Analytical assessment of Akika Pishti based on Ancient and Modern Parameters

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ABSTRACT

Pishti Kalpana is one of the Kharaliya Kalpanas of Rasashastra which brings the heat sensitive substances to micro particle level without applying the heat for better therapeutic efficacy and is considered as Sukshma as Bhasma. In Rasa texts it is said that Marana of Ratna and Uparatna are not worthwhile hence, Pishti Kalpana is advocated. There is no direct reference for the Siddhilakshana of Pishti, but our Acharyas has mentioned that Pishti is an Anagnisidhda Bhasma i.e. Pishti should be Bhasmavat, so the parameters mentioned for quality control of Bhasma in ancient text are applicable for assessing the Siddhilaxanas of Pishti. So an attempt has been made to study analytically Siddhilaxana of Pishti by both ancient and modern parameters with special reference to Akika Pishti. Akika is one of the semiprecious gem grouped under the Paradadi Varga, Uparatna Varga and Spatika Varga Ratna. In present study Pishti was prepared as per pharmacopoeial standards and subjected to both ancient and modern tests to analyze viz. Pishtivarna, Mrudutva and Silakshanatva, Rekhapurnata, Varitara, Nirdhooma, Unama and Nischandratva tests according to classics and according to modern parameters like organoleptic tests, physio-chemical like LOD, pH, ash values, instrumental analysis like XRD, SEM-EDAX and particle size.

Key words: Pishti, Akika, XRD, SEM-EDAX.

INTRODUCTION

The important aims of Analytical study are to know the particular chemical configuration and to point out the Physico-chemical changes and effect of different Samskara (Nirvapa, Bhavana, Mardana etc.) and also to know the probable role of a media during the pharmaceutical processing.

Pishti Kalpana is one of the Kharaliya Kalpanas of Rasashastra which brings the heat sensitive substances to micro particle level without applying the heat for better therapeutic efficacy and is considered as Sukshma as Bhasma.

In Rasashastra texts it is said that Marana of Ratna and Uparatna are not worthwhile[1] hence, Pishti Kalpana is advocated. The Pishtis are mainly examined in terms of physical test and chemical test. There is no direct reference for the Siddhilaxana of Pishti, but our Acharyas has mentioned that Pishti is an Anagnisidhda Bhasma i.e. “Pishti should be Bhasmavat”[2] so the parameters mentioned for quality control of Bhasma in ancient text are applicable for assessing the Siddhilaxanas of Pishti. In the present study, apart from ancient Parameters, Physico-chemical analysis of the drugs are carried out by using current analytical methodologies for better understanding and interpretation of physico-chemical changes occurring during and after pharmaceutical processing and also to understand the chemical reaction between drugs and components of biological system on which the drug action having effective result.
MATERIALS AND METHODS

Raw Akika was procured from Jaipur mines. The Shodhana Dravya - milk and Bhavana Dravyas like Kumari, Ketaki Pushpa, Jalapippali and Kadali Kanda were collected from local sources.

Shodhana of Akika was carried out with Nirvapa method in Godugdha for 21 times and Akika Pishti was prepared by Bhavana method with Kumaripatra Swarasa, Ketaki Pushpa Swarasa, Jalapippali Panchanga Swarasa each 5 times and followed by 6 times Bhavana with Kadali Kanda Swarasa. After 21 Bhavana, Mardana was carried out and stored in air tight container.

Analytical Study

In the present study RA(Raw Akika ), SA(Shodhita Akika) and AP(Akika Pishti) were subjected to both ancient and modern tests to analyze them viz. Pishtivarna, Mrudutva and Slakshnatva, Rekhapurnatva, Varitara, Nirdhooma tests according to classics and according to modern parameters - Organoleptic tests, physico chemical like LOD, pH, ash values, Instrumental analysis like XRD, SEM-EDAX and particle size.

Physical tests

This part of study was carried out at Post Graduate Department of Rasashastra and Bhaishajya Kalpana T.G.A.M.C.Ballari and Quality Control Lab, A.L.N. Rao, Ayurvedic Medical College, Koppa. Karnataka. Particle size analysis of RA, SA and AP was carried out at IISC Bengaluru. Karnataka.

Chemical Tests

X-ray diffraction method for crystallographic study of RA, SA, AP was done at Innovative centre, Manipal Institute of Technology, Manipal. SEM-EDX conducted by means of Selective Electron Microscopy for elemental analysis study of RA, SA and AP was done at Nanotechnology department, IISc, Bengaluru.

RESULTS

Table 1: Classical parameters for the analysis of Pishti

<table>
<thead>
<tr>
<th>SN</th>
<th>Parameters</th>
<th>RA</th>
<th>SA</th>
<th>AP</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Varna</td>
<td>Shweta</td>
<td>Dhusara</td>
<td>Kapota</td>
</tr>
<tr>
<td>2</td>
<td>Chandrika</td>
<td>Present</td>
<td>Absent</td>
<td>Absent</td>
</tr>
<tr>
<td>3</td>
<td>Rekhapurnatva</td>
<td>Negative</td>
<td>Negative</td>
<td>Positive</td>
</tr>
<tr>
<td>4</td>
<td>Slakshnatva,</td>
<td>Negative</td>
<td>Negative</td>
<td>Positive</td>
</tr>
<tr>
<td>5</td>
<td>Mridutva</td>
<td>Negative</td>
<td>Negative</td>
<td>Positive</td>
</tr>
<tr>
<td>6</td>
<td>Varitara</td>
<td>Negative</td>
<td>Negative</td>
<td>Positive</td>
</tr>
<tr>
<td>7</td>
<td>Unama</td>
<td>Negative</td>
<td>Negative</td>
<td>Positive</td>
</tr>
<tr>
<td>8</td>
<td>Nirdhooma</td>
<td>Negative</td>
<td>Negative</td>
<td>Positive</td>
</tr>
<tr>
<td>9</td>
<td>Nischandratva</td>
<td>Negative</td>
<td>Negative</td>
<td>Positive</td>
</tr>
</tbody>
</table>

Organoleptic Characters

Table 2: Organoleptic characters of RA, SA and AP

<table>
<thead>
<tr>
<th>Sample</th>
<th>Color</th>
<th>Odour</th>
<th>Taste</th>
<th>Taste</th>
</tr>
</thead>
<tbody>
<tr>
<td>RA</td>
<td>Light grey</td>
<td>Odourless</td>
<td>Tasteless</td>
<td>Crystalline</td>
</tr>
<tr>
<td>SA</td>
<td>Greyish white</td>
<td>Odourless</td>
<td>Tasteless</td>
<td>Semi amorphous</td>
</tr>
<tr>
<td>AP</td>
<td>Dark grey</td>
<td>Odourless</td>
<td>Tasteless to salty</td>
<td>Amorphous</td>
</tr>
</tbody>
</table>

Table 3: Physical Test results of RA, SA and AP.

<table>
<thead>
<tr>
<th>Contents</th>
<th>RA</th>
<th>SA</th>
<th>AP</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH Value</td>
<td>7.84</td>
<td>7.78</td>
<td>6.87</td>
</tr>
<tr>
<td>Ash Value</td>
<td>99.00%</td>
<td>99.00%</td>
<td>48.50%</td>
</tr>
<tr>
<td>Acid insoluble ash</td>
<td>97.50%</td>
<td>98.80%</td>
<td>14.50%</td>
</tr>
</tbody>
</table>
Table:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value 1</th>
<th>Value 2</th>
<th>Value 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water soluble ash</td>
<td>2.00%</td>
<td>0.5%</td>
<td>33.00%</td>
</tr>
<tr>
<td>Loss on drying at 110°c</td>
<td>0.15%</td>
<td>0.50%</td>
<td>1.90%</td>
</tr>
</tbody>
</table>

X-Ray Diffraction Results
The RA sample shown total 29 d-space values among them 27 peaks were matched with standard peaks of quartz low (α-quartz) with hexagonal crystalline structure.
structure. The SA showed total 28 d-space values among them one peak of SA has matched with standard peaks of silicon and one with quartz high (β-quartz) which are structurally cubic and hexagonal respectively.

The AP shown total 21 peaks, among them only one peak matched with Gamma Fe of standard peak which is cubic in structure. 17 peaks were matched with quartz, only one peak matched with high quartz, and one peak matched with silicon dioxide. Even though the forms of silicon were different in AP, but crystal structure was same i.e, hexagonal.

Table 4: XRD Results of RA, SA and AP

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compound Name</th>
<th>Chemical Formula</th>
<th>Crystal Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>RA</td>
<td>Quartz low</td>
<td>SiO₂</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>SA</td>
<td>Silicon</td>
<td>Si</td>
<td>Cubic</td>
</tr>
<tr>
<td></td>
<td>High quartz</td>
<td>SiO₂</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>AP</td>
<td>Gamma Fe</td>
<td>Fe</td>
<td>Cubic</td>
</tr>
<tr>
<td></td>
<td>Quartz</td>
<td>SiO₂</td>
<td>Hexagonal</td>
</tr>
<tr>
<td></td>
<td>High quartz</td>
<td>SiO₂</td>
<td>Hexagonal</td>
</tr>
<tr>
<td></td>
<td>Silicon dioxide</td>
<td>SiO₂</td>
<td>Hexagonal</td>
</tr>
</tbody>
</table>

Fig. 7: XRD of RA

Fig. 8: XRD of SA

Fig. 9: XRD of AP

SEM-EDAX RESULT

Table 5: Comparative Results of SEM-EDX

<table>
<thead>
<tr>
<th>Elements</th>
<th>RA</th>
<th>SA</th>
<th>AP</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weights %</td>
<td>Atomic %</td>
<td>Weights %</td>
</tr>
<tr>
<td>O</td>
<td>30.5</td>
<td>43.5</td>
<td>38.9</td>
</tr>
<tr>
<td>Si</td>
<td>69.4</td>
<td>56.5</td>
<td>61.1</td>
</tr>
<tr>
<td>Na</td>
<td>0.50</td>
<td>0.3</td>
<td>0.5</td>
</tr>
<tr>
<td>Mg</td>
<td>0.62</td>
<td>0.4</td>
<td>0.6</td>
</tr>
</tbody>
</table>
For the present research work Analytical Study was carried considering both ancient and modern parameters.

**Ancient Parameters**

The obtained AP was dark grey fine powder i.e., possessed *Sukshmatva* which indicates the fineness of Pishti obtained by doing pressurized, uniform and continuous *Bhavana* for 148 hours and the change in color might be due to the ionic exchange between Akika powder and functional groups of herbal extract.

*Rekhapurnata*

This *Pariksha* reveals the particle size softness as well as fineness of the particles. When the diameter of particles is less than the breadth of grooves on finger surface then only *Pishti* can pass this *Pariksha*. Its bioavailability is influenced by this factor.

**Varitara**

In present study the test *Varitara* was positive, indicating that the density of AP is having very less weight, so as to unable to break the surface tension of
water and hence easily absorbable. Rationale behind this test may be that smaller the particle size larger will be its surface area. Particle with larger surface area will float on water. Varitara test is indirectly gives us the idea about the reduced particle size of the Pishti.

Nama Pariksha

It is next step of Varitara. When drug shows Varitara test positive then, it can even load a grain of rice over drug while floating on water indicating that its particle size is very less and surface area is very high hence it will be able to stand on surface of water without breaking its surface tension even when it is loaded by rice grain.

Nirdhooma Pariksha

This is qualitative test which is mainly indicated to check the presence free sulfur molecules in obtained Pishiti. If it is negative than it indicates presence of free sulfur molecules and vice-verse. In present study Nirdhooma test was positive so it shows absence of free sulfur molecules as well as moisture content in AP.

Nischandrika

It checks the presence of free metallic compounds in final product. In present study AP has pass the Nischandrika Pareeksha which proves there is no free metallic compounds in AP.

Physical Parameters

pH

pH is the negative logarithm of hydrogen ion. pH helps to know about acidity and alkalinity of the drugs, acid base and salts come under electrolyte either strong or weak electrolyte. It helps for the absorption of the drug in acid media and intestinal enzyme media.

The results shows RA is weak base and after the shodhana pH was reduced in SA, it may be due to Nirvapa in Godugdha media which is having pH range of 6.4 to 6.8 and by repeated wash in Ushnajala. Once again in AP, pH was declined to weak acidic form; it might be influenced by Bhavana Dravya as all the four drugs were acidic in nature. Acc to pH - partition concept, the pH ranging from 2-5 is well absorb in stomach, from 7-8.5 well absorb in intestine and the pH ranging from 5-7 is absorb throughout GIT. By that we can conclude AP will well absorb throughout the GIT.

Total Ash Value

It is a physical method useful in drug standardization. Total ash value represents the inorganic salts which are naturally occurring in drug or adhering to it or deliberately added to it as a form of adulteration. Therefore it is a criteria to judge the identity or purity of sample. Here the test was carried out to detect the unburnt material and evaporative substance in final product. Total ash usually consists of carbohydrates, phosphates, silicates and silica.

In present study the total ash value of RA-99% and SA-99% has shown high value, as both containing purely silicon as whole compound which is confirmed by SEM-EDAX report, where as in AP drastic reduction of ash value was reported i.e. 48.50% which indicates the less un burnt part in AP. Hence it can be said that the prepared AP is within the standards of physical analysis of herbo mineral drugs.

Acid Insoluble Ash Value

Test for acid insoluble ash was carried out to evaluate the percentage of insoluble inorganic content of the samples in dilute acid. Since a drug must first pass into solution before it can be absorbed, so the acid insoluble ash test for drug is therapeutically very important. It is intended to provide a step towards the evaluation of the physiological availability of the Drug.

In present study Acid insoluble ash of the RA and SA is high i.e. 97.50%, 98.80% respectively it might be due to presence of high amount silicon in them, surprisingly AP has shown very minimal amount of acid insoluble ash i.e. 14.50% it shows high bioavailability of AP.

Water Soluble Ash Value

It indicates selective media of drug administration. In present study water soluble ash value of RA and SA are 2% and 0.5% as Akika is totally insoluble in water.
where as AP shown 33%, means the solubility in water is considerably increased after Pishtikarana denotes that water is a soluble media for it. Apart from this salivary secretions and gastric enzymes may play an important role in the efficacy of AP.

**Loss on Drying**

It is a physical test determines the amount of volatile matter. The moisture content of a drug should be minimized in order to prevent decomposition either due to chemical change or due to microbial contamination. The least loss on drying at 110°C the better will be the drug.

In the present study RA and SA has shown 0.15% and 0.50% of LOD at 110°C respectively whereas AP shown 1.90 %, the reason for marginal increase is might due to Bhavana in organic media (Kumari, Ketaki, Jaliapippali and Kadali Kanda Swarasa). Hence it can be stated that it possess low moisture content and concurrently it can be stated that the shelf life of AP prepared in the present study is more.

**Discussion on XRD**

X-Ray diffraction studies were conducted for 3 samples, namely RA, SA and AP with the aim of determining the structure and composition. The diffraction of X-ray is used in the study of the crystalline materials which produce diffraction. X-ray diffraction leads primarily to the identification of crystalline compound from their diffraction patterns. Phase and structure of the compound is studied after comparing the d-space value with d-standard peak values. The XRD pattern of all the samples showed peaks with sharp lines indicating the crystalinity of the samples.

Among all silica polymorphs, quartz is the only stable form at normal ambient conditions, and all other silica polymorphs of quartz change their structure according to temperature and pressure viz, α-quartz (quartz low) which is more stable below 573°C, and when temperature exceeds more than this it gets converted in to β-quartz i.e. α-quart and β-quartz are interchangeable forms of silica. In present study during nirvapa method processed high temperature lead to conversion of α-quart to β-quartz (quartz high). Hence in sample SA they obtained silica is quartz high. As this transformation is reversible process, some of the β-quartz has transferred to quartz after the bhavana procedure. This might be the reason for presence of both the forms of silica in AP.

**Discussion on SEM EDAX**

SEM-EDX study reveals the accurate elemental analysis of the sample, this study of elements enable us to explore Major, Minor and Trace elements.

In present study the percentage of elements in RA are 0-30.53%, Si-69.47% both are as per the pharmacopeial standards only hence it shows the genuinity of raw drug.

The sample of SA also shown same elements i.e. O-38.90% and 61.10%, here the percentage of oxygen was increased and silicon was decreased it might be due to oxidation reaction during Nirvapa procedure. In both the sample no other trace element were found.

In the sample AP major elements like C, O and Si are reported in the percentage of 30.19, 29.26 and 30.76 respectively. Minor elements like Na, Mg, Al, Cl ,K, Ca and Fe in the percentage of 0.50, 0.62, 1.12, 1.06, 1.76, 1.68 and 3.06 respectively.

**Carbon:** The probable source of carbon in AP is organic extract which are used during Bhavana.

**Silicon:** In AP, the percentage of silicon comparatively reduced than SA and even form has been changed which revealed by XRD it might be due to 21 times bhavana with organic compounds by which other trace elements like Na, Mg etc got imbibed in it.

**Other trace elements:** AP is prepared by Bhavana method processed in four organic extracts like Kumari Swarasa, Ketaki Pushpa Swarasa, Jalapippalli Swarasa and Kadali Kanda Swarasa. Basically plants sources are rich with many trace elements. viz,

- **Kumari** rich in - Na, Mg, Al, Cl and Ca
- **Ketaki pushpa** rich in - Ca, Fe
- Jalaippali - Mg, it is the source of Iron also as it is collected from Sandura. The place is famous for iron mining hence the soil contains rich amount of iron.
- Kadalikanda rich in - Mg, K
So these might be imbibed in AP during bhavana.

Particle size

The dosage of active drugs can be reduced without lowering the efficacy simply by reducing the particle size. Particle size is one of the factors which will affect dissolution and absorption of drug. Particle size and surface area are inversely proportional to each other, as particle size decreases surface area increases. This leads to increase in dissolution of drug and rapid absorption is measure of rate of solution. 

$$\frac{dm}{dt} = kA(C_s-C)$$

$$\frac{dm}{dt} =$$ dissolution rate, 
A = surface area of solid,  
k = dissolution rate constant,  
$$C_s =$$ saturation of drug,  
$$C =$$ concentration of drug in solution

The effective particle size of RA, SA and AP is 1604.5nm, 1539.1nm and 940.0nm respectively. The particle size of AP was drastically decreased in comparison to RA and SA. According to Rasacharyas the Pishi should be Bhasmavat, means nano like Bhasma and in present study AP is in nanometres which show higher bioavailability of AP.

CONCLUSION

Science and scientific reasoning is not pertaining to any particular branch of study or particular era. The ancient system of medicine, Ayurveda explains with perfect scientific background about the analysis of Pishti. Our Acharyas had explained about the quality and non toxic nature of Pishti in their own way. As discussed above each and every classical parameter has its own reasoning and importance. Using modern parameters and sophisticated instruments these Pishti can be analyzed for the same in a different way. Pishti Kalpana is one of the Kharaliya Kalpanas of Rasashastra which brings the heat sensitive substances at micro particle level without applying the heat for better therapeutic efficacy which is considered as Sukshma as Bhasma. This Kalpana is chiefly applied to the Ratnas and Uparatna. Here the aim is to induce the Sheeta Guna in them and also to preserve the original Gunas in them. By triturating the Ratnas with mentioned liquids for several hours particle size becomes micro fine so that it can assimilate in the human body. Akika Pishti was prepared by subjecting Kuttana to Bhavana for 21 times with different Bhavana media like Kumari Swaras, Ketaki Swaras, Jalaippali Swaras and Kadal Kanda Swaras. Average time taken for each Bhavana is around 7 hours 30 minutes. The analytical results shows that the Akika Pishti which is prepared as per classically explained method is safe, having nano particle size revealed by particle size and pH of 6.8 and hence more bio-available. XRD reveals that, there is change in compound from SiO2 to high quartz after the Pishitikarana and simultaneously the organic properties are imbedded to Pishi from Bhavana Dravyas which is well proved by SEM-EDAX.

REFERENCES


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