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STANDARD OPERATING PROCEDURE (SOPs) OF SAMEERA PANNAGA RASA PREPARED THROUGH VERTICAL ELECTRIC MUFFLE FURNACE

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ABSTRACT

Kupipakwa Rasayana is among unique Ayurvedic dosage forms prepared by Kupi Paka method (specific pharmaceutical heat treatment in glass bottle through sand bath). Sameera Pannaga Rasa is a Kupipakwa Rasayana containing Shuddha (processed) Parada (Hg), Shuddha Gandhaka (S), Shuddha Somala (As₂O₃), Shuddha Haratala (As₂S₃) and Shuddha Manahshila (As₂S₂) in equal quantity. Ingredient Manahshila is absent in formulation mentioned in ancient text; Rasa Chandamshu which was later added by author of recent principally compilation text; Ayurvediya Aaushadhi Guna Dharma Shastra which is also mentioned in Ayurvedic Formulary of India. Sameera Pannaga Rasa is extensively used and effective in the management of Tamaka Shwasa (bronchial asthma). In view of critical issues in the preparation of Sameera Pannaga Rasa, present study was conducted to develop its standard operating procedure (SOP), as till date it is lacking. Kajjali was prepared with Shuddha Parada, Shuddha Gandhaka, Shuddha

Haratala, Shuddha Somala & Shuddha Manahshila in equal quantity. Kancha Kupi (mud smeared glass bottle) was filled with Kajjali up to 2/3rd part and Sameera Pannaga Rasa was prepared as per standard procedure. Standard temperature conditions and necessary precautions were followed during the preparation. Talastha (bottom of bottle) greenish black and Kanthastha (neck of bottle) silvery shine coloured Sameera Pannaga Rasa was obtained. Total yield of Sameera Pannaga Rasa (combined products Talastha and Kanthastha) was 83.5%, 78% and 80.55% respectively from each batch of 200g of Kajjali. Total 37:35hrs. of active heating with maximum temperature of 520°C in graded manner through VEMF (Vertical electric muffle furnace) were needed to obtain desired product. The present method can be considered as standard operating procedure (SOP) for preparation of Sameera Pannaga Rasa in VEMF for 200g batch.

KEYWORDS: Arsenic, *Kanthastha, Kupipkwa Rasayana*, Mercury, Orpiment, Realgar, *Sameera Pannaga Rasa, Talastha*, VEMF (Vertical electric muffle furnace).

INTRODUCTION

In Ayurveda, Kupipakwa Rasayana is the most popular, unique and highly significant pharmaceutical preparation. Kupipakwa Rasayana is made up of four words that is Kupiglass bottle, Pakwa- heating or Paka, Rasa- Parada (Hg), Aayana- sthana (place). Formulations prepared in Kanchakupi (glass bottle) are known as Kupipakwa Rasayana and also known as *Sindura Kalpana* as their final product is red in colour. [1] Sri Harisharanananda has mentioned 256 different Kupipakwa Rasayana in Kupi-Pakwa Vijnaniyam. In context of Rasa Bhasma, Rasa Prakasha Sudhakara is the first text to describe Kupipakwa Rasayana, [2] (*Udaya bhaskara Rasa*). According to *Rasendra Chintamani*, there is specific heating pattern for Kupipkwa Rasayana named as Kramagni paka i.e. increasing manner of heat (Mruduagni-mild heat, Madhyamagni-moderate heat and Tivragni-high heat), each for a specified period. This methodology is used in practice now a days with some modifications. These Rasayana are prepared in especially classically designed device called Valuka Yantra (Sand bath assembly). The medicines prepared in 7 layered mud smeared glass bottle, by applying heat through Valuka Yantra or Vertical Electric Muffle Furnace are known as Kupipakwa Rasayana. In Kupipkwa Rasayana Parada (mercury) and Gandhaka (sulphur) are the chief ingredients along with other metals and minerals. Rasaushadhi are considered as best when compared to the drugs of herbal origin in view of their properties like small therapeutic dose, rapid action and palatability made Kupipakwa Rasayana on superior

position. [3,4] Sameera Pannaga Rasa (SPR) is one of the renowned Kupipakwa Rasayana of metallic origin. Primarily Sameera Pannaga Rasa has been mentioned in Rasa Chandamshu with the name of Vata Pannaga, [5] containing Parada, Gandhaka, Haratala and Somala in equal parts, in which Manahshila has not been mentioned as a component. But later, Ayurvediya Aaushadhi Guna Dharma Shastra added Manahshila in Sameera Pannaga Rasa which has been accepted by Ayurvedic Formulary of India. [6] Sameera Pannaga Rasa containing Gandhaka, Haratala and Manahshila are categorized under Uparasa varga, [7] whereas Somala comes under Sadharanarasa varga. [8] Parada, Haratala, Manahshila and Somala are highly potent metal/mineral/metalloids which are included in the list of poisonous drugs by Drug & Cosmetic Act 1940 under Schedule E(1) because of their highly toxic nature in crude form, [9] but they are often used not only in therapeutics but also in pharmaceutics too, necessarily after Shodhana (unique, controlled, pharmaceutical drug processing aimed to alter properties of drug as desired) and or *Marana* etc procedures. Several pharmacological properties of Sameera Pannaga Rasa(Tridoshaghna, Vatahara etc.) are mentioned in classical texts on Rasashastra. [10] It (SPR) is mentioned for the management of Sandhivata (osteoarthritis), Unmada (insanity-mental disorder), Kasa (cough), Jwara (fever) etc. It has been used extensively for Tamaka Shwasa (bronchial asthama). [11] and it also shows good effects on Amavata. [12] In commentary on Rasa Yoga Sagara by Pandit Hariprappanaji Arka Kshira has been advocated as Bhavana dravya of Sameera Pannaga Rasa without Manahshila, whereas Ayurvediya Aaushadhi Guna Dharma Shashtra has mentioned Tulasipatra Swarasa (Basil leaves juice) or Ghritkumari Swarasa (Aloe Vera juice) as Bhavana drava of Sameera Pannaga Rasa with Manahshila. It is to be heated through Valuka Yantra method. [10,13] Rasa Chandamshu advocated dose of SPR as 2 Gunja (250mg) with betel leaf as Sahapana (co-adjuvant), while in Ayurvediya Aushadhi Guna Dharma Shastra; it is mentioned as 1 Gunja (125mg) along with Sahapana (co-adjuvant) of beetle leaf. Review of previous research works reveals that SOP of Sameera Pannaga Rasa has not been developed yet. Hence, the present study was conducted to develop a standard operating procedure (SOP) of Sameera Pannaga Rasa in vertical electric muffle furnace followed by reference of AFI-1.[14]

MATERIAL AND METHOD

In present study, Sameera Pannaga Rasa was prepared as per the reference of Ayurvediya Aushadhi Guna Dharma Shastra (AFI-1).^[14]

Total three batches of Sameera Pannaga Rasa were prepared to develop the SOPs.

Parada, Gandhaka, Haratala, Somala & Manahshila was procured from Pharmacy department, Gujarat Ayurved University, Jamnagar, while leaves of *Tulasi* (Ocimum sanctum Linn) were collected from periphery of Jamnagar and authenticated by Pharmacognosy laboratory of IPGT&RA, GAU, Jamnagar.

Pharmaceutical manufacturing of *Sameera Pannaga Rasa* was carried out at department of Rasa Shastra & Bhaishajya Kalpana, IPGT & RA, GAU, Jamnagar.

Table 1: Ingredients of Sameera Pannaga Rasa with their ratio.

Sr.	Ingredients	English name/Latin	Qty./Proportion	Qty.	
No.	ingredients	Name	(given)	(Taken)	
1.	Shuddha Parada	Mercury	1 Part	40g	
2.	Shuddha Gandhaka	Sulfur	1 Part	40g	
3.	Shuddha Haratala	Orpiment	1 Part	40g	
4.	Shuddha Somala	White Arsenic	1 Part	40g	
5.	Shuddha Manahshila	Realgar	1 Part	40g	
Total Quantity = 200 g					
	Bhavna dravya				
1.	Tulasi Patra Swarasa	Ocimum sanctum Linn.	Q.S.	Q.S.	

Equipments: weighing balance, VEMF (vertical electric muffle furnace), edge runner, iron rod, cotton cloth, mud (fuller's earth), *Kupi* (funnel), *Kancha Kupi* (amber glass bottle), torch, coin.

Table 2: Circumference specification of *Kacha Kupi* (glass bottle).

Bottle	Before Kapadmitti	After 7 layers of Kapadmitti
Height	27.9 cm	28.4 cm
Mouth	09.1 cm	14.08 cm
Neck	14.05 cm	17.08 cm
Bottom	24.2 cm	26.09 cm
Weight	421.04g	636.1 g

Pharmaceutical process of Sameera Pannaga Rasa

Equal quantity of *Shuddha Parada* (Hg) and *Shuddha Gandhaka* were taken and *Mardana* (dry trituration) was carried out in edge runner till it became *Nishchandra* (lusterless) *Kajjali* then equal amount of powdered *Shuddha Haratala* (As₂S₃) and *Shuddha Manahshila* (As₂S₂) were added respectively and homogenous mixture was prepared by continuous trituration. At the end, powdered *Shuddha Somala* (As₂O₃) was added to it with continuous trituration and homogeneous mixture of all ingredients was prepared. *Bhavana* (levigation/wet trituration) of *Tulasipatra Swarasa* (leaves juice of *Ocimum sanctum* Linn.) was given three times to

prepared *Kajjali*. A clean *Kancha Kupi* (amber colored narrow mouth, vertical glass bottle, cylindrical body, base and conical neck-Table 2) with volume of 750 ml was taken and mud (fuller's earth) smeared cotton cloth pieces were wrapped on *Kupi* for 7 times uniformly & consecutively applied only after complete drying of former layer. The *Kajjali* was filled up to $2/3^{\rm rd}$ in 7 layered *Kancha Kupi* (amber colored glass bottle) and properly adjusted in VEMF (vertical electric muffle furnace). *Kajjali* containing *Kancha Kupi*, was heated gradually in vertical electrical muffle furnace at three different temperature i.e. *Mriduagni* (mild heat), *Madhaymagni* (moderate heat) and *Tivragni* (high heat) for certain time period. During preparation of *Sameera Pannaga Rasa*, variation in heating temperature were observed (Table 4).

To confirm the melting & boiling stage of *Kajjali, Shita Shalaka* (cold iron rod) was inserted into the neck of *Kupi* as and when necessary. *Tapta Shalaka* (red hot iron rod) was inserted into the neck of *Kupi*, to clear deposited materials inside the mouth of the *Kupi*. After 37.35hrs of active heating, with maximum temperature of 520°C, vertical electrical muffle furnace was switched off and *Kupi* was kept for self cooling. Once *Kupi* was cool, it was taken out and cleaned carefully. *Kupi* was broken in the middle so as to separate the lower and upper halves. For this, a kerosene dipped thread was wrapped on *Kupi* and ignited; then after the fire extinguished, wet cloth was wrapped immediately on *Kupi*. After breaking of *Kupi*, *Sameera Pannaga Rasa* deposited at either places i.e. *Talastha* (bottom of glass bottle) & *Kanthastha* (neck of glass bottle) was procured by careful scrapping and gentle tapping. The product was scraped, collected & stored in air tight container. During the entire process, care was taken to avoid mixing of glass particles and product. Above mentioned method was repeated in successive batches.

OBSERVATIONS AND RESULTS

Observations during preparation of *Kajjali*

Shuddha Parada & Shuddha Gandhaka were mixed and grinded together firmly turned in to black coloured Kajjali. Kajjali became Nishchandra (lusterless) after 23 hrs. of dry trituration. On addition of powered Shuddha Haratal & Shuddha Manahshila in Kajjali, it becomes slightly yellowish-orange in colour (initially) and after addition of Shuddha Somala powder, Kajjali turned into slight grayish tinge. Bhavana with Tulasi Swarasa to the Kajjali turned it to slight greenish black in colour. Characterstic odour of Tulasi Swarasa was evident during preparation of Kajjali.

Table 3: Organoleptic characters of raw material and Kajjali.

Ingredients	Colour	Odour	Touch
Shuddha Parada	Silvery		Smooth
Shuddha Gandhaka	Light yellow	Characteristic	Slight rough
Shuddha Haratala	Yellow	Characteristic	Smooth
Shuddha Somala	Off white	Characteristic	Smooth
Shuddha Manahshila	Deep orange	Characteristic	Fine
Kajjali (with bhavana dravya)	Greenish black	Characteristic	Fine (<i>Rekhapurnatva</i>)

Table 4: Average observations during preparation of Sameera Pannaga Rasa.

Time in Hrs.	Temperature (C ⁰)	Observations			
00:00	00	Vertical electric muffle furnace switched on			
Start	100	No observation			
1	150	Mild whitish fumes with characteristic smell like <i>Tulasi Swarasa</i>			
1.35	200	Clear whitish fumes followed by mild yellowish fumes			
3:35	210	Yellowish fumes			
4.40	220	Yellowish fumes; neck of <i>Kupi</i> looks yellowish			
5.40	230	Yellowish fumes increased; mild characteristic odour of Gandhaka Sulphur); Kajjali started to melt (slight muddy consistency)			
6.35	240	Neck of <i>Kupi</i> becames yellowish coloured; yellowish fumes with intense smell; whitish fume seen on <i>Kajjali</i> surface inside <i>Kupi</i> .			
6.55	250	Tellowish granules like few crystals stuck to the rod when it was asserted at the level of neck. Thick muddy consistency of <i>Kajjali</i> evealed by insertion of cold rod.			
9.35	270	Yellowish granules like mild crystals stuck to the cold iron rod, intense characteristic smell; <i>Kajjali</i> molten and became muddy in consistency.			
11.35	290	Whitish and yellowish fumes with characteristic unpleasant smell			
13.15	300	Kajjali became soft and dense yellowish fumes was seen			
14.55	315	Molten <i>Kajjjali</i> started to stick on the tip of <i>Sheeta Shalaka</i> ; dense yellowish granules like material stick on <i>Sheeta Shalaka</i>			
16.35	320	Shiny reddish orange coloured crystals seen at inner wall of bottle neck.			
24.25	330	Yellowish fumes disappeared; <i>Kajjali</i> became shiny and got molten; <i>Kajjali</i> started raised up			
25.25	340	Sheeta Shalaka was inserted frequently at regular intervals of 10-15 min., so as to prevent spillage by rising level of molten Kajjali. Sheeta Shalaka was done to pushed down the raised Kajjali;			
26.35	350	<i>Kajjali</i> became soft & molten completely; dark orange colour was visible on <i>cold iron rod</i>			
28.35	360	Mild whitish fume with shiny particles were visible inside the <i>Kupi</i> ; insertion of <i>Sheeta Shalaka</i> was conducted at 10-15 min intervals			
29.35	370	Mild whitish fumes with appearance of shiny particles inside the <i>Kupi</i> ; <i>Sheeta Shalaka</i> inserted 10-15 min intervals; Shining particles float around the neck of <i>Kupi</i> (honey comb appearance); <i>Ushna Shalaka</i> (red hot iron rod)inserted to clean the neck of <i>Kupi</i> and to avoid the choking due to the raised <i>Kajjali</i>			

31.05	390	Sheeta Shalaka inserted to push the material downwards; dense
31.03		orange coloured fumewith mild yellowish on Sheeta Shalaka
	400	Molten Kajjali frequently raised upwards; slight black with dense
32.05		orange coloured deposition on Sheeta Shalaka; Sheeta Shalaka
		inserted at 10-15 min intervals
		Molten Kajjali frequently raised upwards; slight black with dense
33.05	410	orange coloured deposition on Sheeta Shalaka; Cold iron rod inserted
		at 10-15 min intervals
		Deposition of reddish black with orange tint was observed on <i>Sheeta</i>
33.35	420	Shalaka; Kajjali raised upwards & Sheeta Shalaka & Tapta Shalaka
		was done alternatively respectively
	430	Neck was cleaned with red hot <i>Shalaka</i> ; deposition in sequence of
34.05		black, orange, faint yellow & white was seen on Sheeta Shalaka;
		raised Kajjali was pushed down with Sheeta Shalaka.
	450	Mild whitish fumes were observed; boiling of <i>Kajjali</i> was seen inside
24.40		the <i>Kupi</i> with the help of torch light; <i>Sheeta Shalaka</i> was done; faint
34.40		free radicals of mercury was seen on Sheeta Shalaka; compactness of
		Kajjali was seen on Sheeta Shalaka
		A depostion in sequence like black, orange followed by light yellow
	460	were observed; boiling of <i>Kajjali</i> was seen inside the <i>Kupi</i> with the
35		help of torch light; Sheeta Shalaka was done; faint free radicals of
		mercury was seen on Sheeta Shalaka; compactness of Kajjali was
		seen on Sheeta Shalaka
		Deposition on Sheeta Shalaka in sequence of black, orange, yellow
		with faint white were seen from bottom to top; upper layer of boiling
35.35	480	Kajjali was visible and it appears to be golden yellow; Tapta Shalaka
		was inserted to clear the neck of <i>Kupi</i> , bright grayish material gets
		attached on Sheeta Shalaka
		Deposition of dark black followed by reddish orange colour matter
36.20	500	was seen on Sheeta Shalaka, material settled down, a golden layer
		was seen on the upper layer of boiling <i>Kajjali</i>
	520	Deposition in sequence of black (3/4)-reddish orange-yellow-grey
36.50		(1/4) was observed; clear golden layer was seen on the surface of
		boiling Kajjali.
37.35	Switched off	Switched off of vertical electric muffle furnace & Kupi left for self
31.33	Switched Off	cooling.

• During the process, overall heat pattern was as below

Mridu agni for 11:35hrs (100-250°C)

Madhyamagni for 19:07hrs (250- 400°C)

Tivragni for 6:03hrs (400-520°C)

- *Kanthastha* (neck portion of bottle) product (*Sameera Pannaga Rasa*) was shiny and reddish in colored.
- *Talastha* (bottom portion of bottle) *Sameera Pannaga Rasa* was dark greenish black and compact in nature.

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Table 5: Details results of Sameera Pannaga Rasa.

Batch	Total Time	Temperature	Initial	Final Weight		% yield	Loss (%)
Datch			Weight	Talastha	Kanthastha	(Talastha+Kanthastha)	
B1	37 hrs. 35 min	100°C -520°C	200 g	141 g	26 g	83.5%	16.5%
B2	37 hrs. 35 min	100°C-520°C	200 g	136 g	20 g	78 %	22%
В3	37 hrs.35 min	100°C -520°C	200 g	141 g	20 g	80.5%	19.5%

DISCUSSION

There are few critical issues in the preparation of Sameera Pannaga Rasa. Frequently percent yield of Kanthastha (neck of the bottle) and Talastha (bottom of the bottle) product varies, which may also alter the composition of ingredients of either products. As ingredients of formulation are classified under schedule E (1) and are poisonous, if unprocessed. Hence this probable variation in nature of product may be of great significance in view of safety as well as efficacy. Further Manahshila being explosive in nature and all the ingredients (Rasagandha Kajjali, Haratala, Manahshila) are sulphur containing, hence variation in temperature pattern may affect the pressure of gas inside the bottle after corking significantly, if optimum characteristics of completion of Kupipaka during preparation of Sameera Pannaga Rasa are not strictly and precisely monitored. This may lead to blast of bottle leading to accident, loss of product as well as is serious inhalational safety concerns too. Previous researchers have also reported reduction in yield with increase in maximum temperature given during its preparation. However they reported batch failure due to blast of glass bottle. Hence in present study, corking was not done and glass bottle was allowed for self corking by avoiding insertion of hot rod inside neck of *Kupi* and allowing block at bottle neck by product sublimation itself, thus reducing chance of significant increase of gaseous pressure inside the bottle and thus avoided blasting. Batch size was also comparatively less (200g) for similar dimensions of glass bottle. The maximum temperature for product preparation was also kept less (520°C) than that of previous studies so as to get more product yield.[15]

Sameera Pannaga Rasa was prepared with the composition of Sh. Parada, Sh. Gandhaka, Sh. Haratala, Sh. Manahshila and Sh. Somala using the reference of AFI-1. Equal amount of Sh. Parada and Sh. Gandhaka were grounded in edge runner around 20hrs to attain Siddhi Lakshana (specific characters) of Kajjali i.e. fine-smooth texture, black in color and lusterless in appearance. Fine powders of Sh. Haratala, Sh. Manahshila and Sh. Somala were added respectively into Kajjali and trituration was done till mixture became homogeneous. Sh. Haratala must be added in fine powder form otherwise it becomes dificult to make it luster

free. Bhavana (levigation) of Tulasipatra swarasa (leaves juice of Ocimum sanctum Linn.) was given to Kajjali. An average 630ml of Tulasipatra swrasa was used for Bhavana of 1kg of Kajjali. Heat was given for 37:35 hrs in three phases i.e. Mridu agni (mild heat) for 11:35hrs, Madhyamagni (moderate heat) for 19:07hrs and Tivragni (high heat) for 6:03hrs in vertical electric muffle furnace in each 200g batch (3 batches) of Sameera Pannaga Rasa. Mild whitish fumes with characteristic smell of *Tulasi patra swarasa* at 150°C temperature, yellowish & white fumes with characteristic smell of Gandhaka and melting of Kajjali was observed at temperature of 220°C-290°C. Kajjali felt soft upon probing with cold shalaka, got molten and started raising upwards. Shiny reddish colored crystals were seen on inner wall of Kupi (glass bottle) and dark orange color along with mild whitishness was observed on Sheeta Shalaka (cold iron rod) in between the temperature of 320°C-400°C. In between 410°C-500°C temperature, deposition in sequence of black, orange, faint yellow & white was seen on Sheeta Shalaka, boiling of Kajjali was visible inside the Kupi with the help of torch light and few, fine globules of condensed, sublimed mercury were seen on Sheeta Shalaka. Compactness of Kajjali was revealed on Sheeta Shalaka and molten grayish material inside Kancha Kupi attached on Sheeta Shalaka which has tendency to get hard at room temperature. At temperature of 520°C, deposition of Sheeta Shalaka was observed in sequence of black (3/4)-reddish orange-yellow-grey (1/4) respectively and clear golden layer was seen on the surface of boiling Kajjali. At the stage when blackish deposition takes place on cold rod suspended in the middle of the bottle space, deposited blackish material (Sulphur/organosulphur compounds) remains semi liquid even at room temperature for some time and it is very elastic which can be drawn to wires. This is evidence of change of form of Sulphur (possibly beta sulphur / its compounds). Yellow deposition is usually crystalline which is similar to Alpha (rhombohedral) form of sulphur. Whitish deposition is probably due to evaporation of Arsenic vapors initially and on the verge of completion of heating process just before self corking, due to mercury. But there is difference in whitish deposition of sublimates at initial stage and final stage. The whitish deposition at the end of heating process leads to formation of lustrous globules of free mercury, where as initial whitish deposits are amorphous powder with slight elasticity upon rubbing. Thus this cold iron rod test reflects significant chemical processes at different levels inside the Kupi in accord with sublimation temperatures of intermediate products formed during heating process. Raising band width of lower blackish band and vice a versa with other bands with subsequent heating suggests slowly raising of temperature level at different heights inside the Kupi. After this, coin was placed over mouth of glass bottle for a while which showed condensed vapors of

free Hg. Sheeta Shalaka (cold iron rod) as well as Tapta Shalaka (hot iron rod) was inserted into the bottle space throughout the process as per need. Electric muffle furnace was switched off and Kupi was left for self cooling. Final Kanthastha (neck portion of bottle) product was obtained shiny and reddish in color; and the Talastha (bottom portion of bottle) product was porous, dark greenish black and compact in nature. Obtained total yield from each batch of 200g was- 167g (141g of Talastha and 26g of Kanthastha), 156g (136g Talastha and 20g Kanthastha) and 161g (141g Talastha and 20g Kanthastha) respectively. Total duration of 37:35hrs. of active heating with maximum temperature of 520°C was required for the preparation of Sameera Pannaga Rasa in EMF (Vertical Electric Muffle Furnace) with desirable quality. An average yield of Sameera Pannaga Rasa was observed as 80.66% (Talastha+ Kanthastha). Loss of product can be assumed due to evaporation of mercury (Parada) during Paka and during breakage of Kancha Kupi.

CONCLUSION

Sameera Pannaga Rasa was prepared by following the reference of Ayurvedic formulary of India, with modifications i.e. heating through vertical electric muffle furnace at different three phases of heating temperature. Maximum 520°C temperature was given for 37:35hrs heating process. Finally an average of 80.66% (*Talastha+ Kanthastha*) compact, tasteless *Talastha Sameera Pannaga Rasa* was obtained. This present method may be considered as the standard operating process for the preparation of *Sameera Pannaga Rasa* in vertical electrical muffle furnace for the batch of 200g of *Kajjali*.

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