

# Pharmaceutical standardization of *Apamarga kshara*

Hasmukh R. Jadav<sup>1</sup>, R. Galib<sup>1</sup>, Pradeep Kumar Prajapati<sup>1</sup>

<sup>1</sup>Department of Rasa Shastra and Bhaisajya Kalpana, Institute for Post Graduate Teaching and Research in Ayurveda, Gujarat Ayurved University, Jamnagar, Gujarat, India

## ABSTRACT

Standardization of herbal drugs is essential to certify their quality and purity. *Kshara* (alkaline substance) of *Apamarga* (*Achyranthes aspera* Linn.) is an important constituent in many Ayurvedic formulations, but its standard manufacturing process (SMP) is not attempted till date. This study is aimed to establish SMP for *Apamarga kshara*. In pharmaceutical process; generally the sediments of ash obtained at the end of washes will be discarded. However, in the study, we attempted to wash the sediments repeatedly by adding water to extract more *Kshara*. *Apamarga* was collected from the local area and authenticated. *Kshara* was prepared by following standard methods and the preliminary physicochemical profile was developed. It is observed that the ash yields *Kshara* even in the consecutive washes. First wash yielded 21.23% w/w *Kshara*, while the second and third washes yielded 9.38% w/w and 4.76% w/w, respectively. Repeated washes yield more *Kshara*. Hence, it is advocated to wash the ashes repeatedly. As the findings are encouraging, similar experiments can be extended to all other *Kshara* preparations.

**Key words:** *Achyranthes aspera*, *Apamarga*, *Apamarga kshara*, standardization

## INTRODUCTION

In the present era, plant-derived products are gaining importance as medicinal products, nutraceuticals and cosmetics.<sup>[1,2]</sup> Herbal medicines are widely used in health-care in both developed and developing countries. According to an estimate of the World Health Organization, about 80% of the world population still uses herbs and other traditional medicines for their primary health care needs.<sup>[3]</sup> The use of herbal medicines has increased remarkably in line with the global trend of people returning to natural therapies.<sup>[4]</sup> Ayurveda utilizes different forms of herbs in therapeutics. *Kshara* is one among such forms.

*Ksharas* are alkaline substances obtained from the water soluble ashes of herbal drugs. Several *Ksharas* have been explained in Ayurveda and *Apamarga kshara* is one among them. Different opinions exist regarding specifications for the nature of vessels, the proportion of water, time for settling, cloth folding, etc. Regarding method of preparation in *Susruta Samhita*,<sup>[5]</sup> *Sharngadhara Samhita*,<sup>[6]</sup> *Rasatarangini*,<sup>[7]</sup> *Dravyaguna Vigyana*<sup>[8]</sup> and *Ayurveda Sara Samgraha*<sup>[9]</sup> were widely described. However, none of the classics has mention about the repetition of washings. Considering this, it has been attempted to subject the ashes for repeated washings and prepare *Kshara* and develop preliminary physicochemical profile of *A. Kshara*.

## MATERIALS AND METHODS

### Collection of *Apamarga panchanga*

Fresh *Apamarga panchanga* (whole plant of *Achyranthes aspera*) was collected during October to November 2012. Authentication of *Apamarga* was done on the basis of pharmacognostical characters.<sup>[10,11]</sup>

### Address for correspondence:

Dr. Hasumukh R. Jadav,  
Institute for Post Graduate Teaching and Research in Ayurveda,  
Gujarat Ayurved University, Jamnagar, Gujarat, India.  
E-mail: hasmukhjadav005@gmail.com

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### Vessel for *Kshara nirmana*

Generally *Ksharajala* is prepared by using cylindrical earthen, steel or plastic vessel. Collecting supernatant layer of *Ksharajala* from these vessels is inconvenient and difficult; hence, a new vessel has been designed for this purpose.

Cylindrical steel vessel of 10 L capacity with 23.9 cm of depth and 22.2 cm diameter was taken. After few trials; an outlet was made in the wall of the vessel about 3.2 cm above from the bottom that allows draining of clear liquids leaving the sediment in the container [Figure 1].

### Preparation of *Apamarga kshara*

*Apamarga kshara* has been prepared by following classical methods.<sup>[7]</sup> Whole process was divided into three phases.

#### Preparation of ash

Matured *Apamarga Panchanga* was collected and dried completely in sunlight for 8 days. After removing the physical impurities; dried *Panchanga* was burnt completely by placing it in a big iron pan. After the self-cooling, white ashes were collected [Figure 1].

#### Preparation of *Ksharajala*

One part of ash was collected in a specially designed steel vessel and 4 times of water was added to it. The contents were mashed thoroughly with hands and left undisturbed for 3 h. After that, the clear supernatant liquid layers were collected through the outlet and filtered through three layered cotton cloth. The residual ash was again mashed with 6 L of potable water and kept undisturbed for the

next 3h, followed by a collection of the second filtrate. A similar method was followed for the 3<sup>rd</sup> time to collect third filtrate [Figure 2].

#### Preparation of *Kshara*

All the three filtrates (of *Ksharajala*) were individually subjected to heat to evaporate the water content and to obtain *Kshara*, and by following this method; *Kshara* was prepared in five batches [Figure 2].

#### Physicochemical parameters

Preliminary physicochemical parameters like loss on drying at 110°C,<sup>[12]</sup> ash value,<sup>[13]</sup> acid insoluble ash,<sup>[13]</sup> pH value,<sup>[14]</sup> water soluble extractives<sup>[12]</sup> were carried out at pharmaceutical chemistry laboratory, Institute for Post Graduate Teaching and Research in Ayurveda (IPGT & RA), Jamnagar.

#### Atomic emission spectroscopy with inductively-coupled plasma

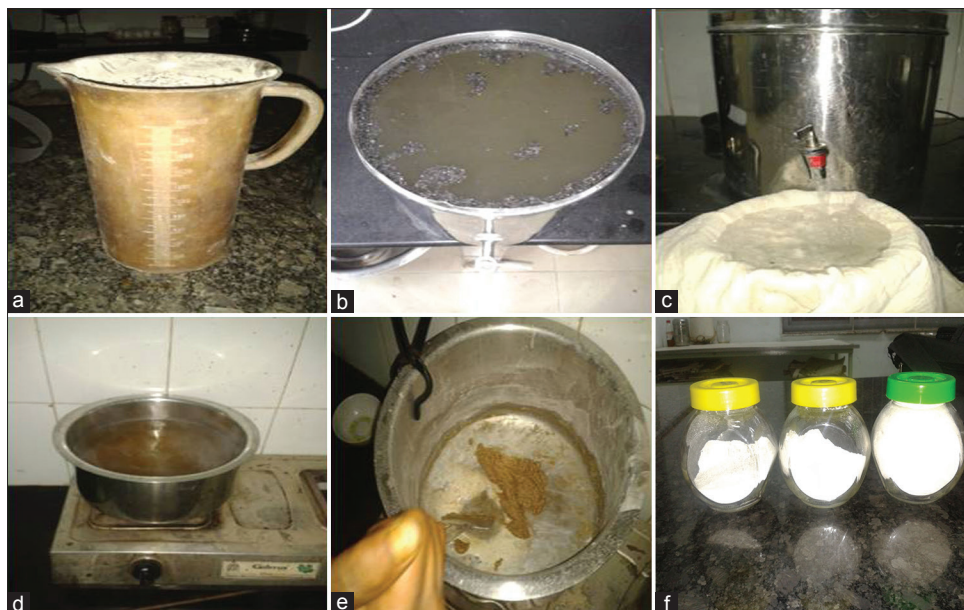
Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) is one of the most common techniques for elemental analysis and useful for standardization as well as to develop an analytical profile. In this technique, all samples of the trial drug were analyzed for ten elements.

#### Estimation of sodium and potassium ions by flame photometer

Estimation of sodium and potassium ions by flame photometer was carried out at the analytical laboratory.



**Figure 1:** Collection of *Apamarga*, preparation of *Apamarga* ash and vessel for *Kshara nirmana*. (a) A fully matured fresh *Apamarga* plant. (b) Drying of *Apamarga Panchanga* under sunlight. (c) Ignited of dried *Panchanga* in a big iron pan. (d) Completely burnt *Panchanga*. (e) Collection of prepared ash. (f) Specially designed vessel for *Kshara* preparation



**Figure 2:** Preparation of *Apamarga Kshara*. (a) Measured *Apamarga* ash. (b) Ash dissolved in water. (c) Filtration of *Ksharajala*. (d) Evaporation of *Ksharajala*. (e) Last stage of *Kshara* Preparation. (f) Prepared *Kshara* stored in air tight glass bottle

### OBSERVATION AND RESULTS

*Apamarga panchanga* burnt quickly and easily as it was completely dried. Comparatively, seeds took more time to burn. The powder of ash thus obtained was whitish with a characteristic taste. 59.81% Weight loss was observed after drying of *Apamarga Panchanga* and total 11.90% ash was obtained from dried *Panchanga* [Table 1]. After addition of water, the contents tasted salty, had characteristic odor and were yellowish in color. Total time required for preparation of *Ksharajala* was 4 h for a single wash. On an average, 76% *Ksharajala* was obtained after the wash [Table 2]. On evaporation of moisture on heat, the yellowish liquid was turned to brownish semisolid mass with aggregation and creaking sounds. The first wash yielded an average of 21.23% *Kshara*, while second and third washes yielded 9.38% and 4.76% respectively [Table 2]. This implies that the sediments at the end of first wash need not to be discarded and should be washed repeatedly. While attempts of further washings were failed to yield significant *Kshara*, hence after the third wash, the sediments were discarded.

Physicochemical parameters of *Apamarga Kshara*, standard given in Ayurvedic Pharmacopeia of India,<sup>[15]</sup> Estimation of sodium and potassium ions<sup>[15]</sup> and results of AES-ICP were given in Table 3.

### DISCUSSION

In the present study, preparation of *Aparmarga Kshara* has been carried out as per reference of *Rasa Tarangini*.<sup>[7]</sup>

**Table 1: Observations and results obtained during preparation of *Apamarga* ash**

Observations	Results
Weight of fresh <i>Apamarga Panchanga</i>	53.49 kg
Weight of dried <i>Apamarga Panchanga</i>	21.50 kg
Weight loss of <i>Apamarga Panchanga</i> after drying	31.99 kg
Percentage of loss after drying	59.81% w/w
Weight of ash obtained	2.559 kg
Percentage of ash obtained from dried <i>Panchanga</i>	11.90 w/w
Percentage of ash obtained from fresh <i>Panchanga</i>	4.78 w/w

**Table 2: Results of *Apamarga Kshara* obtained in different washes (average of five batches)**

Stages	First wash	Second wash	Third wash	Total of three washes
<i>Ksharajala</i> (L)	6.01	6.1	6.12	18.23
Percentage of <i>Ksharajala</i>	75.13	76.45	76.50	76.03
<i>Kshara</i> (% w/w)	21.23	9.38	4.76	35.38
<i>Kshara</i> (% v/v)	9.13	4.1	1.97	15.2

Dried *Panchanga* should be made into small pieces for better drying. The plant should be burnt in a vessel to prevent contamination during burning. For proper burning, *Panchanga* should be added little by little into the fire. Ash and water were taken volumetrically by keeping constant weight for *Ksharajala* preparation. De-mineralized water was used to avoid any interference of inorganic salts present in tap water. Stainless steel or a suitable nonreactive vessel should be used to prevent possible chemical reactions.

Ash should be rubbed well in water for proper mixing and allowed to settle down for at least 3 h. *Ksharajala* is to be obtained very cautiously through the outlet without



**Table 3: Comparative physicochemical parameters and ICP-AES analysis of all samples**

Parameter	APK1	APK2	APK3	API
Loss on drying at 110°C (% w/w)	1.30	1.50	1.40	Not more than 4%
Ash value (% w/w)	94.65	95.30	95.10	-
Acid insoluble ash (% w/w)	0.88	0.94	0.92	Not more than 1%
Solubility in water (% w/w)	97.26	96.72	97.35	-
pH value 10 aqueous solution (% w/w)	10.61	11.12	10.72	10-11
Sodium ions by flame photometer (%)	2.07	5.12	6.19	Not <4%
Potassium ions by flame photometer (%)	38.24	47.49	66.66	Not <29%
Arsenic (As) by ICP-AES	0.205	0.401	0.393	-
Lead (Pb) by ICP-AES	ND	ND	ND	-
Mercury (Hg) by ICP-AES	ND	ND	ND	-
Cadmium (Cd) by ICP-AES	ND	ND	ND	-
Silica (Si) by ICP-AES	116.349	110.614	105.238	-
Iron (Fe) by ICP-AES	0.036	0.038	0.571	-
Sodium (Na) by ICP-AES	>1737.26	>1982.50	>2025.64	Not <4%
Potassium (K) by ICP-AES	>1334.11	>1492.80	>1528.00	Not <29%
Calcium (Ca) by ICP-AES	7.569	12.383	15.236	-
Magnesium (Mg) by ICP-AES	1.833	0.493	6.331	-

ICP-AES: Inductively coupled plasma-atomic emission spectrometry, APK1: *Apamarga Kshara* after first wash, APK2: *Apamarga Kshara* after second wash, APK3: *Apamarga Kshara* after third wash, API: Active pharmaceutical ingredient, ND: Not detected

disturbing the vessel. Measures should be taken to avoid the entry of sediments. A clean cotton cloth should be used for this purpose. *Acharyas* also specified to filter the contents through multi-folded cloth to avoid sediments in the filtrate.

Initially, *Ksharajala* was yellowish colored clear liquid. Aggregation, vapors and crackling sounds were increased proportionally with temperature. Color was changed from yellowish to brownish gradually as the temperature raised. *Kshara* was started sticking to the vessel in final stages, and bumping was observed. It was stirred carefully to prevent bumping and sticking at this stage. Finally, white colored *Kshara* was obtained.

*Kshara* is considered as a water soluble ash, but all water soluble content cannot be obtained within a single wash; some of them may remain as residue. Considering this; total three washes were done to get maximum yield. *Kshara* obtained in the first wash was 21.23%, while at the end of the third wash the total *Kshara* obtained was 35.38% [Table 2]. Thus, current method is better and yielded more in comparison to commercial method used in pharmacy.

Organoleptic characters of APK1 (*Apamarga Kshara* after first wash), APK2 (*Apamarga Kshara* after second wash) and APK3 (*Apamarga Kshara* after third wash) like slimy touch, white color, salty taste, characteristic odor and fine powder appearance. Material absorbs moisture during the storage. In conjunction with a suitable temperature, moisture will lead to the activation of enzymes and given suitable condition to the proliferation of living organism. Hence, moisture contents may affect the quality of the drug. Although the weight loss in the samples is principally

due to water, small amount of other volatile materials will also contribute to the weight loss. There was considerable difference observed in a loss on drying in APK1, APK2 and APK3 [Table 3].

Total ash is important and indicates to some extent the amount of care taken in the preparation of the drug. In the determination of total ash value, the carbon must be removed at as low temperature (450°C) as possible because alkali chlorides, which may be volatile at high temperature, would otherwise be lost. The pH value of a given sample expresses the degree of acidity or alkalinity of a sample solution. APK1, APK2, APK3 samples have pH 10.61, 11.12 and 10.72, respectively. The alkalinity of the drug indicates the site of absorption and action of the drug [Table 3].

Analysis of *Ayurvedic* medicines reveals a great deal about their elemental composition. ICP-AES analysis of all the samples revealed that sodium, potassium, calcium and magnesium are the main constituent of all samples may be due to *Kshara* present in all samples. Heavy metal like arsenic, lead, mercury and cadmium were not detected or within permissible limits. Silica and iron showed around same results in all sample of trial drugs. Silica was present in all samples. In the present study, it was observed that sodium, potassium, calcium and magnesium constantly increased in APK1, APK2, and APK3 [Table 3].

## CONCLUSION

The residues after a first wash should never be discarded; they are to be processed further twice to obtain more

*Kshara*. After three washes, maximum 35.38% yield was obtained. The current observations can be considered as a lead for future studies. The dimensions of a newly designed vessel are specific to the current batch size that will vary according to the quantity of raw material.

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### Conflicts of interest

There are no conflicts of interest.

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