Research Article

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A COMPARATIVE STUDY OF *KALYANAKA KSHARA* PREPARED BY *PUTA* METHOD AND FURNACE METHOD TO ASSESS ITS PHYSICOCHEMICAL PROPERTIES

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ABSTRACT

Ayurveda, the science of life is as old as the Vedic age and now a days *Ayurvedic* medicines are becoming popular day by day all across the world. The *Ayurvedic* therapeutics are mainly based on various kinds of dosage forms. *Panchavidha kashaya kalpana* is the basic form and many formulations are developed over time having long shelf life, stability, strong action etc. *Kshara kalpana* is the alkaline substance obtained from the ash of plants, minerals and animal products and has been widely used due to its minimal dose and potential action. *Kalyanaka Kshara* is a widely used formulation mentioned in our classics which is prepared by *Antardhooma vidhi*. In the present study *Kalyanaka Kshara* has been prepared using *puta* method as well as using muffle furnace. Physicochemical characterization of both the samples were done.

Keywords: *Antardhooma vidhi, Kshara, Kalyanaka kshara, Puta.*

INTRODUCTION

Ayurveda, the science of life is one of the most ancient and wide-ranging system in the Indian systems of health care. Here medicines are used either as single drugs or as formulations. Different forms of *Ayurvedic* formulations are *Kwathas, Churnas, Gutikas, Ghritas, Avalehas* etc. *Kshara Kalpana* is one among the dosage forms which is considered superior among surgical (*sastra)* and para-surgical (*anusastra)* measures[1]. It can be used both internally as well as externally.

Kalyanaka Kshara is a widely used formulation having a spectrum of indications. It is mentioned in *Sahasrayoga*[2] in the context of *Bhasmaksharadi yoga* and *Ashtanga sangraha*[3] and *Ashtanga hridaya*[4] in the context of *Arsho chikitsa*. This formulation contains *Shunthi, Maricha, Pippali, Haritaki, Vibhitaki, Amalaki, Saindhava, Sauvarchala, Bida, Danti, Aruskara, Chitraka* along with *gomutra* and *tila taila*. It is indicated in *Arsha, Gulma, Pandu, Udara, Mutrasanga, Ashmari, Prameha, Pleeha, Shwasa* and *Kasa.*

In our classics, *Puta* is explained as the degree or intensity of heat that is to applied for the proper *Paka* of *dravyas*[5]. In other words, the *Puta* indicates the quantitative as well as qualitative measure of heating. In the context of *Kalyanaka Kshara*, *acharyas* explained *Antardhooma vidhi* for its preparation. But, the *puta* and temperature pattern to be followed has not been mentioned. So, in the present era of globalization, to meet the international standards, Good Manufacturing processes are necessary[5]. It can provide Quality assurance and reproducibility of the drug. Hence Standardisation[6] of process is a mandatory step to keep up quality and efficacy of the product. In order to develop a Standard Operating Procedure for *Kalyanaka Kshara* preparation two different heat sources were used. Initially muffle furnace was opted for the study and after fixing the temperature the process was repeated using *Puta*. Physicochemical assessments like organoleptic characters, pH, loss on drying, acid insoluble ash, solubility along with volumetric evaluation, PSA, XRF etc. were done.

MATERIALS AND METHODS

Collection and authentication of raw drugs

The herbal drugs used in the formulation were procured from the local market of Trivandrum and were authenticated by the Quality control tests mentioned in *Ayurvedic* Pharmacopoeia of India. The QC parameters of single drugs include Total ash, Acid insoluble ash, Water soluble extractive, Alcohol soluble extractive[7]. The genuine samples of *Saindhava, Sauvarchala* and *Bida* were collected from the place of origin and *Gomutra* was collected afresh. Three samples of *Tila taila* were taken and genuine sample was taken after doing the quality assessing tests like Refractive index, Acid value, Saponification value, Iodine value.

Preparation of *Kalyanaka kshara*

Shodhana of *Chitraka*[8] and *Bhallataka*[9] were done as per classical methods. *Chitraka Shodhana* done using *Choornodaka*. Three batches of *Kalyanaka Kshara* were prepared using *Puta* method. The ingredients *Triphala, Trikatu, Tripatu, Danti, Shodhita Bhallataka* and *Shodhita Chitraka* were taken in equal amount, coarsely powdered and mixed uniformly. *Tila taila* and *Gomutra* were added as required to form a homogenous mixture. Finally, *Trilavana* were added to the above mixture and the whole mixture was transferred into a *sarava* and closed with another *sarava*, *sandhibandhana* done for 7 times.

As specified *puta* was not mentioned in the classical references regarding *Kalyanaka Kshara,* the method initially adopted was using muffle furnace at a temperature of 700 degree Celsius and maintained for 1 hour, then allowed for *swaangaseeta* and the product was collected in the next day. It was finely powdered and

done physicochemical characterization. As the pH of the sample obtained was 3.8, the procedure was repeated again with a temperature of 450 degree Celsius. The pH of the sample obtained was 7.8. As alkaline pH was obtained at 450 degree, the peak temperature for the classical *puta* method was set as 500 degree Celsius.

***Puta* method**

A pit having the dimension of 45 cm was opted. Initially 300 cow dung cakes each weighing 12 gm were taken for the study. 200 cow dung cakes were kept then the sarava was placed, then the remaining 100 cakes above it and ignited. The whole process took about 5-6 hours for completion. After self-cooling the product was collected, powdered and analyzed.

To Standardize the *puta* method, two more times the same procedure was repeated and the temperature pattern was noted. During the second *puta* the cow dungs having weight of 48 gm was obtained (4 times of the initial one). Hence only 80 cow dung cakes were taken (approximately one –fourth of the initial cakes). Both the times the peak temperature attained was about 516-520 degree Celsius. Physicochemical characterization of the final products was done. The final products were named as KK1, KK2 and KK3.

Both the *puta* sample and furnace sample were subjected for analytical parameters mentioned for *kshara* in API. Along with these parameters AAS, XRF, PSA and Volumetric tests[11] were done.

Atomic Absorption Spectroscopy (AAS) is a Spectro analytical procedure for the quantitative determination of chemical elements using the absorption of optic radiation by free atoms in the gaseous state. AAS is based on absorption of light by free metal ions. The atoms absorb ultraviolet radiation or visible light and make transitions to higher electronic energy levels. The analyte concentration is determined from the amount of absorption. Concentration measurements are usually determined from a working curve after calibrating the instrument with standards of known concentration. It is a very common technique for detecting metals and metalloids in environmental samples.

X-ray fluorescence (XRF) is a powerful imaging technique for the quantitative mapping of distributions and dynamics of elements and chemical species at the spatial sub micrometer resolution within biological samples. Upon excitation by an X-ray photon, a core shell electron from the specified atom is ejected as a photoelectron. The formed core-hole is then filled by a neighboring high energy orbital electron, which results in the emission of an X-ray photon. The energy of the emitted photon is equal to the difference in binding energies of the two shells involved in the transition. Since the binding energy is varied with the nuclear charge, each element has a unique photon energy, i.e., characteristic fingerprint x-ray fluorescence, which enables the multielement analysis.

Regarding the measurement of size of a nanoparticle, a number of techniques have been developed that operate under various principles. Microscopy techniques such as SEM, TEM, Atomic force microscopy, scanning transmission electron microscopy, focused ion beam SEM cannot provide any data about the properties of NP when it is in solution. Techniques such as Taylor Dispersion Analysis (TDA), Dynamic Light Scattering (DLS), Nanoparticle Tracking Analysis (NTA) can be utilized in these situations. It is important to note as the bio distribution and biological responses of a material can be correlated to its dispersion state and solubility. The ability to characterize a nanomaterial sample in a solution is of paramount importance where the hydrodynamic diameter is the most widely accepted critical quality attribute. Particle distribution ('D' value) parameters are also becoming increasingly requested. D values set at 10%, 50% and 90% provide valuable statistical distribution insight in the broadness of particle size range. The distribution from NTA is number based, compared to light scattering intensity for DLS.

RESULT

Organoleptic evaluation

Table 1: Organoleptic characters

Odour	Faint
Colour	Blackish
Taste	Salty
Touch	Fine

Physicochemical evaluation

Table 2: Physicochemical characters

	At 600^{0}C	**At 450^{0}C**
pH	3.8	7.8
Acid insoluble ash	1.2%	0.84%
Loss on drying	5%	3.8%

Physicochemical characteristics of *Puta* samples

pH

Table 3: pH of *Puta* samples

Sample	**pH**
KK1	10.56
KK2	10.7
KK3	10.83

Acid Insoluble Ash

Table 4: Acid insoluble ash

Sample	**Acid insoluble ash**
KK1	0.98%
KK2	0.84%
KK3	0.92%

Loss on Drying

Table 5: Loss on drying

Sample	**Loss on drying**
KK1	3.40%
KK2	4.10%
KK3	3.60%

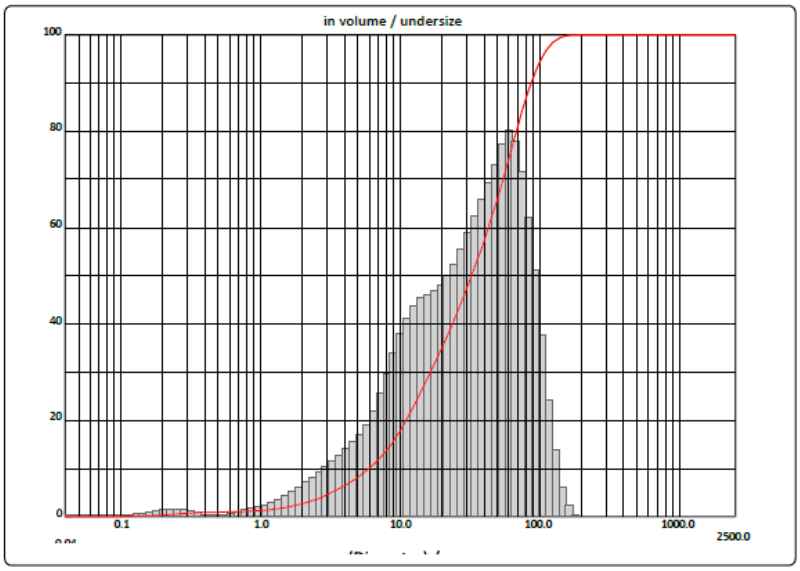
XRF analysis

Table 6: XRF of Samples

Sample	KK_1	KK_2	KK_3	Furnace Sample
SiO_2	0.98%	0.94%	0.94%	0.95%
TiO_2	358 ppm	350 ppm	354 ppm	398.7 ppm
Al_2O_3	0.50%	0.45%	0.46%	0.505%
MnO	661.6 ppm	660.1 ppm	662 ppm	573.7 ppm
Fe_2O_3	0.33%	0.32%	0.33%	0.359%
CaO	11.46%	11.43%	11.46%	11.03%
MgO	1.36%	1.35%	1.36%	1.59%
Na_2O	15.62%	16.02%	15.66%	15.31%
K_2O	12.74%	12.66%	12.74%	10.76%
P_2O_5	2.22%	2.22%	2.23%	2.12%
Cl	48.34%	49.01%	48.44%	40.91%
SO_3	6.22%	6.23%	6.25%	6.231
CuO	88.4 ppm	88.2 ppm	88.3 ppm	83 ppm
V_2O_5	0 ppm	0 ppm	0 ppm	0 ppm
ZnO	127.9 ppm	126.5 ppm	127.8 ppm	148.2 ppm
Br	219.1 ppm	219.1 ppm	219 ppm	206.4 ppm
Rb_2O	119.1 ppm	190.2 ppm	193.2 ppm	121.5 ppm
SrO	540.5 ppm	540.2 ppm	540.3 ppm	512.3 ppm
ZrO_2	20.3 ppm	21.3 ppm	20.4 ppm	62.5 ppm
BaO	194.1 ppm	194.3 ppm	193.2 ppm	174.3 ppm
Eu_2O_3	3.7 ppm	3.5 ppm	3.6 ppm	28.6 ppm
Re	3.9 ppm	3.9 ppm	3.2 ppm	2.7 ppm
Nd_2O_3	0 ppm	0 ppm	0 ppm	0 ppm

Particle size of furnace sample

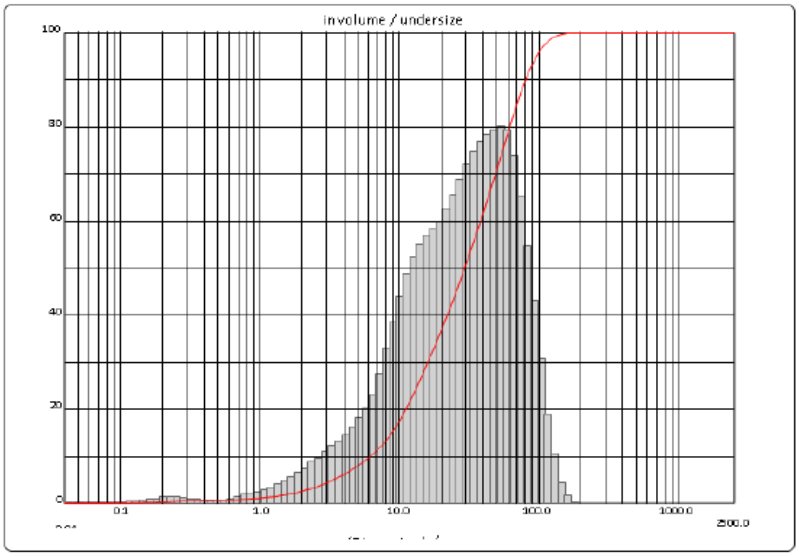
Sample ref. Sample	: PS 1	Ultrasounds	: 60 s
Name Sample type	: Ayurveda	Obscuration	: 8 / 0.40 %
	: Ash (Furnace)	Diameter at 10%	: 5.96 µm
Comments	: Ayurvdea Tvm, 0.069 g	Diameter at 50%	: 32.63 µm
Liquid	:	Diameter at 90%	: 87.20 µm
Dispersing agent	: calgon	Mean diameter	: 40.57 µm
Operator	: cess	Fraunhofer	
Company	: NCESS	Density/Factor	----------
Location	: Trivandrum	Specific surface	----------
Date : 14-03-2018	Time : 02:39:01PM	Automatic dilution	: No / No
Index meas.	: 2175	Meas./Rins.	: 60s/60s/4
Database name	: Granulog	SOP name	: CESS



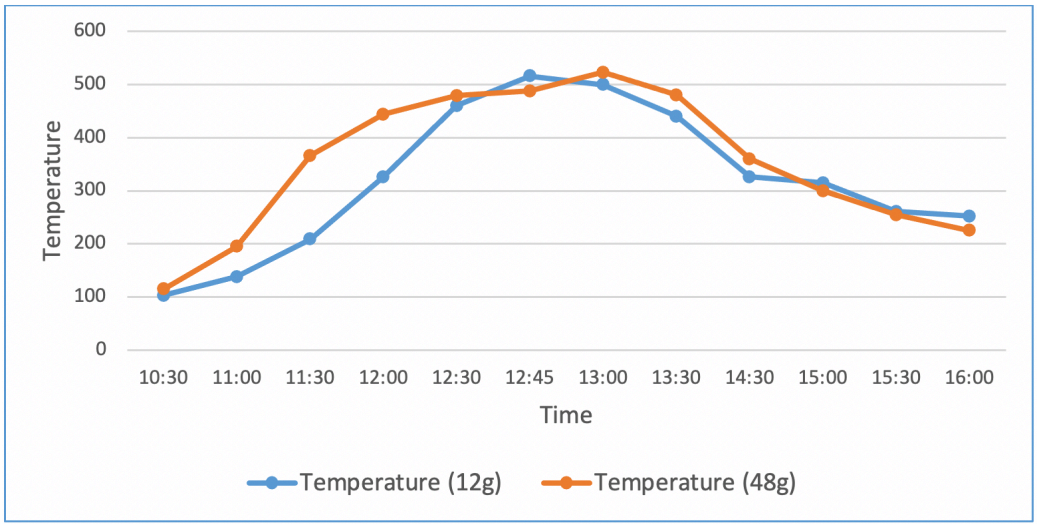
Graph 1: PSA of Furnace sample

Particle size of puta sample

Sample ref.	: PS 2	Ultrasounds	: 60 s
Sample Name	: Ayurveda	Obscuration	: 14 / 0.62 %
Sample type	: Ash (Putam)	Diameter at 10%	: 6.16 µm
Comments	: Ayurvdea Tvm, 0.1434 g	Diameter at 50%	: 29.45 µm
Liquid	: Water (eau)	Diameter at 90%	: 80.41 µm
Dispersing agent	: calgon	Mean diameter Fraunhofer	: 37.38 µm
Operator	: cess	Density/Factor	----------
Company	: NCESS	Specific surface	----------
Location	: Trivandrum	Automatic dilution	: No / No
Date : 14-03-2018	Time : 02:54:40PM	Meas./Rins.	: 60s/60s/4
Index meas.	: 2176	SOP name	: CESS
Database name	: Granulog		



Graph 2: PSA of *Puta* sample



Graph 3: Comparison of temperature pattern using cow dung cakes weighing 12 gm and 48 gm

Figure 1: Preparation of *Kalyanaka Kshara*

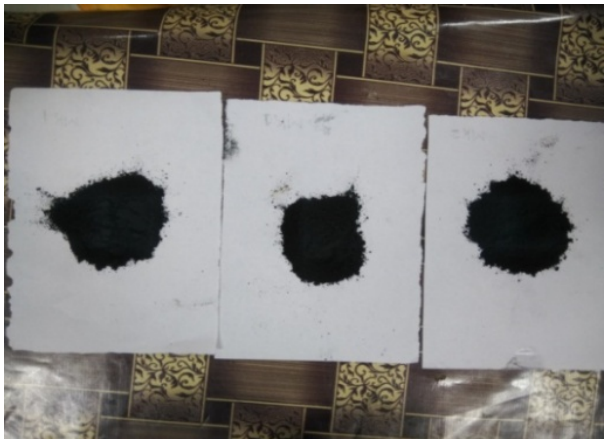


Figure 2: Prepared samples of Kalyanaka Kshara (Puta)

DISCUSSION

As there is no pharmaceutical literature available so far regarding the preparation of *Kalyanaka kshara*, initially an attempt has been made to fix the temperature. After doing the physicochemical characterization of *Kalyanaka kshara* prepared by furnace and *puta* method, they differ in respect of Ph and particle size analysis.

Ph value of a given sample expresses the degree of acidity or alkalinity of the sample. Alkalinity of the drug indicates the site of absorption and action of the drug. Initially at the temperature of 700 degree Celsius in muffle furnace, the Ph of the sample was 3.8. This may be due to the increased heat and the usage of *Ashodhita Bhallataka* for the first preparation. When the temperature was reduced to 450 degree and *Shodhana* of *Bhallataka* was done, the pH was noted as 7.8, which comes under the alkaline range. In *Puta* method Ph was observed as 10.56, 10.7 and 10.83 respectively.

Acid insoluble ash value helps in detecting the presence of silica and oxalates in the drugs. This test is designed to measure the amount of ash insoluble in dilute HCl. As per API, the Acid Insoluble Ash of *Kalyanaka kshara* is told to be less than 1%. In the *Kshara* prepared using furnace, the values are noted as 1.2 (at 600 degree) and 0.84 (at 450 degree). In the samples prepared by *Puta* method the values are 0.98%, 0.84% and 0.92%.

Loss on drying is widely used test method to determine the moisture content of the sample, although occasionally it may refer to the loss of volatile matter from the sample. It is an important parameter to be assessed for *Ksharas*, as it is hygroscopic in nature due to the presence of alkaline compounds. Lesser the LOD value more stable the *Kshara* is. As per API, the LOD value is told to be less than 6%. Here all the samples were having LOD values within normal limits.

Qualitative analysis of Anions and Cations were done using volumetric tests. In all the prepared samples there were more proportion of sodium and chloride ions. Calcium and Potassium were also present, but magnesium in traces only. Sulphates were detected in more proportion in the product prepared in classical *puta* method. Carbonates and phosphates were present in both samples.

In XRF analysis, sodium, potassium and calcium were present in more amount in the *Kshara* prepared in *puta* method. There were also traces of rubidium, strontium, zirconium, rhenium and europium in both the samples.

When the particle size of a drug is decreased, its larger surface area allows the increase in surface area to volume ratio thus increasing the surface area available for solvation. Hence the reduced particle size enhances the bioavailability of the drug. Here it was found that *Kshara* prepared in *Puta* is having particle size with mean diameter of 37.38 microns and that of furnace sample with a mean diameter of 40.57 microns.

CONCLUSION

From the above analytical studies, it was found that the *Kalyanaka kshara* prepared in *Puta* method differs in some aspects compared to the furnace sample. The main findings are the differences in pH and the PSA values. As *Ksharas* are preparations having alkaline pH, *Puta* can be considered as a standard procedure for the preparation due to the alkaline pH range obtained by this. Particle size determines the bio availability, hence lesser the particle size more available the drug

will be. As *Ksharas* are dosage forms having minimal dose they should be having lesser PSA values. Considering these facts, we can conclude that the classical method is a better method for *Kalyanaka kshara* preparation.

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