Research Article

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PHYSICO-CHEMICAL ANALYSIS OF KR̥ṢṆATILA KṢĀRA

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ABSTRACT

Genuine medicine is an important aspect of treatment. The success of the treatment relies highly on a properly prepared medicine. Manufacturing procedures and parameters for evaluating the quality of the preparations have been mentioned in various Ayurvedic texts. In this era of commercialization when the need of large scale production is very high, the standardization of the product is essential to assure its quality. Therefore, after the manufacture, the physico-chemical analysis of the product helps in the standardization of the product. The aim of the present research work was to prepare kshara (alkali preparation) from the drug Krishnatila (*Sesamum indicum* Linn.) and perform its physico-chemical analysis. Krishnatila (*Sesamum indicum* Linn.) is a drug which has tremendous capacity in inducing menstrual bleeding and is used in treating diseases like Artavakshaya (Oligomnorrhea and Hypomenorrhea). It is usually made as kashaya or kalka and consumed. Kshara kalpana is the process of extraction of kshara (alkali) from the ash of dried plants. Kshara helps in curing diseases that are difficult to treat and also reduces complications and recurrence. It is considered superior among the surgical and para-surgical measures in Ayurveda. Kshara has minimal dose and more shelf life. In the present study, the analysis of kshara was done by employing various physico-chemical parameters as, per the Ayurvedic Pharmacopoeia of India, for quality assurance and standardization. The kshara can be thus used for further clinical study.

Keywords: Kr̥ṣṇatila, kṣāra, pañcāṅga

INTRODUCTION

Every drug in the universe is considered as a medicine. There are many factors which determine whether to use the drug as such or to combine it with other drugs and convert it into a formulation. Samskara or processing always brings about changes in the guna (quality) of the drug. Rasashastra, the pharmaceutical branch of Ayurveda deals with the conversion of the natural resources like herbs, metals and minerals into medicines. There have been drastic changes from time to time in the different kalpana (preparations). Many new dosage forms have been invented according to the need of time. In the present era, people are accustomed to using everything in 'readymade' form; even medicines.

Kṣāra kalpana is the process of extraction of Kṣāra (alkali) from the ash of dried plants. It is considered superior among the surgical (śāstra) and para-surgical (anuśāstra) measures. Kṣāra helps in curing diseases that are difficult to treat and also reduces complications and recurrence. It can be used both internally and externally. In the present study, an effort has been made in converting Kr̥ṣṇatila (*Sesamum indicum* Linn.), a drug which is very effective in inducing menstruation and which is commonly used in the form of kashaya, or kalka, into kṣāra (alkali), thereby making it easy to consume, reducing the dose and increasing the shelf life. The alkaline nature of kṣāra along with the basic qualities of the drug used performs the therapeutic action.

It was prepared in the laboratory attached to the Department of Rasashastra and Bhaishajya kalpana, Amrita school of Ayurveda, Vallickavu, Kollam, Kerala. The analytical study was carried out in the Quality Control laboratory attached to the Department of Rasashastra and Bhaishajya Kalpana of Amrita school of Ayurveda, Vallickavu, Kollam, Kerala and CARE Keralam, Trissur.

Aim of the study

- To collect genuine raw material for the pharmaceutical preparation i.e. Kr̥ṣṇatila pañcāṅga or the whole plant.
- To prepare Kr̥ṣṇatila kṣāra as per reference from Rasatarangini¹ by maintaining standard operative procedures.
- To perform the physico-chemical analysis of Kr̥ṣṇatila Kṣāra.

MATERIALS AND METHODS

Collection of Raw material

The plant Kr̥ṣṇatila was cultivated for the study. The seeds of Kr̥ṣṇatila were purchased from an authentic source. These seeds were sown in a field in Chirakkara locality of Kollam district and the plant was cultivated for the purpose of this study. After attaining full maturity, the plant was collected as a whole, completely dried and preserved. It was authenticated by the Department of Dravyaguṇavijñānam, Amrita School of Ayurveda, Vallickavu, Kollam, Kerala.

Preparation of Kṛṣṇatila kṣāra

The preparation of Kṛṣṇatila kṣāra was done according to the reference from Rasatarangini¹.

The preparation was in two steps

Preparation of Ash

Preparation of Kṣāra

Preparation of Ash

Materials Required: Weighing balance, lightener or match box, sieve or mesh

Raw material used: Dried Kṛṣṇatila pancāṅga (whole plant)

Procedure

The preparation of ash is an oxidative process and has to be done in open air. A small amount of the dried pancāṅga (whole plant) was made into a heap in the centre of cleaned courtyard. It was then ignited. After it completely caught fire and burned, small amounts of dried pancāṅga (whole plant) was added to it until the whole pancāṅga (whole plant) was fully burnt to ash. After cooling the ash was collected and sieved through a mesh to remove any carbon particles. Then the ash was weighed and stored in air tight container.

Observations

The residue first obtained was black in color, indicating the presence of Carbon particles.

On heating, again and again with more pancāṅga, the final ash was observed to be white.

Precautions

After the burning of a heap of Krishnatila, the next bundle was added over that ash itself, i.e. on the same place, so as to ensure maximum heat to the drug in order to remove all carbon particles and obtain white ash.

The following table shows the quantity of drug used for the preparation of ash and the yield of ash.

Table 1: Quantity of drug used and Ash obtained

Drug	Quantity
Fresh pancāṅga	420 kg
Dried pancāṅga	150 kg
Ash obtained	21 kg

Preparation of Kṣāra**Materials required**

Stove, stainless steel vessels, ladle, cloth

Ingredients

Ash of Kṛṣṇatila: 21 kg

Water: 84 l

Procedure

The ash of Kṛṣṇatila was taken in a large stainless-steel vessel and four times water was added to it. It was macerated well for 3 hours and then left idle for three more hours. It was then strained through a three-folded cloth to collect the supernatant liquid. It was then heated over a stove in a low flame until all the water content got evaporated. The kṣāra was found in the bottom and sides of the container as flakes. It was scraped off and collected in an air tight container.

The following table shows the quantity of ash taken and kṣāra obtained:

Table 2: Quantity of Ash taken and Kṣāra obtained

Drug	Quantity
Ash of Kṛṣṇatila	21 kg
Kṣāra of Kṛṣṇatila	2.25 kg

PHYSICO-CHEMICAL ANALYSIS

The physico-chemical analysis was performed according to the standards of the Ayurvedic Pharmacopeia of India. The study was done in 3 aspects:

Organoleptic characters

Physico-chemical parameters

Assay of Sodium, Potassium and Iron

Organoleptic characters

It is the analysis of the sample using the sensory organs (jnanendriya). It includes

1. Colour (Rupa)
2. Odour (Gandha)
3. State (Rupa)
4. Taste (Rasa)

These characters are helpful to have a primary idea regarding the quality of different formulations without using chemical tests.

Physico-chemical parameters

These are the physical and chemical characteristics of the formulation assessed by using various laboratory methods. In the present study, the physico-chemical parameters of the sample were assessed by the following methods:

1. pH
2. Loss on drying
3. Total ash
4. Acid insoluble ash

pH²

The pH value of an aqueous liquid may be defined as the negative logarithm of hydrogen ion concentration. It gives the quantitative indication of acidity or alkalinity of a solution.

The pH value of a liquid can be determined by means of a glass electrode, a reference electrode and a pH meter either of the digital or analogue type. If the sample is in the form of powder, pH can be assessed by preparing 5% or 10% aqueous solution.

Materials required

Beaker, Spatula, Distilled water, pH meter.

Procedure

10 % aqueous solution was prepared by taking approximately 5g of the sample in 95 ml of distilled water in a beaker. The pH of the aqueous solution was determined by using the pH meter. The electrode of the pH meter was dipped in the solution in the beaker and the pH was noted from the display monitor.

Loss on Drying (LOD)³

Loss on drying is the loss of mass expressed as percent m/m. (British pharmacopoeia, 2012 online - Appendix IX D). This test determines the amount of volatile matter of any kind (volatile compounds; not necessarily water) that is driven off under specified conditions (110-120°C).

Materials required

Vessel, Spatula, Shimadzu Moisture Balance.

Procedure

The dome of the instrument was opened and the Aluminum dish was placed on the tripod needle. The tare weight of the dish was noted. Approximately 2 g of the sample was spread on the Aluminum dish. Heating started automatically to evaporate the moisture from the sample. The reading from the display monitor was noted after the evaporation was over.

Total Ash⁴

Total ash is the measure of the inorganic residue remaining after the removal of water and organic matter by heating in the presence of an oxidizing agent at approximately 555°C. It determines the non-volatile inorganic content in the sample. Ash value is used in determining the quality of the formulation, especially in powder form. The objective of this is to remove all traces of organic matter. On incineration, drug sample normally leaves an ash usually consisting of carbonates, phosphates and silicates of sodium, potassium, calcium and magnesium.

Materials required

Crucibles, Muffle furnace, Weighing balance, Spatula, Desiccator.

Procedure

2 to 3 g of accurately weighed sample was taken in a pre-tared silica crucible and placed inside a muffle furnace. Temperature was set at 550°C and the sample was incinerated at 550°C for 3 hours. After 3 hours, power of the Muffle furnace was switched off and could cool. After complete cooling down, the crucible was weighed at room temperature and the difference in weights was noted.

The total ash percentage of the sample was calculated from the following formula

$$\% \text{ Total Ash} = (W \text{ ash} / W \text{ sample}) \times 100$$

where, W ash = Weight of ash, W sample = Weight of Sample

Acid-Insoluble Ash⁴

It is the part of the total ash remaining after treatment with Hydrochloric acid solution under conditions specified. It gives an idea about the inorganic content of the sample, which is not soluble in dilute HCl. Acid insoluble ash primarily consists of silica and silicates.

Materials required

Crucibles, Muffle furnace, Weighing balance, Spatula, Desiccator, Dilute Hydrochloric acid, Watch glass, Filter paper and Hot water.

Procedure

To the crucible containing total ash, 25 ml of dilute Hydrochloric acid was added. It was covered with a watch glass and boiled gently for 5 minutes. The insoluble matter was collected on an ash less filter paper (Whatman 41) and washed with hot water until the filtrate was neutral. The filter paper containing the insoluble matter was transferred to the original crucible and was incinerated for 45 minutes in Muffle furnace at a temperature of 570°C. The residue could cool in a suitable desiccator and was weighed at room temperature. The acid-insoluble ash of the given sample was calculated with reference to air dried material.

Calculation

$$\% \text{ Acid Insoluble Ash} = (W \text{ acid insoluble ash} / W \text{ sample}) \times 100$$

where, W acid insoluble ash = Weight of acid insoluble ash, W sample = Weight of Sample

Estimation of Sodium and Potassium by Flame Photometer⁵

Preparation of Standard solutions

2.542 g of AR sodium chloride was weighed and dissolved in purified water and made up to 1000 ml in a volumetric flask. 1 ml of the stock solution was diluted to 100 ml. This gave the standard of 1mg of sodium per 100 ml (10 ppm). 20, 30, 40 and 50 ppm standard solution were prepared. 1.9115g of AR potassium chloride was weighed and dissolved in purified water and made up to 1000 ml in a volumetric flask. 1ml of the stock solution was diluted to 100ml. This gave the standard of 1mg of sodium per 100 ml (10 ppm). 20, 30, 40 and 50 ppm standard solution were prepared.

Preparation of Sample solution

10 g of sample was taken in a pre weighed silica dish and heated in a muffle furnace for 1hr at 600°C. The ash was cooled and dissolved in purified water and made up to 100ml in a volumetric flask. The instrument was first switched on and then the pump. Distilled water was kept for aspiration and it could stand for 15 min (warming time). The glass cylinder was opened and ignited. The instrument was adjusted to zero. The maximum concentration solution was introduced and adjusted to 100. Again the purified water was added so that instrument showed zero. Then the standard solution was introduced in ascending concentration. The reading was noted each time. Purified water was introduced for aspiration in between the standard solutions. The sample solution was introduced and the reading was taken. The curve was plotted with concentration in ppm against reading obtained. The concentration of the sample solution was found. Three readings were taken and the average was calculated. The concentration of sodium and potassium was calculated.

Determination of Iron (Fe) ⁶

Preparation of sample solution

A suitable quantity of the sample (in the presence of organic matter) was ignited in a crucible in a muffle furnace at 500-550° until the residue was free from organic matter. 5-10 ml of Hydrochloric acid was moistened, boiled for two minutes, 30 ml of water was added, heated on water bath for a few minutes, filtered and the residue was washed thoroughly with water and made up to volume in a volumetric flask.

Solutions

Stannous chloride solution – 5 g stannous chloride (A.R) was dissolved in 25 ml Conc.Hydrochloric acid and dilute to 100 ml (5 per cent solution)

Mercuric chloride – saturated solution in water.

Sulphuric acid + orthophosphoric acid mixture – 60 ml water + 15 ml conc.

Sulphuric acid + 15 ml H3PO4 cool and diluted to 1000ml.

Diphenylamine barium sulphonate – 0.25 g was dissolved in 100 ml water.

0.1 N Standard potassium dichromate solution. 4.9035 g AR grade was dissolved in water and diluted to 1000 ml.

Procedure

A suitable aliquot from the stock solution in 250 ml was taken in duplicate. It was diluted to about 100 ml with distilled water. 1-2 drops of Methyl red indicator and 1-2 g Ammonium chloride was added. Dil. Ammonium solution was added till brown precipitate appeared. The solution was boiled with ppt. for 4-5 minutes. The content was cooled and filtered through Whatman 41 no. filter paper. The residue was washed with hot water 4-6 times. The residue was dissolved in dil. HCl in 250 ml beaker.

Washed with hot water and made the volume to 100 ml approx. The solution was boiled on burner. Fe^{3+} to Fe^{2+} was reduced by adding stannous chloride solution drop wise till solution became colourless. Drops of stannous chloride solution was added in excess. The content was cooled in water. 10-15 ml 10per cent solution of mercuric chloride was added plus 25 ml acid mixture plus 2-3 drops of diphenylamine barium sulphonate indicator. It was titrated against standard potassium dichromate solution. Appearance of violet colour showed end point. Each

ml of 1N $\text{K}_2\text{Cr}_2\text{O}_7$ solution is equivalent to 0.05585 g Iron

RESULTS

After the pharmaceutical preparation, the formulation was put forward to various physical and chemical tests for its quality assurance and standardization. The results of the analytical study of *Kṛṣṇatila kṣāra* are illustrated in the following tables:

Table 3: Organoleptic Characters of *Kṛṣṇatila Kṣāra*.

Parameter	Result
Colour	Off white
Odour	Characteristic of kṣāra
Taste	Characteristic of kṣāra
State	Powder

Table 4: Physico-Chemical Parameters of *Kṛṣṇatila Kṣāra*

Parameter	Result
pH (10% aqueous solution)	10.92
LOD	6.37%
Total ash	86.84%
Acid insoluble ash	0.93%

Table 5: Estimation of Sodium, Potassium and Iron

Assay	Result
Sodium (Sodium as Na)	1.20%
Potassium (Potassium as K)	15.09%
Iron	921 mg/kg

PREPARATION OF ASH FROM KṚṢṆATILA

PREPARATION OF KṢĀRA FROM ASH OF KṚṢṆATILA

Figure 1: Dried Kṛṣṇatila pancanga, Figure 2: Bundles of Kṛṣṇatila pancanga, Figure 3: Burning of Kṛṣṇatila pancanga in open air
Figure 4: Ash obtained from Kṛṣṇatila pancanga, Figure 5: Ash kept for sedimentation, Figure 6: Filtering after sedimentation
Figure 7: Supernatant liquid obtained, Figure 8: Boiling of Supernatant liquid, Figure 9: Kshara obtained as flakes

DISCUSSION

The pH value (10% aqueous solution) 10.2 indicated the characteristic alkalinity of kṣāra. Loss on Drying at 110 °C was analysed to find out the moisture content. The LOD came within the standard range of kṣāra. The Total ash gives the percentage of non-volatile inorganic matter in the product which was found to be 86.84% and the value of Acid insoluble ash was 0.93%. Both the values came within the standard range of kṣāra.

The assay of Potassium, Sodium and Iron were also done. The value of Potassium as K was found to be 15.09%, Sodium as Na was 1.20% and Iron was 921mg/kg. Potassium is very important in the human body. Along with sodium, it regulates the water balance and the acid-base balance in the blood and tissues. Iron and sodium are important minerals iron for blood and cell functions.

CONCLUSION

The conversion of the drug Kṛṣṇatila into kṣāra form helps in preparing a formulation with a reduced dose as well as increased shelf life. From the results of the analytical study it can be concluded that Kṛṣṇatila kṣāra prepared passes all the physico-chemical standards as per API and can be used for further clinical trial.

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