



ISSN 2456-3110

Vol 2 · Issue 3

May - June 2017

Journal of
**Ayurveda and Integrated
Medical Sciences**

www.jaims.in

JAIMS



Charaka
Publications

Indexed

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 a *Anagnisidhdha 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 pharmacopeial standards and subjected to both ancient and modern tests to analyze viz. *Pishtivarna*, *Mrudutva* and *Slakshanatva*, *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 *Anagnisidhdha 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.

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Submission Date : 25/05/2017 Accepted Date: 27/06/2017

Access this article online

Quick Response Code



Website: www.jaims.in

DOI: 10.21760/jaims.v2i3.8215

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^[3] and *Akika Pishti*^[4] 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 *Slakshanatva*, *Rekhapurnata*, *Varitara*, *Nirdhooma* tests according to classics and according to modern parameters - Organoleptic tests, physico chemical like LOD,^[5] pH,^[6] ash values,^[7] Instrumental analysis like XRD,^[8] SEM-EDAX^[9] and particle size.^[10]

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

SN	Parameters	RA	SA	AP
1	Varna	Shweta	Dhusara	Kapota
2	Chandrika	Present	Absent	Absent
3	Rekhapurnatva	Negative	Negative	Positive
4	Slakshnatva,	Negative	Negative	Positive
5	Mridutva	Negative	Negative	Positive
6	Varitara	Negative	Negative	Positive
7	Unama	Negative	Negative	Positive
8	Nirdhooma	Negative	Negative	Positive
9	Nischandratva	Negative	Negative	Positive

Organoleptic Characters

Table 2: Organoleptic characters of RA, SA and AP

Sample	Color	Odour	Taste	Taste
RA	Light grey	Odourless	Tasteless	Crystalline
SA	Greyish white	Odourless	Tasteless	Semi amorphous
AP	Dark grey	Odourless	Tasteless to salty	Amorphous

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

Contents	RA	SA	AP
pH Value	7.84	7.78	6.87
Ash Value	99.00%	99.00%	48.50%
Acid insoluble ash	97.50%	98.80%	14.50%

Water soluble ash	2.00%	0.5%	33.00%
Loss on drying at 110°C	0.15%	0.50%	1.90%



Fig. 1: Raw Akika



Fig. 2: Akika after Kuttana



Fig. 3: Akika Pishti



Fig. 4: Rekhapurnata Pariksha

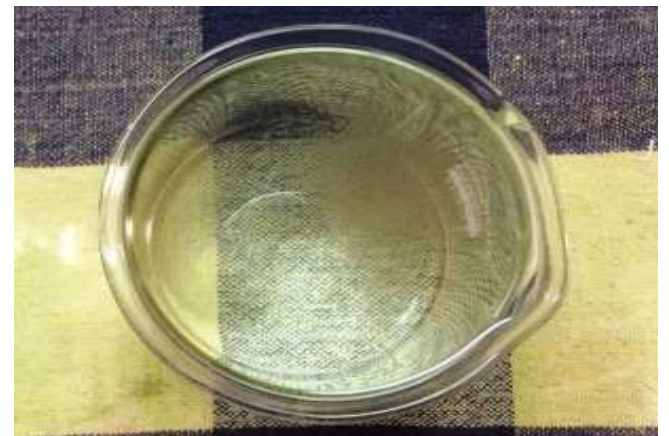


Fig. 5: Varitara Pariksha



Fig. 6: Unama Pariksha

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. 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

Sample	Compound Name	Chemical Formula	Crystal Structure
RA	Quartz low	SiO ₂	Hexagonal
SA	Silicon	Si	Cubic
	High quartz	SiO ₂	Hexagonal
AP	Gamma Fe	Fe	Cubic
	Quartz	SiO ₂	Hexagonal
	High quartz	SiO ₂	Hexagonal
	Silicon di-oxide	SiO ₂	Hexagonal

Fig. 7: XRD of RA

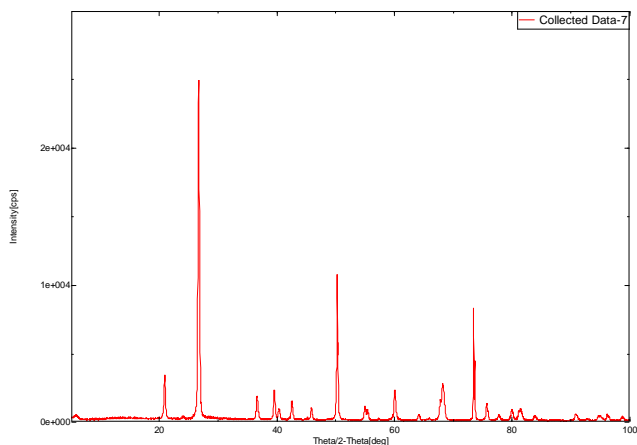


Fig. 8: XRD of SA

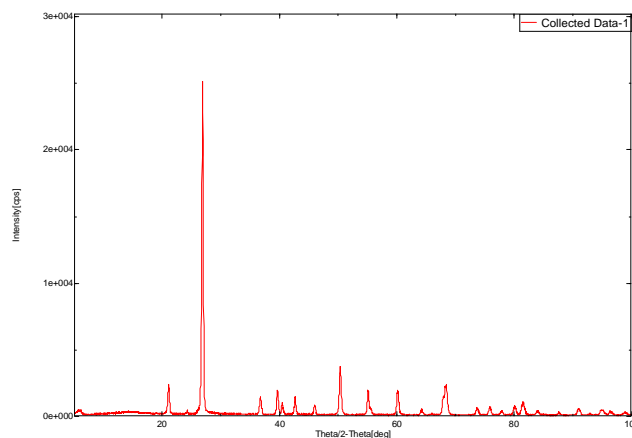
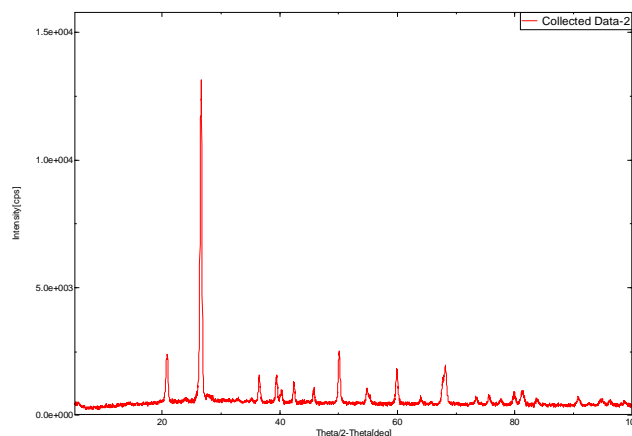


Fig. 9: XRD of AP



SEM-EDAX RESULT

Table 5: Comparative Results of SEM-EDX

RA			SA			AP		
Elements	Weight %	Atomic %	Elements	Weight %	Atomic %	Elements	Weight %	Atomic %
O	30.53	43.55	O	38.90	52.78	C	30.19	44.11
Si	69.47	56.45	Si	61.10	47.22	O	29.26	32.10
						Na	0.50	0.38
						Mg	0.62	0.4

								5
						Al	1.12	0.73
						Si	30.76	19.22
						Cl	1.06	0.52
						K	1.76	0.79
						Ca	1.68	0.73
						Fe	3.06	0.96

Fig. 10: SEM –EDAX of RA

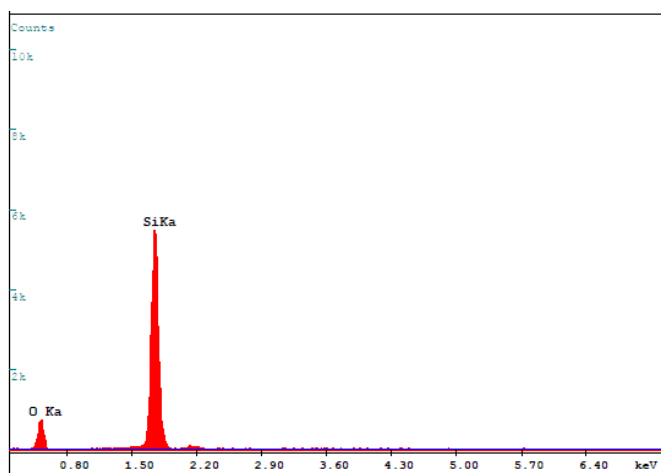


Fig. 11: SEM –EDAX OF SA

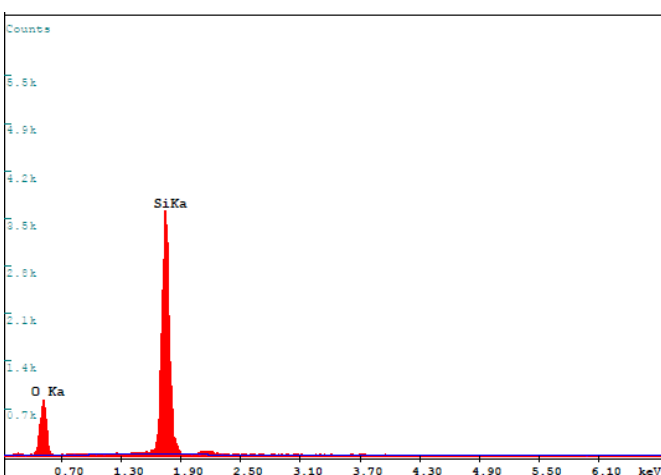


Fig. 12: SEM-EDAX OF AP

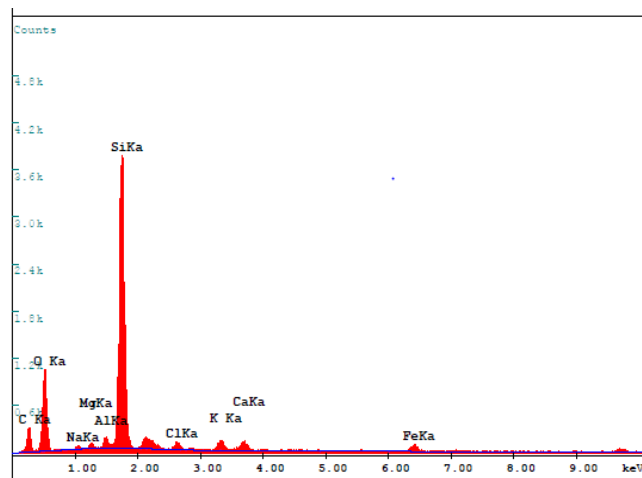


Table 6: Report of Particle Size of RA, SA, and AP

Sample	Particle size
RA	1604.6nm
SA	1539.1nm
AP	940.0nm

DISCUSSION

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*.

Unama 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 *Pishti*. 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 ashvalue 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 crystallinity 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 -quartz and β -quartz are interchangeable forms of silica. In present study, during nirvapa method processed high temperature

lead to conversion of α -quartz 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 O-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, Jalapippali 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

- *Jalapippali* - 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.

$dm/dt = kA(C_s - C)$

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 *Pishti* 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 Swarasa*, *Ketaki Swarasa*, *Jalapippali Swarasa* and *Kadali Kanda Swarasa*. 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 SiO_2 to high quartz after the *Pishtikarana* and simultaneously the organic properties are imbedded to *Pishti* from *Bhavana Dravyas* which is well proved by SEM-EDAX.

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How to cite this article: Vidyashree, Gowda Shankar, Doddamani M. S. Analytical assessment of *Akika Pishti* based on Ancient and Modern Parameters. J Ayurveda Integr Med Sci 2017;3:92-100.
<http://dx.doi.org/10.21760/jaims.v2i3.8215>

Source of Support: Nil, **Conflict of Interest:** None declared.
