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SEM EDX Analysis of *Swayamagni Loha Bhasma*

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ABSTRACT

Rasashastra is a special branch of *Ayurveda* where in different Herbs, Mineral and Metals are used in various formulations.These metals and minerals are subjected for different purification methods to make it suitable for internal administration. The *Marana* is the procedure in which the purified metals or minerals are further triturated with specific drugs and subjected to heat (*Puta*). For various Herbs, Metals and Minerals the applications of *Puta* is essential to reduce the particle size for its better absorption in the body. *Swayamagni Rasa* is the herbo-mineral preparation were in, self generated heat transforms the particle size from *Loha Churna* into *Bhasma* form. In this study Iron fillings where used to prepare the *Bhasma* by subjecting it to *Samanya* and *Vishesha Shodana* and later on doing the procedure of *Marana* according to *Rasachintamani*. During the process of *Shodana* care has to be taken while heating the iron fillings throughout the procedure and measured quantity of liquid media for quenching has to be maintained for doing *Shodana* in bulk. During the process Iron fillings tend to flush over the face during each *Nirvapa* which has to be dealt with precaution. Thereafter analysis of the *Swayamagni Rasa* was analysed using SEM-EDX. It was observed that the percentage of Oxygen content in *Loha Bhasma* increased there by stating it to be in Oxide form and The particle size ranges around 173.3 nanometre scale which proves it being in its minutest form.

Key words: *Swayamagni Rasa, Marana, Niragni, Loha Bhasma, Dhanyarashi.*

INTRODUCTION

Ayurveda is the science of life aiming to achieve healthier life Physically, Mentally and Spiritually.[1] *Rasashastra* is a special branch of Ayurveda where in different Herbs, Mineral and Metals are used in various formulations. During Vedic period, much importance was given to Herbal drugs for therapeutic purposes. In due course of time, drugs of Metal and Mineral origin came into existence, which led to the establishment of Herbal, Mineral and Herbo-Mineral formulations.

The *Marana* is the procedure in which the purified metals or minerals are further triturated with specific drugs and subjected to heat (*Puta*). For various metals and minerals the applications of *Puta* is essential to reduce the particle size. The current trend of giving *Puta* is most widely accepted throughout the pharmaceutical companies. The classical method of giving *Puta* using cow dung cakes is replaced by the Electric Muffle Furnace which is more feasible when compared to Classical method due to unavailability of cow dung cakes, the available once are of varied size and shape which again hinder in the process of giving *Puta*.

The current trend of giving *Puta* and preparing *Bhasma* is well known, but our *Acharyas* have explained the *Niragni* method of *Loha Marana* which will save the fuel and energy, if done precisely and systematically. The *Swayamagni Rasa* is one of the *Niragni* method of *Marana* Procedure for *Loha dhatu*., It is one of the *Anubhuta Yoga* for *Marana* of *Loha.*

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Materials and Methods

This involves the following procedures:

a) *Samanya Shodhana* of *Loha*.[2]

b) *Vishesha Shodhana* of *Loha*.[3]

c) Preparation of *Dviguna Kajjali*.[4]

d) Preparation of *Swayamagni Rasa*[5]

a) *Samanya Shodhana* of *Loha*

Name of the Practical: *Samanya Shodhana* of *Loha*.

Reference: *R.T 15/4-6*

Date of preparation: 10/12/2018

Date of completion: 09/01/2019

Instruments

- Iron vessel
- Steel vessel
- Spatula
- Cloth
- Gas stove
- Strainer

Ingredients: *Ashuddha Loha* (Iron fillings): 750g

Liquid Media: Q.S

Media

- *Kanji*
- *Takra*
- *Kulattha Kwatha*
- *Gomutra*
- *Tila Taila*

Procedure

- *Ashudda Loha* (Iron fillings) was taken in an Iron vessel and was heated in *Teevra Agni*, till it became red hot.
- It was then quenched in specific liquid media placed in a stainless steel vessel.
- After cooling down, *Loha* was taken out from the vessel and again put in the Iron vessel and heated till it becomes red hot. This process was repeated 3 times in each media.
- Temperature of *Loha* during red hot state was noted.
- Weight of the *Loha* was measured repeatedly.
- Time taken for each process was noted.

Observations

Table 1: Tabulation of changes observed during the *Nirvapa* in *Kanji*

SN	*Nirvapa Dravya*	Loss after each *Nirvapa*	Time taken for red hot	Changes in *Loha*	Changes in media
1.	*Kanji*	750g	30min	Took long duration for heating. Colour of *Loha* changed from brown to black. *Loha* became little brittle.	The smell of kanji was evident during quenching. Colour of *kanji* changed from white to brown. The temperature of *Kanji* was increased after quenching of Iron fillings into it. *Kanji* became viscid and slimy after *Shodhana.*
2.	*Kanji*	748g	30min		
3.	*Kanji*	745g	28min		

Table 2: Tabulation of changes observed during the *Nirvapa* in *Takra*

SN	*Nirvapa Dravya*	Loss after each *Nirvapa*	Time taken for red hot	Changes in *Loha*	Changes in media
1.	*Takra*	745g	28 min	*Loha* took comparatively less time to become red hot. Colour of	Foul smell was felt during quenching. Colour of the media
2.	*Takra*	740g	26min		

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3.	*Takra*	738g	24min	*Loha* turned black. *Loha* turned brittle. Some part of *Loha* turned into coarse powder form. Prominent cracks were observed on the surface of Iron fillings.	changed from white to greyish. *Takra* came out from the vessel while quenching. *Takra* split into solid and liquid parts during quenching and solid part settled down at the bottom of the vessel.

Table 3: Tabulation of changes observed during the *Nirvapa* in *Kulatha Kwatha*

SN	*Nirvapa Dravya*	Loss after each *Nirvapa*	Time taken for red hot	Changes in *Loha*	Changes in media
1.	*Kulatha Kwatha*	738g	20min	Colour of *Loha* turned from blackish brown to deep brown. Iron fillings were more brittle. *Loha* turned more into coarse powder form. *Loha* started getting stuck to the ladle. Some powder flew away from the vessel as vapour while quenching.	*Kulatha* smell was appreciated during quenching. Sound and fumes were more observed during *Nirvapa.* *Kulatha Kwatha* became brown to bluish brown in Colour. Its consistency became thicker.
2.	*Kulatha Kwatha*	735g	20min		
3.	*Kulatha Kwatha*	729g	16min		

Table 4: Tabulation of changes observed during the *Nirvapa* in *Gomutra.*

SN	*Nirvapa Dravya*	Loss after each *Nirvapa*	Time taken for red hot	Changes in *Loha*	Changes in media
1.	*Gomutra*	725g	14min	Iron filings were turned in to *Loha churna, churna* turned finer. *Loha* was dark brown in colour *Loha* took considerably less time to become red hot. *Gomutra* smell was appreciated in *Loha.* *Loha* was more brittle	*Gomutra* colour changed from light yellow to dark brown. Comparatively more Sparkles were observed during quenching. A pungent smell was coming out during quenching. Media took longer time for cooling.
2.	*Gomutra*	720g	10min		
3.	*Gomutra*	719g	8min		

Table 5: Tabulation of changes observed during the *Nirvapa* in *Tila Taila*

SN	*Nirvapa Dravya*	Loss after each *Nirvapa*	Time taken for red hot	Changes in *Loha*	Changes in media
1.	*Tilataila*	719g	10 min	Colour of *Loha* was completely black. Metallic lustre of *Loha* was lost. *Loha* got	Colour of oil turned light brown in colour. Oil became viscid after *Shodhana*. A pungent smell and
2.	*Tilataila*	744g	13min		
3.	*Tilataila*	742g	16min		

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				fire while heating. Cracks were seen on the surface of *Loha*. Brittleness was increased. *Loha* turned completely into coarse powder form. It took 10 minutes to get completely red hot.	black fumes was observed after quenching.

Precaution

- *Loha* had to be heated in *Teevra Agni*, in order to become red hot.
- The red hot state had to be perceived accurately.
- It was poured carefully into each media to prevent loss.
- *Loha* was allowed to cool down after quenching.
- After quenching, collection of *Loha* was done carefully.

Result

- Total Quantity - 750g
- Loss - 180g
- Quantity after *Shodhana* - 600g

b) *Vishesha Shodhana of Loha*

Name of the Practical: *Vishesha Shodhana* of *Loha*

Reference: *R.T 20/15*

Date of Preparation: 01/02/2019

Date of Completion: 15/02/2019

Instruments:

- Iron vessel
- Steel vessel
- Spatula
- Cloth
- Gas stove
- Strainer

Ingredients

- *Shoditha Loha Churna: 600* gm
- *Triphala Kashaya :* 14 liters

Procedure

- Same as *Samanya Shodhana* of *Loha* (*Nirvapa* in *Triphala Kashaya* 7 times)

OBSERVATIONS

Table 6: Tabulation of changes observed during the *Nirvapa in Tripahala Kwatha*

SN	*Nirvapa* Dravya	Loss after each *Nirvapa*	Time taken for red hot	Changes in *Loha*	Changes in media
1.	*Triphala Kwatha*	600g	17m	A reddish texture was observed over *Loha* during red hot state. A crackly sound came out while heating till it became red hot. Small particles of *Loha* got attached to the Iron pan. Powder of *Loha* was formed as vapour while quenching.	Colour of decoction turned blackish brown. *Kashaya* started to boil during quenching. *Kashaya* overflowed from the vessel.
2.	*Triphala Kwatha*	593	15m		
3.	*Triphala Kwatha*	587	12m		
4.	*Triphala Kwatha*	574g	10m		
5.	*Triphala Kwatha*	560g	10m		
6.	*Triphala Kwatha*	542g	8m		
7.	*Triphala Kwatha*	536g	7m		

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				Colour of *Loha* turned blacker.	

Precautions

- Similar to *Samanya Shodhana* of *Loha*.

Result

Total Quantity - 600g

Loss - 64g

Quantity after *Vishesha Shodhana* - 536g

c) Preparation of *Dviguna Kajjali*

Name of Practical : Dwiguna Kajjali preparation.

Reference : R.T 6/107

Date of preparation : 03/5/2018

Date of completion : 10/11/2018

Duration : 140 days

Instruments

- *Khalva Yantra*
- Weighing Balance
- Spoon

Ingredients

- *Shuddha Parada*: 250gm
- *Shuddha Gandhaka*: 500gm

Procedure

- *Shodita Parada* and *Shodita Gandhaka* were taken in equal quantity.
- It was then triturated in a *Khalva Yantra*.
- The mixture of *Shuddha Parada* and *Shuddha Gandhaka* turned greyish.
- During the process of trituration, few drops of water was sprinkled over the powder for preventing it from spilling.
- Trituration was continued till the powder became greyish black in colour.
- To this mixture, again 250g of *Gandhaka* was added and *Mardhana* was continued till the mixture became fine.
- The test for *Sushlakshnata, Rekapurnata, Nischandratva, Varitaratwa* of *Kajjali* was analysed and stored.

OBSERVATIONS

Table 7: Tabulation of changes observed during preparation of *Kajjali*

SN	No. of hours	Changes in Colour	Observation
1.	3rd	Grayish	The mixture was easy to triturate. Course powder form
2.	12th	Grayish black	The *Parada* and *Gandhaka* got mixed up completely. The powder became little soft.
3.	17th	Turned slightly blackish	*Parada* globules disappeared. When rubbed between the fingers, silvery Particles were observed. The *Kajjali* was becoming fine and softer
4.	54th	Turned almost to black	under sunlight, free mercury particles were visualized. The *Kajjali* became very soft and fine.
5.	82nd	Blackish in colour	*Kajjali* fulfilled the *Nischandratva*, *Rekhapurnata*, *Varitara* tests were passed. *Kajjali* formed was very fine, smooth & lusterless.

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Precautions

- To prepare *Kajjali, Gandhaka* should always be taken in fine powder form.
- Trituration should be done slowly and carefully to prevent the loss.
- Few drops of water should be sprinkled over *Kajjali* to prevent spillage during trituration.
- *Khalva* should be kept covered when the process is not in progress.

Results

- No. of days taken : 140days
- Total time taken for preparation of *Kajjali* : 82hrs
- Weight of *Kajjali* obtained : 695gm
- Total wt. loss : 55gm.

Cause of weight loss

- Spillage of mixture during the process of trituration.
- *Kajjali* gets adhered to the *Khalva* which makes it difficult to collect after completion.
- Little quantity of *Kajjali* was lost while performing its confirmatory test.

Table 8: Tabular representation of examination of *Kajjali*

Organoleptic characteristics	Observation of *Kajjali*
Appearance	Black Colour
Touch	Smooth, Fine & *Rekhapurna*
Smell	Smell of *Gandhaka*
Form	Fine powder
Tests passed	*Nishchandratva*, *Rekapurnata* and *Varitara*

d) Preparation of *Swayamagni Rasa*

Name of the Practical : *Swayamagi Loha bhasma*

Reference : *Rasa.chinthamani* 9/25

Date of Preparation : 11/03/2019

Date of Completion : 05/04/2019

Instruments

- Copper vessel
- Spatula
- Cloth
- *Khalva Yantra*
- *Dhanyarashi*
- Thread

Ingredients

- *Dviguna Kajjali* - 20g
- *Samanya Vishesha Shodhita Loha Churna* - 20g
- *Bhavana Dravya - Kumari Swarasa* (Q.S)
- *Prakshepaka* (all ingredients equals to 10g)
  - *Triphala*
  - *Trikatu*
  - *Jatipahala*
  - *Ela*
  - *Lavanga*

Procedure

- The *Dviguna Kajjali* was taken and added with *Shuddha Tikshna Loha Churna* in a clean *Khalva Yantra*.
- The ingredients are given *Bhavana* with quantity sufficient *Kumari Swarasa*.
- The *Mardana* is continued and the mixture is rolled it into 2 *Golakas*.
- The *Golakas* were wrapped using *Eranda Patra* and tied with the thread.
- The tied *Golakas* are kept in copper vessel and closed with *Eranda Patra*.
- This copper vessel was kept under sunlight for 6hrs.
- The copper vessel was shifted into *Dhanyarashi*, and kept for 8 days.

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- The 9th day, the Copper vessel was taken out of *Dhanyarashi* and weighed.
- The *Golaka* was powdered in *Khalva Yantra* and seived with the seiver.
- The fine *Churnas* of *Prakshepaka* were added and stored.

OBSERVATIONS

Table 9: Tabulation of changes observed in *Swayamagni Loha Bhasma*

SN	Parameters		Observations	
1.	Colour		Blakish colour	
2.	Touch		Very Soft and fine	
3.	Weight after *Bhavana*,		60g (2 *Golaka*)	
			1st *Golaka*	2nd *Golaka*
4.	Weight after taking off from *Dhanyarashi*		21g	22g
5.	Temperature when kept for 6hrs under sunlight	Morning (10am)	34^{0}C	36^{0}C
		Afternoon (2pm)	44.3^{0}C	43^{0}C
		Evening (5pm)	36^{0}C	35.3^{0}C

- After keeping in sunlight, the leaves of *Eranda* were dried completely.
- After removing from *Dhanyarashi*, the *Eranda* smell was appreciated.
- During powdering of *Golaka*, the *Eranda* smell was more felt.
- After the addition of *Prakshepaka*, the smell of *Prakshepaka* was also appreciated.
- The temperature of *Tamra Paatra* was 42^{0}C degree while taking it out of *Dhanyarashi.*
- The powder obtained was very soft and black is colour.
- The *Rekhapurna, Varithara* and *Unama* test was also passed.

Precautions

- The wrapping of *Eranda Patra* to be done properly and tightly or else mixing of *Dhanyarashi* with *Golaka* might take place.
- The *Golaka* with *Tamra Paatra* should be kept in the hot sun to serve the purpose.
- The mixing of *Prakshepaka* should be done properly.

Fig. 1: *Loha* and *Kajjali* - *Bhavana* with *Kumari*

Fig. 2: *Golaka* kept in *Tamrapatra*

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Fig. 3: *Golaka* after removing out of *Dhanyarashi*

Fig. 4: Cut Surface of the *Golaka*

Fig. 5: *Golaka* after removing *Eranda Patra*

Fig. 6: *Varitara Pariksha* of *Swayamagni Loha Bhasma*

Fig. 7: *Unama Pariksha* of *Swayamagni Loha Bhasma*

Fig. 8: *Rekhapuranata Pariksha*

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Fig. 9: *Prakshepaka Dravyas* used in *Swayamagni Rasa*

RESULT

Table 10: Tabulation of Results obtained in *Swayamagni Rasa*

SN	Parameter	Quantity
1.	Total quantity after sieving	40g
2.	Gain	-
3.	Loss	3g
4.	Total quantity after addition of *Prakshepaka*	50g

RESULTS

Table 11: Physico-chemical analysis report of *Swayamagni Rasa*

SN	Parameters	Sample - SP
1.	Solubility	1.2%
2.	Ash Value	98.8%
3.	Acid insoluble Ash	89.3%
4.	Water insoluble Ash	87.2%
5.	Bulk Density	1.178gm/ml
6.	Loss on Drying @105°	1.20%
7.	Ph	3.9

Table 12: SEM-EDX Analysis report of *Swayamagni Rasa*

Element	Weight %	Atomic %
C L	0.41	1.24
O K	20.14	33.79
Mg K	1.03	1.24
Al K	0.00	0.00
Si K	1.09	1.18
S K	3.69	5.36
Mn K	0.02	0.02
Fe K	70.92	56.05
Zn K	0.00	0.00
As L	0.00	0.00
Ag L	0.00	0.00
Cd L	0.00	0.00
Hg M	2.70	1.12

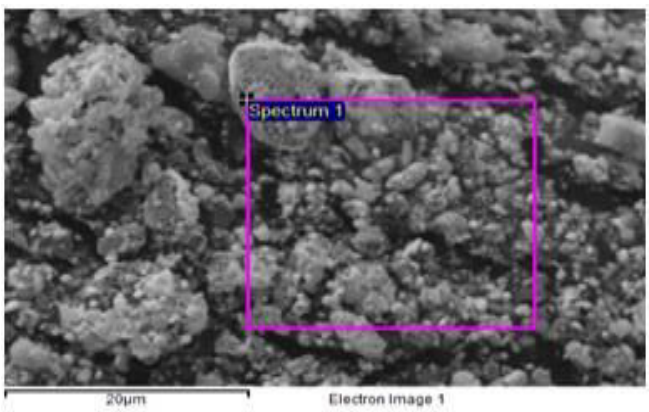

Fig. 10: Spectrum Analysed for *Swayamagni Rasa*

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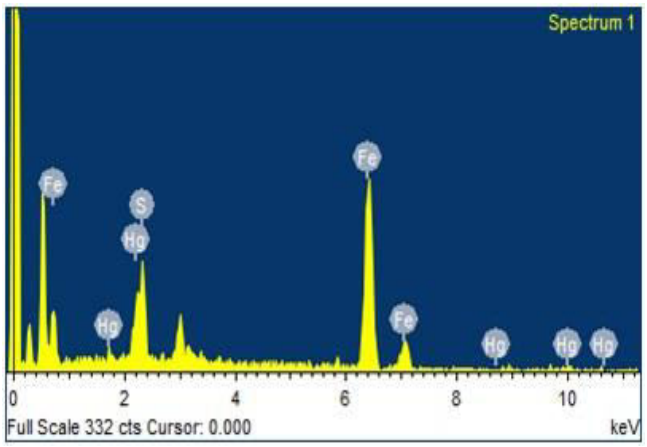

Fig. 11: SEM-EDX imaging of *Swayamagni Rasa*

- In elemental analysis of *Swayamagni Rasa* there was presence of Sulphur and Mercury.
- Mercury weight % is 2.76 and atomic % is 1.12.
- Sulphur weight % is 3.69 and atomic % is 5.36.
- Iron weight % is 70.92 and atomic % is 56.05.
- Oxygen weight % 20.14 and atomic % is 33.79.
- Carbon weight % 0.41 and atomic % is 1.24%.
- The particles present in the preparation were clearly distinctive.
- The particle size were clearly analysed and were in the range of 411.2 to 432.6 nm at a magnification of 10.00 K X.
- The particle size were clearly analysed and were in the range of 173.3nm to 294.8nm at a magnification of 20.00 K X.
- The particle size were clearly analysed and were in the range of 215.7 nm to 244.1nm at a magnification of 30.00 K X.

DISCUSSION

Physico-chemical analysis report of *Swayamagni Rasa*

The Solubility of the samples is 1.2%, The solubility is the property of substance which states how fast a material can dissolve; the smaller the particle is, the faster is it dissolves. 1.2% shows that the sample is easily soluble. (https://en.m.wikipedia.org)

The ash value of *Swayamagni Rasa* is 38.6. Ash value is useful in determining authenticity and purity of sample and also these values are important qualitative standards. On incineration, crude drugs normally leave an ash usually consisting of carbonates, phosphates and silicates of sodium, potassium, calcium and magnesium. Higher Ash value indicates low quality products. The final products contain Carbon, silica and also manganese in trace quantity, which are inorganic. By this value it is evident that the sample was authentic and pure.

The Acid insoluble ash of *Swayamagni Rasa* is 32.6. Acid insoluble ash value helps in determining the contamination of product by earthy matter i.e sand mud etc. The presence of inorganic matter like silica and carbon might have left behind without forming ash. By this value it is evident that the sample is genuine.

The Water insoluble ash of *Swayamagni Rasa* is 26.6. The insoluble content are very less in the samples, By which it is evident that the samples is easily soluble in water. This may be because of light weight of the Bhasmas.

The Bulk density of *Swayamagni Rasa* is 1.17gm/ml. The bulk density is the one which shows how compact the molecules are with each other in a given sample. The sample show significant values which means they are compact with good inter molecular bonding.

The Loss on drying of *Swayamagni Rasa* is 1.40%. This is mainly done to estimate the moisture content of the sample. Presence of more moisture indicates towards lesser shelf life and increased risk of microbial contamination. The sample show lesser moisture content which means it has good shelf and this may be because there are no hygroscopic substance in the samples and no herbal drugs are added.

The pH of *Swayamagni Rasa* is 8.2. The sample is Basic in nature which means that, it is alkaline and easily soluble in water. This may be because the oxides formed in the formulations are alkaline.

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SEM EDX analysis report of *Swayamagni Rasa*

The presence of certain trace elements in the *Swayamagni Rasa,* are evident from the reports of SEM-EDX and the probable reasons might be as follows.

- Magnesium was found in the sample of *Swayamagni Rasa,* this may be because of *Kumari,* as it has trace elements in it and by *Bhavana* procedure Mg might have incorporated.
- The Carbon indicates that the oxidation has taken place in the compound.
- Silicon, Manganese are found in trace quantity which might have formed into its oxides and may have its specific role in the body.
- The presence of oxygen in the sample indicates that the final product might be in the oxide form.
- The Iron might have oxidised in the presence of the atmospheric oxygen and formed into an oxide of iron.

Bhasma Pariksha

Bhasmas are unique preparations in *Rasashastra*, for its preparation *Marana* has to be done. Before the process of *Marana, Shodhana* of the metal has to be done. For the obtainment of a pure *Bhasma*, *Bhasma Pariksha* plays an important role.

Rekhapurna[6] **:** This *Parikshaa* mainly deals with the particle size of the *Bhasmas* and also deals with is softness. The *Bhasma* can only pass this *Parikshaa* when the diameter of the particles is less than the breadth of grooves on the finger surface. Also, deals with the consistency of the particles. The particles of the *Bhasma* only get entangled over the fingers if they are smooth & soft. If they are hard in consistency they will not get adhered to the finger surface though they are sufficiently small.

Varitara[7] **:** The probable cause behind floating of *Bhasma* over water can be described as the atoms of water are bounded with each other due to an attractive force in between them due to which they remain in contact with each other forming a flat surface. When a fine powder is spread on its surface tension of the water doesn't allow the particle to enter/sink thus, keeping them floating. Hence, can be considered as *Laghu* (particles having light weight). *Bhasma* particles which are *Laghu* will float on water and if it contains any unconverted heavy particles of metal it tends to sink. *Acharya Vagbhatta* states that *Bhasma* becomes ready for consumption only if its *Varitara*.

Unama[8] **:** Additional test to confirm the *Varitara Parikshaa*. It is similar to that of *Varitara* test but further on stating the *Laghutva* of the particle which will not allow the grain to sink.

Nischandratva[9] **:** Test is carried out to check the presence of free metal, if its present there will be presence of lustre.

CONCLUSION

Swayamagni Rasa has passed the classical *Bhasma pariksha* that is *Rekha Purnatha, Varithara, Nishchandratwa* and *Unama.* Also, elemental analysis of *Swayamagni Rasa* shows the reduction in the particle size ranging with in nanometre scale. This *Niragni* method of *Loha Bhasma* can be prepared and analysed by Pharmaceutical Industries with the adoptive changes by making it feasible for the current trend.

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