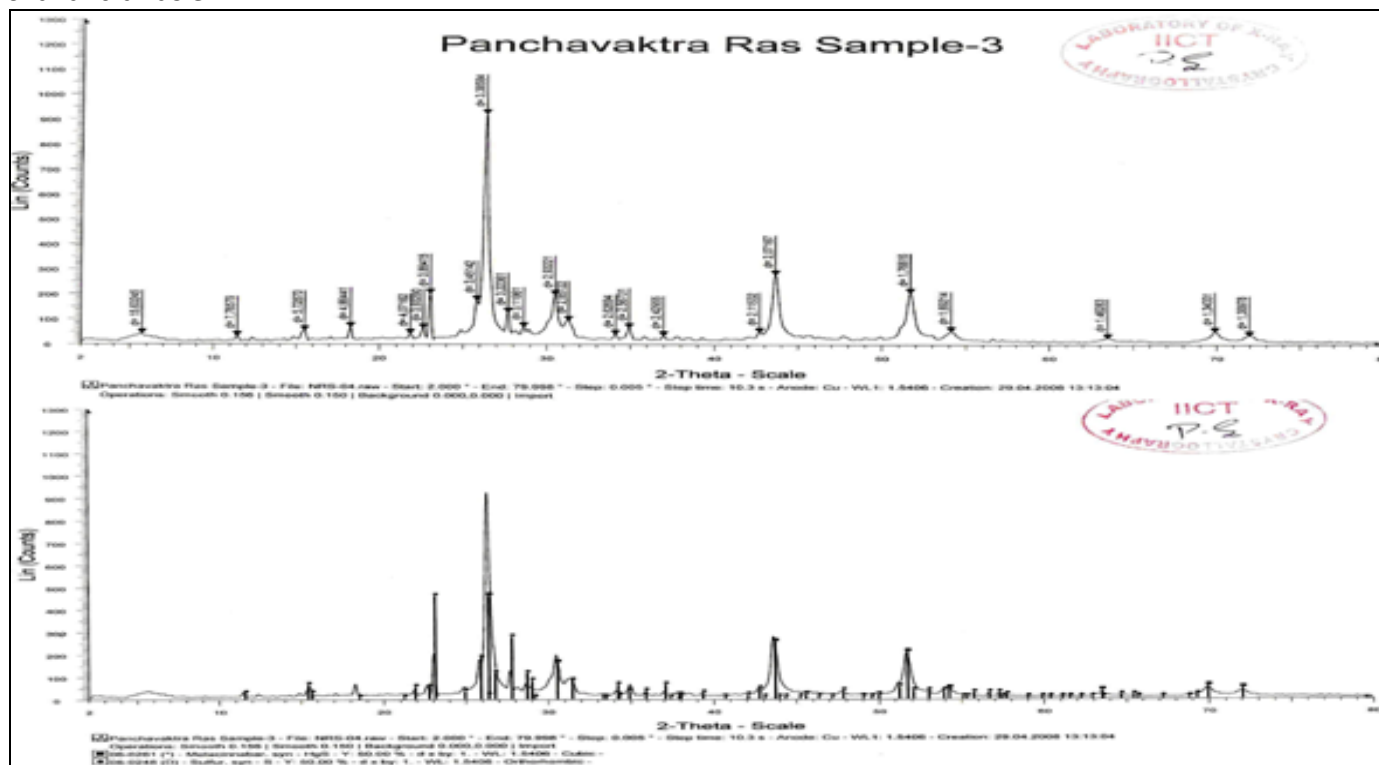
**FIGURE 6: X-RAY DIFFRACTION OF THE 2ND SAMPLE OF PANCHAVAKTRA RAS**

- ⇒ HgS (Metacinnabar)
- ◆ ⇒ S (Sulphur)

**TABLE 3:SHOWING 2θ (°) VALUE OF STRONGEST PEAKS OF 2ND SAMPLE OF PANCHAVAKTRA RAS**

| Panchavaktra ras | 2θ value                               |
|------------------|--|
| HgS              | 26.4, 43.8, 51.7, 30.6, 70, 72, 54.2   |
| S                | 23.1, 27.8, 25.9, 26.9, 28.8, 29, 31.5 |

**Panchavaktra ras 3:**



**FIGURE 7: X-RAY DIFFRACTION OF THE 3RD SAMPLE OF PANCHAVAKTRA RAS**

- ⇒ HgS (Metacinnabar)
- ◆ ⇒ S (Sulphur)

**TABLE 4: SHOWING 2θ (°) VALUE OF STRONGEST PEAKS OF 2ND SAMPLE OF PANCHAVAKTRA RAS**

| Panchavaktra ras | 2θ value                               |
|------------------|--|
| HgS              | 26.2, 43.8, 51.7, 30.6, 70, 72.1, 54.2 |
| S                | 23.1, 27.8, 26, 26.9, 28.8, 29, 31.5   |

**RESULTS:** In X-RD Graph d-spacing [Å] values of major peaks are compared with standard JCPDS card table for Mercury Sulphide CARD and Sulphur. Hence this card is studied for crystal structure present in this sample is Cubic and Orthorhombic (Figure 5). XRD pattern shows Mercury sulphide, free Sulphur and Chabazite (Ca-exchanged) dehydrated-Ca respectively.

**Panchavaktra Ras 1:** There are three compounds seen in the 1<sup>st</sup> sample

- A. HgS (Metacinnabar)

Structure: Cubic

- B. S (Sulphur)

Structure: Orthorhombic

- C. Chabazite (Ca-exchanged) dehydrated-Ca.

Structure: Rhombohedral

**Panchavaktra Ras 2:** There are two compounds seen in the 2<sup>nd</sup> sample.

- A. HgS (Metacinnabar)

Structure: Cubic

- B. S (Sulphur)

Structure: Orthorhombic

**Panchavaktra Ras 3:** There are two compounds seen in the 3<sup>rd</sup> sample.

- A. HgS (Metacinnabar)

Structure: Cubic

B. S (Sulpher)

Structure: Orthorhombic

**DISCUSSION & CONCLUSION:** All the three Panchavakra Ras samples show the several XRD peaks corresponding to HgS (Metacinnabar), Sulphur (S). The 50% of strongest peaks of HgS were present at 2-Theta scale between 26-31, 43-44, 51-55, 70-72 degrees. Free sulphur was also observed in the 2-Theta scale between 23, 28, but no free Mercury was detected in this study.

This study revealed that high peaks of HgS (Metacinnabar), S (Sulfur), Chabazite (Ca- exchanged), Chabazite (Ca- exchanged), dehydrated Ca (structure-Rhombohedral) in the final products. The structures of the HgS (Metacinnabar) found as cubic, S (Sulfur) as

Orthorhombic in all the samples. This paper points out the importance of XRD, as a standard tool for further studies and research of herbo-mineral formulations. X-ray diffraction (XRD) is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactured drugs.

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