



Research Article

PREPARATION AND PHYSICOCHEMICAL CHARACTERISATION OF *SURYAPRABHA GULIKA*

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ABSTRACT

Suryaprabha gulika is a *Kharaleeya* preparation mentioned in the *Gulika prakarana* of *Sahasrayogam* indicated for *Sula*, *Swasa*, *Kasa* and *Mahajwara*. It contains eleven ingredients namely *Suddha Parada*, *Suddha Gandhaka*, *Suddha Hingu*, *Amalaki*, *Harithaki*, *Vibheethaki*, *Sunti*, *Pippali*, *Maricha*, *Yavani* and *Suddha Vatsanabha* triturated for 6 hours in *Jambeera swarasa*. In this paper, the work done on pharmaceutical and analytical aspects of *Suryaprabha gulika* is detailed. Pharmaceutical study included *Sodhana* of *Parada*, *Sodhana* of *Gandhaka*, preparation of *Kajjali*, *Sodhana* of *Hingu*, *Sodhana* of *Vatsanabha* and the preparation of final product whereas the analytical study included evaluation of organoleptic characters, physico chemical parameters and analysis based on modern instrumentation techniques such as X-ray Fluorescence (XRF), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and particle size analysis.

INTRODUCTION

Ayurveda means 'The Science of Life'. Ayurvedic knowledge, also known as the "Mother of All Healing," has its origin in India and has been practiced for more than 5000 years. It has been passed down orally from successful masters to their followers for many thousands of years and has its roots in the ancient Vedic civilization. Ayurveda emphasizes prevention and promotes health maintenance by emphasising balance in one's life, proper thinking, a healthy diet, active lifestyle, and the use of herbs, minerals, and metals. *Rasasastra* is one of such evolved branches in Ayurveda, which is today can be considered as Indian pharmaceuticals of herbo-mineral preparations. Literally 'Rasa' means mercury and 'Sastra' means science, hence initially *Rasasastra* is considered as the science of mercury. The available literatures of *Rasasastra* strongly indicates that mercury is the basic reason for origination of this science although, therapeutic utilisation of metallic-mineral substances through

various processing methods for treating numerous diseases is the core subject of *Rasasastra*.

Numerous formulations are explained in the literature containing mercury, sulphur, minerals, metals and herbs of innate poisonous properties. The present work concentrates on one such formulation mentioned in *Gulika prakarana* of *Sahasrayogam*, a Malayalam classical treatise on Ayurveda. *Rasaushadhis* are the drugs considered to be superior to all other therapies because of the attributes such as low dosage, absence of distaste, rapid effectiveness and wide range of therapeutic utility.^[1] *Suryaprabha gulika* is one such herbo-mineral formulation classified under *Kharaleeya rasayana* in which mercury is ground along with other drugs in a *Kharala* or *Khalwa* indicated for *Sula*, *Swasa*, *Kasa* and *Mahajwara*.^[2] It consists of eleven ingredients in which four of them such as *Parada*, *Gandhaka*, *Hingu* and *Vatsanabha* require proper purificatory procedures before its use in therapeutics. Ingredients like *Parada*, *Gandhaka* and *Vatsanabha* cause serious health hazards in human if used without purification. Other ingredients include *Amalaki*, *Harithaki*, *Vibheethaki*, *Sunti*, *Pippali*, *Maricha* and *Yavani*. *Jambeera swarasa* is used as *Bhavana dravya* in order to prepare *Gulika*.

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MATERIALS AND METHODS**Pharmaceutical Study**

Various steps for the preparation of the *Suryaprabha gulika* are as follows;

1. Collection of raw materials
2. *Sodhana* of *Parada*

3. *Sodhana* of *Gandhaka*
4. Preparation of *Kajjali*
5. *Sodhana* of *Hingu*
6. *Sodhana* of *Vatsanabha*
7. Preparation of *Churnas* of herbal ingredients
8. Preparation of the final drug

Composition of *Suryaprabha Gulika***Table 1: Composition and proportion of each ingredient in *Suryaprabha gulika***

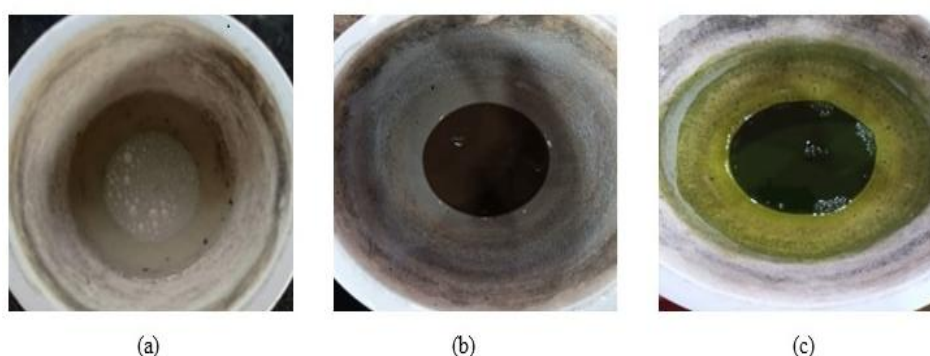
Drug	Botanical/Chemical name	Family	Parts/Form used	Ratio in <i>yoga</i>
<i>Parada</i>	Hg (Hydrargyrum)	-	Purified	1 part
<i>Gandhaka</i>	S ₈ (sulphur)	-	Purified	1 part
<i>Ramata</i>	<i>Ferula foetida</i>	Umbelliferae	Oleo gum resin	1 part
<i>Amalaki</i>	<i>Emblica officinalis</i>	Euphorbiaceae	Fruit	1 part
<i>Harithaki</i>	<i>Terminalia chebula</i>	Combretaceae	Fruit	1 part
<i>Vibheethaki</i>	<i>Terminalia belerica</i>	Combretaceae	Fruit	1 part
<i>Sunti</i>	<i>Zingiber officinale</i>	Zingiberaceae	Dried rhizome	1 part
<i>Pippali</i>	<i>Piper longum</i>	Piperaceae	Fruit	1 part
<i>Maricha</i>	<i>Piper nigrum</i>	Piperaceae	Fruit	1 part
<i>Yavani</i>	<i>Trachyspermum ammi</i>	Umbelliferae	Fruit	1 part
<i>Visha</i>	<i>Aconitum ferox</i>	Ranunculaceae	Tuberous root	1/16 th of total weight

Collection of Raw Materials

99.9% pure distilled mercury and sulphur powder were purchased from the local laboratory supply store at Thiruvananthapuram. Tubers of *Vatsanabha* was collected from genuine raw material dealer from Belgaum, Karnataka and authenticated by *Dravya guna* expert while all other herbal ingredients were procured from authentic sources in Thiruvananthapuram.

Sodhana* of *Parada

Sodhana or purification of *Parada* was done as per the reference in *Rasendra Sara Sangraha*. It involved the *Mardana* (trituration) of 150g of *Parada* in three different medias such as *Kumari swarasa* (juice of *Aloe vera*), *Chithraka kashaya* (decoction of *Plumbago zeylanica*) and *Kakamachi swarasa* (juice of *Solanum nigrum*) in a *Khalwa yantra* (mortar and pestle).^[3] *Chithraka sodhana* was carried out as per the reference in *Rasatarangini* using *Churnodaka* prior to the preparation of *Kashaya*.^[4]

**Fig no 1: *Parada sodhana* in different medias**

(a) *Kumari swarasa bhavana* (b) *Chithraka kashaya bhavana* (c) *Kakamachi swarasa bhavana*

Table 2: *Parada sodhana*

Weight of <i>Parada</i> taken	<i>Bhavana dravya</i>	Amount of <i>Bhavana dravya</i> used	Time taken for <i>Bhavana</i>	Weight of <i>Parada</i> after <i>Sodhana</i>	Loss of <i>Parada</i>
150g	<i>Kumari swarasa</i>	40ml	12 hrs	148.9g	1.1g
148.9g	<i>Chithraka kashaya</i>	40ml	12 hrs	146	2.9g
146g	<i>Kakamachi swarasa</i>	40ml	12 hrs	144.43	1.57g

Sodhana of Gandhaka [5]

Gandhaka sodhana was done as per *Kurmaputa* method explained in *Ayurveda Prakasha*. A wide-mouthed earthen pot with a capacity of five litre with an approximate clay lid was taken for the procedure and the inner surface of the pot was smeared with sufficient quantity of *Ghritha*. Then the pot was filled with three litres of cow's milk. Mouth of pot was tied with a thin and clean double layered cloth and a small depression was made in the centre to hold the *Gandhaka*. Over this cloth, 500g of finely powdered *Gandhaka churna* was evenly spread and covered with a *Sarava* and *Sandhi bandhana* was done with *Multhanimitti* smeared cloth for seven rounds. On drying, the apparatus was carefully placed inside a round pit enclosing the pot upto the above neck portion. Fine sand was used to fill the gap between the pit wall and the earthen pot in a way that the top portion of the apparatus was exposed. On the uncovered portion of the *Sarava*, coconut husks were placed and ignited. New pieces of coconut husks were inserted as the method progressed until all the pieces (50 pieces) were consumed. Peak temperature reached was about 448°C. Due to heat, the sulphur on the cloth melts and falls down into the milk. The apparatus was allowed to self cool and after cooling, the vessel was carefully taken out from the pit and *Sandhi bandhana* was removed. *Sodhitha gandhaka* was collected from the bottom of vessel and it was washed with hot water till *Gandhaka* gets free from oiliness of ghee. Finally, it was collected, dried, weighed and stored.

Table 3: Gandhaka sodhana

<i>Gandhaka</i> before <i>Sodhana</i>	500g
<i>Gandhaka</i> after <i>Sodhana</i>	475g
Loss of weight	25g
Time taken for the procedure	4 hrs

**Fig no 2: Different steps in Gandhaka sodhana****Preparation of Kajjali**

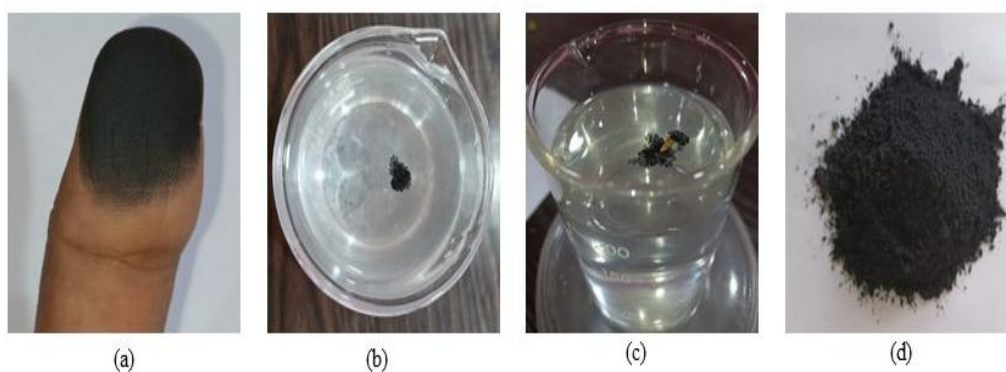
Kajjali was prepared by grinding equal quantities of *Suddha parada* and finely powdered *Suddha gandhaka* (sieved through sieve no. 120) in a *Khalwa yantra*. *Mardana* was carried out until *Kajjali siddha lakshanas* were obtained.

Table 4: Observation during preparation of Kajjali

Quantity of <i>Suddha parada</i>	15g
Quantity of <i>Suddha gandhaka</i>	15g
Time taken for grinding	42 hours
Weight of <i>Kajjali</i> obtained	28g
Loss of weight	2g

Table 5: Kajjali siddha lakshanas

<i>Slakhnatwa</i>	7 hrs 30 mts
<i>Rekhapurnatwa</i>	7 hrs 30 mts
<i>Varitaratwa</i>	10 hrs
<i>Unama lakshana</i>	10 hrs
<i>Nischandratwa</i>	42 hrs

Fig no 3: *Kajjali siddha lakshanas*(a) *Rekhpurnatwa* (b) *Varitaratwa* (c) *Unama* (d) *Nishchandratwa***Sodhana of Hingu** [6]

Purification of *Hingu* was done as per the reference in *Rasatarangini* by the method of *Bharjana* (frying) of *Hingu* in equal quantity of ghee in an iron pan until it became brown in colour and crispy enough to powder it.

Table 6: Hingu sodhana

<i>Hingu</i> before <i>Sodhana</i>	150g
Quantity of ghee	150ml
<i>Hingu</i> after <i>Sodhana</i>	148g
Loss of weight	2g

Sodhana of Vatsanabha [7]

Purification of *Vatsanabha* was done as per the classical method explained in *Rasatarangini*. Rhizomes of *Asodhitha vatsanabha* were cut into small pieces and kept in a mud vessel containing fresh *Gomutra* which was taken enough to immerse the samples in it. This was kept in bright sunlight for three days changing the cow's urine every day. After three days *Sthapana* in *Gomutra*, *Vatsanabha* pieces were washed with warm water. Outer corticular layer was peeled off by wearing sterile hand gloves. Only inner pulp was collected and dried well.

Table 7: Vatsnabha sodhana

<i>Vatsnabha</i> before <i>Sodhana</i>	80g
<i>Vatsanabha</i> after <i>Sodhana</i>	36g
Loss of weight	44g

Preparation of Churnas of herbal ingredients

150g each of *Amalaki*, *Harithaki*, *Vibheethaki*, *Sunti*, *Pippali*, *Maricha* and *Yavani* were washed to remove the external impurities like mud, stone etc and dried well separately. Each of them was then powdered and sieved through sieve no.85 to get fine powders separately. 148g of *Sodhitha hingu* and 36g of *Sodhitha vatsanabha* were also powdered in the similar way. All of them stored in a clean and dry air tight containers separately.

Extraction of Jambeera swarasa

Fresh and mature 30 *Jambeera* fruits (*Citrus limon*) were collected from the local market and washed well. Later its external skin was peeled off and squeezed well to extract juice from it. This extracted juice was filtered through a double layered cloth. Totally 450ml of *Jambeera swarasa* collected at the end of procedure.

Preparation of the Final Drug

Ingredients such as 28g of *Kajjali*, 14g each of *Hingu*, *Amalaki*, *Harithaki*, *Vibheethaki*, *Sunti*, *Pippali*, *Maricha* and *Yavani* were taken in a mortar. The amount of *Vatsanabha churna* taken was 1/16th of total quantity i.e., 8.75g. All the ingredients were mixed uniformly to obtain a homogenous mixture. 300ml of *Jambeera swarasa* was added to the mixture taken in a mortar so as to completely immerse the whole *Churna* and *Bhavana* was done by continuous grinding. The trituration was continued for 6 hours till the formulation attained pill rolling consistency. It was then rolled into pills of size that of *Gunja* (seeds of *Abrus precatorius*). Average weight of one *Gunja* was estimated to be 125mg and the pills were made accordingly and dried under shade. After complete drying, pills were stored in a clean and dry air tight container.

RESULTS

Analytical study of *Suryaprabha gulika* involved 3 steps;

1. Evaluation of organoleptic characters
2. Physicochemical analysis
3. Instrumental analysis

Evaluation of organoleptic characters

Table 8: Organoleptic characters of *Suryaprabha gulika*

Parameters	Observations
Colour	Black
Odour	Characteristic of <i>Hingu</i>
Taste	Characteristic of its ingredients
State	Solid

Physicochemical analysis of *Suryaprabha gulika* [8,9]

Table 9: Physicochemical parameters of *Suryaprabha gulika*

Parameters	Observations
pH (5% solution)	3.1
LOD at 110°C	7.86 %
Total ash	4.38 %
Acid insoluble ash	0.199 %
Alcohol soluble extractive	16.84%
Water soluble extractive	25%
Hardness test	2.083kg/cm ²
Friability test	0.207 %
Weight variation	Within acceptable range of $\pm 7.5\%$ weight variation
Disintegration test (a) In water	90 minutes

XRF [10]

XRF of *Suryaprabha gulika* was carried out to determine the elemental composition of the prepared product at Central Sophisticated Instrumentation Facility (CSIF), Calicut.

Table 10: Elemental composition of *Suryaprabha gulika*

Element	Concentration	Element	Concentration
Mercury	10.05%	Magnesium	0.362%
Sulphur	12.18%	Aluminium	0.343%
Potassium	2.058%	Phosphorous	0.238%
Calcium	0.737%	Chlorine	0.238%
Sodium	0.702%	Iron	0.0224%
Silicon	0.605%	Tungsten	0.0141%

XRD [11]

Powder X-ray diffraction of *Suryaprabha gulika* was conducted to determine the crystallographic structure of the sample at Central Sophisticated Instrumentation Facility (CSIF), Calicut.

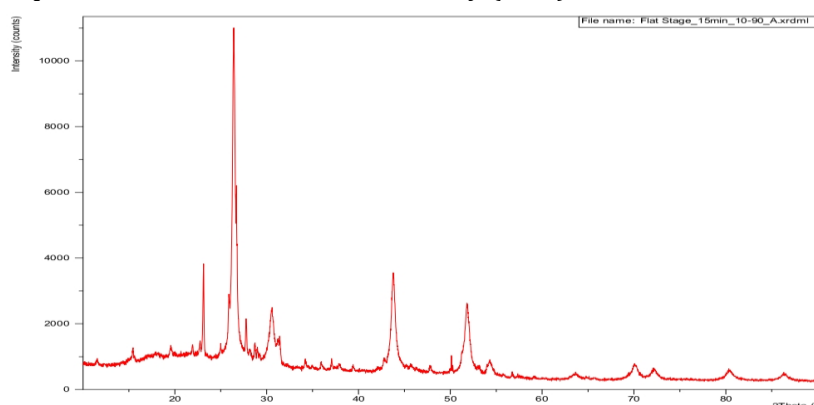


Fig no: 4 XRD analysis of *Suryaprabha gulika*

The diffractometer scans were made from 10.0084 [$^{\circ}2\theta$] to 89.9764 [$^{\circ}2\theta$] with a step size of 0.0170 [$^{\circ}2\theta$] and 22.8600 seconds as scan step time. The 2-theta value and intensity of the peak (counts) are represented on X-axis and Y-axis respectively. Total 5 strong peaks were identified from angle [2θ] from 23.1130 to 51.7899.

The XRD patterns of sample *Suryaprabha gulika* confirmed the presence of Mercuric sulphide with that of ICSD data of HgS available in the literature. HgS crystals usually occur in two allotropic forms such as α -HgS and β -HgS. Powder XRD measurements suggested the presence of β -HgS or metacinnabar particles with cubic crystalline structure in the given sample because of its high correlation with its characteristic peaks with that in literature.

The high intensity peaks at 2θ scale values 26.3985, 30.5651, 43.7429 and 51.7899 indicated the presence of metacinnabar in the sample. Similarity in the D-space values between the standard metacinnabar such as 3.37981, 2.92700, 2.06970, 1.76505 and the formulation such as 3.37350, 2.92246, 2.06777, 1.76382 in the XRD pattern strongly confirmed the presence of β -HgS in the sample.

Sample showed certain small peaks which indicated the presence of free sulphur with orthorhombic structure. The high intensity peak at 2θ scale value 23.1130 corresponds to that of free sulphur with D-space value 3.84507 which is similar to that of standard sulphur data in the literature. XRD pattern obtained also showed the different peaks corresponding to minor elements present in the formulation.

SEM^[12]

Scanning electron microscopy of *Suryaprabha gulika* was carried out in order to determine the surface morphology of the prepared sample at Central Sophisticated Instrumentation Facility (CSIF), Calicut.

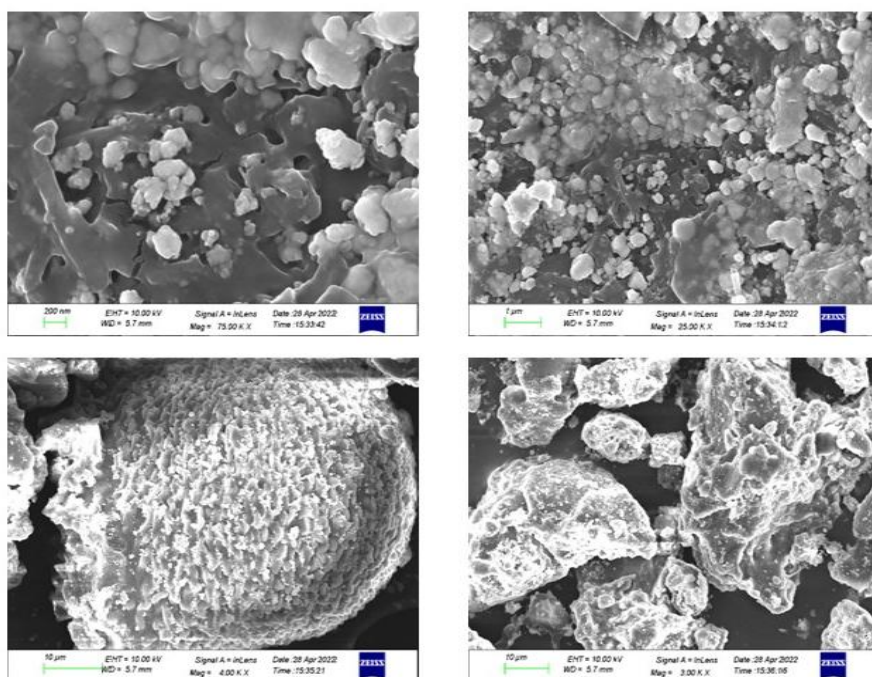


Fig no 5: SEM analysis of *Suryaprabha gulika*

Particle Size Analysis

Particle size analysis of *Suryaprabha gulika* was conducted at NIIST- National Institute for Interdisciplinary Science and Technology (CSIR), Thiruvananthapuram. The hydrodynamic diameter of particles in *Suryaprabha gulika* was found to be 663.5.

DISCUSSION

Various steps were involved in the preparation of *Suryaprabha gulika*. First step was *Parada sodhana* which was done as per the classical reference in *Rasendra sara sangraha* using *Kumari swarasa*, *Chithraka kashaya* and *Kakamachi swarasa* by means of *Mardana* in these medias for a period of twelve hours. The *Dravyas* used for *Sodhana* are mentioned in our classics to eliminate *Doshas* of *Parada*. *Kumari swarasa* and *Chithraka kashaya* help to eliminate *Mala*

dosha and *Visha dosha* respectively. It was evident from the literary search that this method could eliminate *Naisargika doshas* of mercury as *Dravyas* mentioned here have a specific role against specific *Dosha*. Due to the practical difficulty in performing *Ashta vidha samskaras* of *Parada*, *Visesha sodhana* method was adopted here. The peculiar property of mercury to split up into very tiny globules on *Mardana* was observed in all the three media. The loss after *Sodhana* was found to be more in *Chithraka kashaya* and *Kakamachi swarasa* since it formed cement like appearance while continuous grinding.

Gandhaka sodhana was done as per the reference in *Ayurveda Prakasha* by utilizing method of *Kurmaputa*. It is a practice that necessitates both talent

and labour. *Kurmaputa* method has an advantage of purification of large quantity of *Gandhaka* in single turn with more yield along with assurance of quality of *Gandhaka mani* obtained after *Sodhana* as it remains for several hours in milk and ghee. The loss of 25g observed after *Sodhana* is due to the removal of impurities from sulphur. *Saajya kurmaputa* method has one more advantage than *Nirajya* method because *Ghritha* has special *Vishahara* property, that is more beneficial to remove the toxic impurities of *Gandhaka*.

Kajjali was prepared using *Suddha parada* and *Gandhaka* in the ratio 1:1 as per the ration mentioned in the formulation. The persistent processing as trituration of mercury with sulphur was carried out till the classical quote specified criteria was achieved. Classical endpoints included *Slakshnatwa*, *Varitaratwa*, *Unama lakshana*, *Rekhapurnatwa* and *Kajjalabhava*. These parameters evaluate the physical nature of the formulation redirecting to the drug dissolution and drug absorption particulars. *Nischandratwa* is found to be most valuable to know the binding of particles in *Kajjali*. The procedure of grinding has to be continued until the mixture of *Parada* and *Gandhaka* become lustreless which indicates the absence of free particles of mercury.

Hingu sodhana was done as per the *bharjana* method in which oleo gum resin was fried in ghee until it became crunchy enough to powder it. When fried in ghee, *Ushna*, *Tikshna guna* and pungent odour gets reduced due to *Snigda*, *Sita guna* and pleasant odour of *Ghritha*. Negligible loss was observed. It became crispier and more brittle due to removal of excess moisture content. As per our classics, this procedure helps to get rid of *Utkledana* after the intake. *Vatsanabha sodhana* was done as per *Nimajjana* in *Gomutra* for 3 days. About 55% of loss was observed after *Sodhana*. Though there are other media mentioned in classics for *Vatsanabha sodhana*, it is found that *Gomutra* should be considered as the best media for *Sodhana* whether it is *Sthapana* or *Swedana* procedure. *Sodhana* treatments remove toxic effects from raw aconite. The diterpene alkaloids such as aconitine, hypaconitine and mesaconitine are poisonous components present in the root tubers, which is converted to less toxic alkaloids such as aconine, benzoylaconine and pyroaconine by means of deacetylation and oxidation reaction after *sodhana* procedure which renders *vatsanabha* fit for human consumption.^[13] The final product *Suryaprabha gulika* was prepared by continuous grinding of all the ingredients as per the reference until pill rolling consistency was observed. *Bhavana dravya* was taken in sufficient quantity to immerse the complete mixture of constituent drug powders so as to ensure proper processing of medicaments in *Jambeera swarasa*.

The physicochemical factors can be used to assess the purity and quality of a drug. pH, loss on drying, total ash, acid insoluble ash, alcohol soluble extractive, water soluble extractive, hardness, friability, weight variation and disintegration test were determined in the current study. The 5% drug solution of the drug was found to be acidic as its pH lies below 7 probably owing to the presence of *Jambeera swarasa* which is used as *Bhavana dravya*. Loss on drying was found to be 7.86% showing that the dosage form should be protected from humid atmosphere, otherwise the increased water content may result in developing fungus on the tablet. The total ash and acid insoluble ash values were found to be 4.38% and 0.199% respectively. Water soluble extractive value were found to be more than that of alcohol soluble extractive value and is useful for estimation of chemical constituents soluble in that particular solvent used for extraction. Friability is termed as the ability to withstand mechanical wear and tear. The friability of *Suryaprabha gulika* was found to be 0.207%, which was within the acceptable limit. *Gulikas* were rolled on an average weight of 125mg and the weighed within the acceptable range of $\pm 7.5\%$ weight variation as mentioned for tablet form. This parameter is suggestive of the uniformity of the prepared tablets. Both hardness and disintegration time determines the bio-availability of the drug. Sample prepared showed hardness of 2.083kg/cm² and disintegration time of 90 minutes in water as media.

Multi elemental analysis of the sample was done using X-ray fluorescence spectroscopy which identified the presence of more than 10 elements other than mercury and sulphur along with their trace concentration in the compound. As the test drug is a herbo-mineral preparation containing mercury and sulphur, it was subjected to XRD analysis to find the crystalline patterns of the *Kajjali* added in it. The presence of mercuric sulphide was confirmed in the samples especially in the form β -HgS with the help of ICSD data available in the literature. The XRD pattern of the given sample showed 4 peaks corresponding to that of mercuric sulphide which was confirmed by 2θ scale values and the d-spacing values of metacinnabar. One peak corresponding to free sulphur was also identified in the pattern which was also confirmed by 2θ and d-spacing values for the same. Scanning electron microscopy of the *Suryaprabha gulika* was done in order to determine the surface topography. Micrographs were presented at different magnifications and it identified the particles of varying sizes ranging between 200nm and 10 μ m implying the non-homogenic nature of mixture. Particle size analysis of the sample was conducted and found that average particle size in the compound is in nanometre range.

CONCLUSION

Suryaprabha gulika mentioned in *Sahasrayogam* is a herbo-mineral formulation with considerable therapeutic properties according to its constituents and is widely used in the management of fever associated with respiratory tract infections nowadays by the Ayurvedic practitioners in Kerala. The conventional pharmaceutical procedures were followed in the production of *Suryaprabha gulika*. For the standardisation and quality control of this formulation, the standards established in this study can be employed as a validating tool.

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