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Validation Of *Samanya* And *Vishesha Shodhana* Procedures Of *Loha* To Develop Their Standard Manufacturing Procedure

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Abstract:

Standards are living documents, which reflect science, technology and systems. To maintain their value, they should be first decided, achieved, set and then periodically validated to maintain their reproducibility. In this study an attempt has been made to validate and develop standard manufacturing procedure of Samanya and Vishesha Shodhana of Loha so that these observations can be reproducible on large scale production too.

Key words: *Bhasma, Loha, Shodhana, Gomutra, validation*

Introduction:

Validation literally means to render the process valid after substantiating known process with a scientific ground in order to deliver a particular product. On other hand, standardization is a process resulting from a consensus based on scientific findings, obtained by parties most affected by it.[1] But the term validation is used more recently replacing standardization and is more globally accepted. For the pharmaceutical processing and formulations contributed by the Rasa Classics, validation stands at the far end of the road, with many milestones to be crossed by bridging innumerable gaps. As for the classics and for their uses, the formulations stand already standardized. What actually meant by validation is substantiation of the existing processes. Once the process is established to be cogent enough to yield persistently unvarying results, that process is said to be valid. The main use of process validation is that it yields the same result (reproducible) as the skilled, even if performed by an unskilled person, and it also helps in batch wise record maintenance for elaborate and lengthy processes. A wide range of *Shodhana* (purification and/or detoxification) methods are prescribed for metals, minerals, and other substances in different classics of *Rasashastra*. These methods not only detoxify the raw material by chemical transformations but also enhance their therapeutic potentials.[2] In pharmaceutical viewpoint this procedure is performed to make the material feasible for further processing. In case of metals and minerals, for *Samanya* (general) *Shodhana*, mainly the procedure of *Nirvapa* (heating and quenching) in different media is advocated in various classics. Whereas for *Vishesha* (specific) *Shodhana* different methods like *Swedana* (boiling), *Nirvapa* in specific medium, etc have mentioned. *Loha* (Iron) is one of such metals which are being used in *Rasashastra* for various purposes like transformation of lower metals to higher ones (*Lohasiddhi)*, for living disease free long life (*Dehasiddhi)*, therapeutics etc. But before using in therapeutics, ancient Acharyas have deliberately focused on its toxic nature. [3] They have warned that it should not be used in *Ashodhita* (unpurified) or *Apakwa* (improperly processed) form and has equated these with poison because of its hazardous effects on the body and due emphasis have been given to its *Shodhana* procedure.[4]

Though the process of validation of such a versatile subject of *Rasashastra* is like harnessing a wild horse, the trend of setting standards for Ayurvedic drugs is fast approaching. But it can be so done only by validating the existing processes. In this study, an attempt has been made to validate *Samanya* and *Vishesha Shodhana* procedures of *Loha* and develop their standard manufacturing procedure.

Materials and Methods:

Procurement of basic raw material:

Loha (Iron Coils) of 0.5 mm diameter was procured from an electrician. There was no coating over the wire. This wire was converted to thin sheet of 36 guage thickness by passing it through a roller

press. The sheet was then converted to pieces of 1×1 cm. *Tila Taila* [*Sesamum indicum* Linn. (Oil)], *Tandula* (*Oryza sativum* Linn.) and *Kulattha* (seeds of *Dolichos biflorus* Linn.) were procured from local market. *Gomutra* (cow urine) was collected from local cow shed. For the purpose of validation, each step of the procedure of each unit operation was considered as an independent procedure and a pharmaceutical proforma was prepared to record minute facts and observations regarding the procedure. For fixing the standards, minimum of three readings of each step were taken.

Pharmaceutical processing like preparation of *Takra* (buttermilk) [5], *Aranala* (sour gruel) [6], *Kulattha Kwatha* (decoction) [7], *Samanya Shodhana* [8], and *Vishesha Shodhana* [9] of *Loha* were considered as unit operative procedures and were carried out as per classical references.

***Samanya Shodhana* of *Loha*:** It was done by the procedure of *Nirvapa* (heating and quenching) sequentially in following media (7 times in each): *Tila Taila*, *Takra*, *Gomutra*, *Aranala*, and *Kulattha Kwatha* [Fig 1]. Equipments used for this purpose were: Gas burner, iron pan, iron ladle, iron rod, stainless steel vessels (2), spatula, measuring mug, weighing machine, pyrometer, and thermometer (360^0C).

Procedure:

Raw Iron coils were taken in iron pan and was heated on gas stove up to red hot and quenched in specific liquid media, which was taken in a stainless steel vessel. After cooling *Loha* was taken out from the vessel, again taken in iron pan and heated and quenched. The process was repeated for 7 times in each media. Every time fresh and gravimetrically same amount of media was taken. Temperature of iron pan, and *Iron coils* during red hot state was noted. Weight of *Iron coils*, volume of media, time taken for each process etc were noted at each time. All the data was recorded in the pharmaceutical proforma. The same procedure was followed for other batches.

***Vishesha Shodhana* of *Loha*:**

Samanya Shdhit Loha was kept in iron pan, heated upto red hot and dipped into a pot containing Triphala kwath. Same procedure was repeated for 7 times. Every time extra Triphala kwath was added to maintain volume of it. After 7 episodes of nirvapan the iron coils were washed with warm water and dried.

Analysis of the samples:

Estimation of iron content in percentage was carried out in the samples of raw *Loha*, *Samanya* and *Vishesha Shodhita Loha*.[10] Different media used for the purpose of *Samanya* and *Vishesha Shodhana* were subjected to physico-chemical analytical parameters like determination of pH[11], specific gravity[12], refractive index[13], total solid contents[14] etc . For this purpose, samples of before and after Shodhana were used.

Tables

Table 1: Details of *Takra* preparation and observations

Batch No.	Curd (g)	Water (ml)	Butter (g)	Amount of Takra obtained	Color	Taste	Smell
1	3000	1500	200	4190	White	Sour	Curdy
2	3000	1500	218	4210	White	Sour	Curdy
3	3000	1500	235	4150	White	Sour	Curdy
Ave.	**3000**	**1500**	**217.67**	**4183.33**	**White**	**Sour**	**Curdy**

Table 2 :- Details of Aranal preparation and observation.

Batch No.	Rice (g)	Water (ml)	Aranala obtained (ml)	Residue (g)	Color	Taste	Odor
1	1500	16000	10200	930	Whitish	Sour	Sour
2	1500	16000	9080	930	Whitish	Sour	Sour
3	1500	16000	9050	930	Whitish	Sour	Sour
Avg.	**1500**	**16000**	**9443.33**	**976.67**	**Whitish**	**Sour**	**Sour**

Table 3 :- Details of Kulattha Kwatha preparation and observation

Batch No.	Rice (g)	Water (ml)	Aranala obtained (ml)	Residue (g)	Color	Taste	Odor
1	3000	48000	8.4	6000	Brown	Astringent	Specific
2	3000	48000	8.1	6000	Brown	Astringent	Specific
3	3000	48000	8.3	6000	Brown	Astringent	Specific
Avg.	**3000**	**48000**	**8.27**	**6000**	**Brown**	**Astringent**	**specific**

Table 4 :- Details of Kulattha Kwatha preparation and observation

Media	Observation	Nirvapa no.							
		1	2	3	4	5	6	7	Avg.
Tila Taila	Time taken by Loha patra to become red hot (Mins:Secs)	9:10	4:08	4:30	3:50	4:20	4:18	3:48	**4:69**
	Temp. of Loha recorded at red hot state (◦C)	460	450	458	466	450	462	460	**458**
	Temp. of Tila Taila immediately after Nirvapa (◦C)	92	88	90	94	90	92	96	**91.71**
	Loss of Tila Taila(ml)	30	27	25	26	24	28	26	**26.57**
Takra	Temp. of Loha recorded at red hot (Mins:Secs)	10:40	6:15	7:30	6:35	5:50	6:48	5:48	**6.8**
	Temp. of Takra immediately after Nirvapa (◦C)	430	440	445	460	450	452	450	**446.71**
	Temp. of Takra immediately after Nirvapa (◦C)	89	88	90	84	90	90	94	**89.28**
	Loss of Takra (ml)	18	17	15	12	16	18	20	**16.57**
Gomutra	Time taken by Loha Patra to become red hot (Mins:Secs)	12:50	8:25	7:50	6:55	6:50	6:46	7:34	**7.87**
	Temp. of Loha recorded at red hot state (◦C)	450	445	435	450	452	456	445	**447.57**
	Temp. of Gomutra immediately after Nirvapa (◦C)	88	84	82	80	79	82	84	**82.71**
Arnala	Temp. of Loha recorded at red hot state (◦C)	430	440	438	435	450	426	438	**436.71**
	Temp. of Arnala immediately after Nirvapa (◦C)	78	82	84	82	79	86	80	**81.57**
	Loss of Arnala (ml)	10	12	14	18	16	12	14	**13.71**
Kulattha Kwatha	Time taken by Loha Patra to become red hot (Mins:Secs)	9:40	6:52	5:40	6:30	5:55	6:20	6:48	**6.55**
	Temp. of Kulattha Kwatha immediately after Nirvapa (◦C)	76	80	79	80	82	84	77	**79.71**
	Loss of KK (ml)	15	12	10	16	18	14	12	**13.86**

Table 5 : Weight of sediment after 7 Nirvapas in each media.

Batch no.	Ashuddha Tamra taken for Shodhana (g)	Sediment found after 7 Nivapas (g)				
		Tila Taila	Takra	Gomutra	Arnala	Kulattha Kwatha
1	500.0	4.0	nm	53.8	112.8	4.9
2	500.0	6.0	nm	55.0	114.0	5.7
3	500.0	10.0	nm	58.0	110.0	8.0
Ave.	**500.0**	**6.66**	**nm**	**55.6**	**112.26**	**6.0**

nm = not measurable

Table 6 : Change in weight of Loha after 7 Nirvapas in each media.

Batch no.	Ashuddha Loha taken for Shodhana (g)	Wt. of Loha after 7 nirvapas (g)					Shuddha Loha obtained	Wt. Loss (g)	Wt. Loss (%)
		Tila tail a	Takra	Gomutra	Arnal	Kulattha Kwatha			
1	500.0	514.1↑	488.9↓	478.4↓	465.4↓	460.62↓	460.62	39.38	7.8
2	500.0	508.8↑	485.7↓	475.9↓	466.5↓	462.75↓	462.75	37.25	7.4
3	500.0	518.7↑	483.8↓	472.6↓	462.8↓	458.92↓	458.92	41.08	8.2
Ave.	**500.0**	**513.9↑**	**486.2↓**	**475.7↓**	**464.9↓**	**460.76↓**	**460.76**	**39.24**	**7.8**

Table 7: Loss in weight of Loha after Vishesha Shodhana.

Batch no.	Samanya Shodhita Tamra taken (g)	Loha after Nirvap (g)	Wt. Loss (g)	Wt. Loss (%)
1	452.0	446.0	6.0	2.38
2	455.0	450.3	4.7	1.75
3	450.0	444.2	5.8	2.28
Ave.	**452.33**	**446.83**	**5.5**	**2.14**

Table 8: Analysis of liquid media before and after Shodhana of Tamra

Sr.No.	Material	Parameter	Before process	After Process
Samanya Shodhana	Tila Taila	Specific gravity	0.92	**0.93**
		Refractive index	1.47	**1.47**
	Takra	pH	4.13	**4.09**
		Total Solid content (%w/v)	5.92	**6.25**
		Specific gravity	1.02	**1.31**
	Gomutra	pH	7.40	**7.67**
		Total solid content (%w/v)	6.03	**7.17**
		Specific gravity	1.04	**1.04**
	Aranala	pH	2.89	**2.97**
		Total solid content (%w/v)	5.17	**5.51**
		Specific gravity	1.50	**1.07**
	Kulattha Kwatha	pH	7.56	**7.20**
		Total solid content (%w/v)	8.43	**8.51**
		Specific gravity	1.04	**1.04**
Vishesha Shodhana	Triphala Kwath	pH	6.10	**7.00**
		Total solid content (%w/v)	6.03	**6.38**
		Specific gravity	1.03	**1.030**

Table 9: Percentage of iron and iron in raw Loha samanya and Vishesha Shodhita Loha

Parameter	Value	After Samanya Shodhana	After Vishesha Shodhana
Iron content as Fe (%w/w)	99.87	90.84	90.20

Observations:

Various observations and average values during the *Takra*, *Aranala* and *Kulattha Kwatha* preparation are described in Tables 1, 2 and 3 respectively. The results and findings of *Samanya* and *Vishesha Shodhana* are described in Tables 4, 5, 6 and 7. The variation in Iron percentage and change in physico-chemical values of media have been presented in Tables 8 and 9.

In *Samanya Shodhana*, after seven *Nirvapa* in *Tila Taila*, borwnish color of *Loha* became black and its metallic luster was lost. Some part of *Loha* got powdered and weight of *Loha* was found increased. Though it was washed with hot water some oil was still sticking to it. Color of Tila Taila became light yellow to light brown and it became more viscid. Oil caught fire with dense fumes during quenching. A pungent smell and a rush of black fumes were observed after quenching. After *Shodhana* in *Takra*, *Loha* caught fire during first heating, but did not catch in next heating. Color of *Loha* became black to blackish gray. Some part of *Loha* was converted to coarse powder form. Some cracks were observed on the surface of *Iron coils*. After quenching, *Takra* started to boil and it separated in solid and liquid parts during quenching and solid part got settled down. Fine particles of *Loha* which were suspended were found difficult to collect due to thickness of *Takra*.

During *Shodhana* in *Gomutra*, color of *Loha* became blackish gray to brown; some *Loha* coils were broken into small pieces and some of it's became coarse powder. Color of *Gomutra* became yellowish to brownish after seven *Nirvapa*. A pungent smell was perceived during quenching.

During *Shodhana* in *Arnal*, color of *Loha* became brown to blackish brown; flakes of *Loha* became more brittle and were transformed to more coarse powder form. Color of *Arnala* became yellowish to brownish and became more viscid after seven quenching. A specific burning smell was coming out during quenching. After *Shodhana* in *Kulattha Kwatha*, color of *Loha* became blackish brown to deep brown; it became more in coarse powder form. Color of *Kulattha Kwatha* became brown to bluish brown and its consistency became thicker. A specific obnoxious smell was coming out during quenching. observed in *Gomutra* but it subsided after 5 minutes. During *Swedana*, color of *Gomutra* turned to blackish green with a strong irritating smell. Blackish color of *Loha* changed to greenish after *Swedana*. [Fig 3]

Discussion:

Shodhana is a procedure of elimination of *Dosha* (impurity/toxicity/flaw) from the drug.[15] The term *Dosha* indicates not only impurities but also all that which makes the drug unsuitable for further process or therapeutic use. In case of metals and minerals, it is a physico-chemical and therapeutic transformation of a substance making it feasible for the next process (*Marana*) or directly for therapeutic use.

During *Samanya Shodhana* procedure, the metallic form of *Loha* gets converted slowly to compound form (FeO) [17]; thus its heat conductivity is reduced which is the reason why *Iron coils* take more time to become red hot as the *Shodhana* procedure advances [Table 4]. Repeated heating and quenching in specific media in this specific order (pH: acidic, acidic, basic, acidic and basic) disrupts the compression- tension equilibrium in the internal structure of *Loha* which leads to cracks on its surface. (Griffith theory [18], Stress corrosion theory [19], and Theory of thermal expansion [20]) As a result of this, some part is converted to coarse and some in fine powder. After each quenching, this powder was found as sediment in media. Since *Aranala* falls under the strong acidic category and also has *Tikshna* (penetrating) property[21], maximum amount of sediment was found in *Arnala* medium (Table 5). Due to thicker consistency of *Takra* medium, sediment was not observed but black particles were seen suspended in it which was difficult to collect. Due to adherence of *Tila Taila*, 1.42 % weight gain was observed in spite of weight loss after *Nirvapa* in *Taila* medium. Use of acidic and alkaline media may cause removal of acid and alkali soluble impurities from the metal.[22]

In *Vishesha Shodhana*, components of *Triphala K* may pierce through the micropores and cracks created during *Samanya Shodhana* and may produce the required change specific to *Loha* for further process. In this process Triphala kwath alters chemical form of Iron content with acidic properties of kwath. This complex gets washed away during washing with hot water. This may be the reason behind the weight loss after this procedure.

In physico-chemical analysis of media no major changes were observed. Total solid content of all the media after *Shodhana* procedure were increased slightly just because of the fine particles of *Loha* which came into it during *Shodhana* procedure. Further analysis like atomic absorption spectrometry of these media is also possible to assess the amount of Iron in them at different stages of *Shodhana*.

Conclusions:

Nirvapa is the commonest method of *Samanya Shodhana* of metals including *Loha*. For 500 g of *Loha*, on an average 3500 ml of each media is required along with 7.8 % of weight loss in this process whereas for *Vishesha Shodhana* of 452.33 g, 5000 ml of *Triphala Kwath* is required along with 2.14% of weight loss. This whole procedure makes *Loha* more brittle and leads to partial conversion into coarse powder form. Data obtained from the present study is reproducible. To avoid the batch to batch variation, this SMP may be applied to large scale. The values of iron percentage in different samples can be taken for quality assurance.

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