Process standardization of *Swarna Makshika Shodhana* (purification) in *Triphala Kwatha* (decoction)

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Abstract

Background: *Swarna Makshika* (SM) is a brassy golden yellowish mineral with chemical composition of $CuFeS_2$ that is widely used in therapeutics to treat various disease conditions such as *Prameha* (diabetes), *Paṇḍu* (anemia), *Kushtha* (skin diseases) and *Jwara* (fever). This mineral needs to be processed by the following specified Ayurveda guidelines in order to make it therapeutically safe and more potent. These processes include *Shodhana* (preliminary process of eliminating unwanted substances)*, Marana* (incineration) and *Amritikarana* (nectorization) that are mandatory and play a crucial role in therapeutics. However, till date, no published reports are available on standard manufacturing procedure of SM *Shodhana*. **Objective:** The objective of this study is to develop the standard manufacturing procedure of SM *Shodhana*. **Materials and Methods:** Methods described in Rasaratna Samuchhaya were followed to perform *Shodhana* process. *Shodhana* of SM was carried out in three batches (600 g in each batch) by seven quenching in *Triphala Kwatha* (TK, decoction of *Terminalia chebula* Retz., *Terminalia bellirica* Roxb. and *Phyllanthus emblica* Linn.) maintaining batch manufacturing records. Organoleptic and physicochemical analysis of media, i.e., TK and SM was carried out. **Results:** After *Shodhana*, golden yellowish luster of SM was completely lost and it turned into dark black coarse powder. The hardness went on decreasing and brittleness went on increasing. Average 532 g of *Shodhita* SM (88.67%) from 600 g of SM was obtained. Average time required for achieving red hot stage was 24.81 min. Analysis of the media revealed an increase in pH, specific gravity, and total solid contents. **Conclusion:** The adopted method for *Shodhana* of 600 g of SM can be considered as easy, convenient and standard.

Keywords: Chalcopyrite, Rasa Shastra, *Shodhana*, standardization, *Swarna Makshika*

Introduction

Ayurvedic system of medicine has its own methodology of drug manufacturing with the highest care toward safeguarding of products. The quality of prepared *Bhasma* (incinerated mineral) depends on the quality of raw material and standard manufacturing practices followed during its preparation. Standardization techniques help in ensuring safety and efficacy of the product. Different pharmaceutical processing techniques such as *Shodhana* (preliminary process of eliminating unwanted substances), *Marana* (incineration), *Jarana* (roasting) and *Amritikarana* (nectorization) are mandatory for manufacturing of Ayurveda medicines, especially when a formulation contains metals or minerals or some poisonous substances in its composition. *Shodhana* process is said to remove soluble impurities from raw material, adds some organic materials, and reduces toxicity to the great extent.[1]

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Swarna Makshika (SM) is an important mineral used in Ayurveda medicines and *Swarna Makshika Bhasma* (SMB) possesses *Vrishya* (aphrodisiac), *Rasayana* (immunomodulation), *Yogavahi* (targeted drug delivery), *Swarya* (good for voice) and *Chakshushya* (helpful in eye diseases) properties. It alleviates disorders caused by *Tridosha* (*Vata*, *Pitta,* and *Kapha*) and is widely used in the treatment of various diseases such as *Kshaya* (emaciation), *Prameha* (diabetes), *Basti Shoola* (bladder pain), *Pandu* (anemia), *Shotha* (edema) and *Kushtha* (skin disorders).[2] This mineral needs to be processed (*Shodhana*) meticulously before its conversion

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into *Bhasma*, and if used in impure form, it manifests a few untoward effects such as *Agnimandya* (dyspepsia), *Vanti* (vomiting), *Vibandha* (constipation), *Krimi* (worm infestation), *Vrana* (ulcer), *Kushtha* (skin disorders), *Netraroga* (eye diseases), *Daurbalya* (generalized weakness), *Kshaya* (emaciation), *Balanasha* (loss of strength) and even *Marana* (death).[3,4] Hence, its processing in terms of *Shodhana* becomes must to make it more potent therapeutically. Earlier scholars have documented on the pharmaceutical methods, but their observations were not comprehensive. Hence, this study is designed to put forward and ensure that those shortcomings are addressed. Earlier scholars have worked on pharmaceutical preparation of SM *Bhasma* with *Nimbu Swarasa* (fresh lemon juice) and *Saindhava Lavana* (rock salt) as *Shodhana* media.[5,6] Around 26 different methods of SM *Shodhana* are found in Ayurveda texts that can be categorized into five basic methods, namely *Swedana* (boiling), *Bharjana* (frying or roasting), *Nirvapa* (heating and quenching), *Mardana* (trituration) and *Putapaka* (incineration). These procedures use more than 35 liquid media.[7] Among these liquids, *Triphala Kwatha* (TK) is more frequently used, easily available, and affordable too. *Triphala* is also emphasized as an *Anupana* (vehicle) for SMB.[8] Hence, using TK as media in the processing may also enhance therapeutic qualities of SMB. Considering this, an attempt has been made to develop standard manufacturing procedures of SM *Shodhana* by *Nirvapa* in TK by following the classical methods.

Materials and Methods

Procurement of raw drugs

Raw samples of SM were collected from central hang wall of Malanjkhand copper project, Balaghat district, Madhya Pradesh, India,[9] and identified to have all acceptable characters such as *Swarna Sannibham* (having golden color and luster), *Guru* (heaviness), *Snigdha* (smooth), *Nishkona* (devoid of angle), and *Kalimam Vikirettattu Kare Ghrushtam* (leaves a black impression when rubbed on palm or white paper) [Figure 1].[10,11] Ground level of the sample collection area was 580 mean reduced level (mRL) and sample was collected from 380 mRL. Collected samples were quartz reef mineralization of chalcopyrite and pyrites. Dried *Triphala* (pericarp of *Terminalia chebula* Retz., *Terminalia bellirica* Roxb., and *Phyllanthus emblica* Linn.) was procured from Pharmacy, Gujarat Ayurved University, Jamnagar.

Method of preparation

The procedure of *Shodhana* was divided into two stages.

Preparation of Triphala Kwatha (decoction)

Individually coarse powder (#10) of *Triphala* was prepared and collected into a stainless steel vessel. The contents (4.2 kg) were added with eight parts (33.6 l) of potable water and left undisturbed overnight. On next morning, the contents were boiled over mild flame maintaining temperature in between 85°C and 95°C and reduced to one-fourth (8.4 l) of its initial volume with constant stirring. The contents were filtered through clean muslin cloth to obtain decoction.[12]

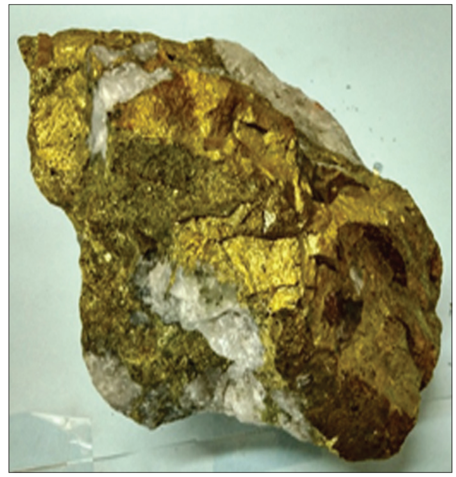

Figure 1: Unit Operative procedure of *Swarna Makshika Shodhana*. Raw *Swarna Makshika* as per the classical characteristics

Shodhana of Swarna Makshika by Nirvapa (quenching)

Raw SM was taken in a clean and dry *Khalva Yantra* (mortar and pestle), pounded well to prepare ½–1-cm small crystals [Figure 2], shifted to a clean and dry iron pan, and subjected to intense heat in charcoal furnace at about a temperature of 800°C–850°C [Figures 3 and 4]. Subsequently after achieving red hot stage [Figure 5], it was quenched in another vessel containing TK [Figure 6]. After self-cooling, SM was collected from the *Kwatha* and dried and the process of quenching was repeated for six more times. At the end of the seventh procedure, the contents were collected carefully, shade dried, powdered, labeled, and stored in airtight containers for further use [Figures 7-9].[13] The brief video of Unit Operative procedure of *Swarna Makshika Shodhana* was also created for clear perception.[14] The whole process was repeated in two more batches. Each time fresh and same amount of TK was taken. Temperature and volume of media before and after quenching were noted. Temperature of coal and SM was monitored by electric digital pyrometer. Weight of SM before and after quenching was noted.

Analysis

Organoleptic parameters

Organoleptic characters, i.e., color, odor, taste and feel of drug upon touch by sensory observations of SM as well as TK before and after *Shodhana* were noted.[15]

Physicochemical parameters

Physicochemical parameters of TK such as pH,[16] specific gravity (Sp. Gr.)[17] and total solid content[18] were determined before and after each *Nirvapa.* pH (5% aqueous suspension), loss on drying[19] and ash value[20] of raw and *Shodhita* SM were carried out according to standard methods as mentioned in the Ayurvedic Pharmacopoeia of India.

Results

During SM *Shodhana*, specific crackling sound was observed when SM was heated during the first quenching process, and few small particles of SM were escaping

Figure 2: *Swarna Makshika* converted to ½–1-cm particles

Figure 3: *Swarna Makshika* subjected to heat

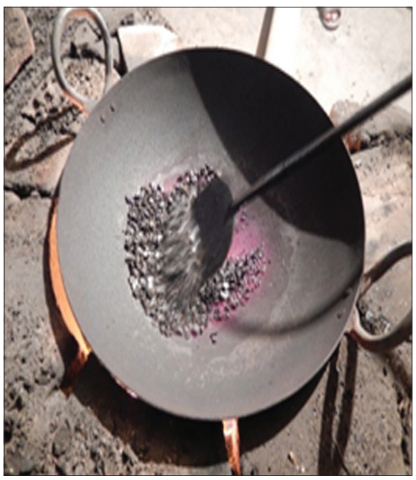

Figure 4: Roasting of *Swarna Makshika* in iron pan

Figure 5: *Swarna Makshika* achieving the red hot stage

Figure 6: Quenching of *Swarna Makshika* in *Triphala Kwatha*

Figure 7: Decantation of *Swarna Makshika*

from the iron pan. Characteristic smell and fumes of sulfur (sulfur dioxide) were observed during the process. Sulfur fumes were found increased at the time of red hot stage of SM and complete cessation of sulfur fumes was observed during the seventh *Nirvapa*. As sulfur fumes produce irritation to the nose and throat, precautions should be taken during the *Shodhana* process. Brassy golden yellowish SM changed to black after the first and second *Nirvapa* and turned to blackish red by the fourth *Nirvapa*. By the end of the fifth *Nirvapa*, it turned to dark black coarse powder. Brown-colored TK was turned to dark black and its consistency became thicker. Characteristic odor was observed during quenching.

Some particles were stuck to the vessel after quenching which needed to collect carefully. Details along with results obtained during *Nirvapa* process, changes observed in the media, and organoleptic characters of SM and TK are depicted at Tables 1-3, while the changes in physicochemical profiles of media and SM has been presented in Tables 4 and 5 and the equipment specifications in Table 6.

Discussion

Shodhana is a procedure of elimination of *Dosha* (impurity/toxicity/flaw) from the drug.[21] The term *Dosha* indicates not only impurities but also all that which makes the drug unsuitable for further process or therapeutic use.

Figure 8: *Swarna Makshika* after the third *Nirvapa*

Figure 9: *Swarna Makshika* after the seventh *Nirvapa*

Table 1: Observations during *Swarna Makshika Shodhana*

Batch	Number of *Nirvapa*	Temperature of coal (°C)	Temperature of SM in red hot stage (°C)	Total time taken to reach red hot stage (min)	Weight of SM (g)		
					Before quenching (g)	After quenching	Weight loss
I	1	845	436	35	600	595	5
	2	832	440	28	595	586	9
	3	847	410	20	586	578	8
	4	825	445	18	578	564	14
	5	816	423	16	564	558	6
	6	850	451	15	558	549	9
	7	812	431	15	549	542	7
	Average	832.43	433.71	21.00	-		58
II	1	811	412	40	600	593	7
	2	837	450	34	593	581	12
	3	822	426	29	581	570	11
	4	832	435	21	570	559	11
	5	819	444	20	559	543	16
	6	849	395	18	543	534	09
	7	820	418	19	534	521	13
	Average	827.14	425.71	25.86			79
III	1	848	428	42	600	592	8
	2	808	408	36	592	583	9
	3	825	430	31	583	577	6
	4	838	385	26	577	564	13
	5	815	433	21	564	553	11
	6	850	390	19	553	546	7
	7	828	441	18	546	533	13
	Average	830.28	416.43	27.57			67
Average of three batches (%)		829.95	425.28	24.81	600	532 (88.67)	68 (11.33)

Table 2: Weight loss and temperature of *Triphala Kwatha* after each *Nirvapa*

Batch	Number of *Nirvapa*	Temperature of *Kwatha* before *Nirvapa* (°C)	Temperature of *Kwatha* after *Nirvapa* (°C)	Loss of media after *Nirvapa* (ml)			
				Before quenching	After quenching	Weight loss	Percentage loss
I	1	33	82	1200	1150	50	4.16
	2	32	79	1200	1140	60	5.00
	3	30	84	1200	1160	40	3.33
	4	31	86	1200	1155	45	3.75
	5	33	78	1200	1150	50	4.16
	6	34	88	1200	1130	70	5.83
	7	32	90	1200	1145	55	4.58
	Average	32.14	83.85	1200	1147.14	52.86	4.40
II	1	36	80	1200	1152	48	4.00
	2	37	83	1200	1126	74	6.17
	3	34	75	1200	1143	57	4.75
	4	35	90	1200	1120	80	6.66
	5	36	85	1200	1132	68	5.67
	6	33	82	1200	1165	35	2.92
	7	35	76	1200	1140	60	5.00
	Average	35.14	81.57	1200	1139.71	60.28	5.02
III	1	38	84	1200	1134	66	5.50
	2	39	89	1200	1121	79	6.58
	3	37	79	1200	1144	56	4.67
	4	35	90	1200	1136	64	5.33
	5	36	85	1200	1150	50	4.16
	6	34	80	1200	1130	70	5.83
	7	36	78	1200	1115	85	7.08
	Average	36.43	83.57	1200	1132.85	67.14	5.59
Average of three batches		34.57	82.99	1200	1139.90	60.09	5.00

Table 3: Organoleptic characters of *Swarna Makshika* and *Triphala Kwatha*

Parameters	*Swarna Makshika*		*Triphala Kwatha*	
	Before *Shodhana*	After *Shodhana*	Before *Shodhana*	After *Shodhana*
Color	Brassy golden yellow	Blackish	Dark brown	Dark black
Odor	Odorless	Odorless	Characteristic	Sulfurous
Texture	Hard crystalline, Shiny	Brittle, course powder	Liquid	Viscous liquid
Taste	Metallic	Metallic	Astringent	Astringent

In case of metals and minerals, it is a physicochemical and therapeutic transformation of a substance making it feasible for the next process (*Marana*) or directly for therapeutic use. It is a mandatory process of metals and minerals that help to expose maximum surface area of drug for chemical reactions and also in impregnation of organic materials and their properties into the drug. This makes the mineral brittle and helps in particle size reduction. There are different types of *Shodhana* methods mentioned in Ayurveda, and *Nirvapa* is one among them.

During *Shodhana,* gravimetrically double amount of TK was taken for quenching. This requirement may change depending on the vessel specifications. Ideally, cylindrical vessels with suitable wide mouth will be justifiable. Ideally, quantity of liquid should be sufficient enough to completely immerse the material being quenched. The process of *Nirvapa* was carried out first by making pieces of SM crystals approximately in ½–1-cm dimensions. Big pieces of SM could not get red hot because of their size during *Nirvapa* process. Considering this, a powder (60# mesh) form of SM was attempted that was adhering to the iron pan and leading to difficulty in quenching and significant loss. Hence, in subsequent practicals, crystals of approximately ½–1 cm were prepared, and the same process was repeated.[22] It was observed that particles of SM became red hot uniformly and do not adhere to the iron vessel and suitable for *Nirvapa.* While performing *Nirvapa*, strong sulfur smell was observed from the first to the fourth cycle of quenching that was due to sulfur dioxide (SO_2) vaporization and oxidation of SM.[23] From the fifth cycle onward, the intensity of odor was reduced, and by the seventh cycle, no odor was observed.

Table 4: Changes in the physicochemical constants of *Triphala Kwatha* before and after *Nirvapa*

Batch	Before/after *Nirvapa*		pH	Specific gravity	Total solid contents (%)
I	Before *Nirvapa*		3.25	1.057	15.5
	After *Nirvapa*	1	3.34	1.058	17.0
		2	3.37	1.063	16.0
		3	3.40	1.067	18.0
		4	3.46	1.074	19.0
		5	3.53	1.079	23.0
		6	3.67	1.080	20.0
		7	3.88	1.086	25.0
		Average	3.52	1.072	19.71
II	Before *Nirvapa*		3.24	1.050	15.1
	After *Nirvapa*	1	3.36	1.062	15.1
		2	3.38	1.060	16.0
		3	3.47	1.072	18.0
		4	3.54	1.076	18.0
		5	3.59	1.079	19.0
		6	3.63	1.081	20.0
		7	3.74	1.084	23.0
		Average	3.53	1.074	21.86
III	Before *Nirvapa*		3.25	1.057	15.3
	After *Nirvapa*	1	3.30	1.059	16.0
		2	3.37	1.060	17.0
		3	3.47	1.075	19.0
		4	3.51	1.070	20.0
		5	3.64	1.080	22.0
		6	3.69	1.080	23.0
		7	3.84	1.090	24.0
		Average	3.55	1.077	20.14
Average of three batches			3.53	1.074	20.57

Table 5: Physicochemical parameters of *Swarna Makshika* before and after *Shodhana*

Parameter	*Swarna Makshika*	
	Before *Shodhana*	After *Shodhana*
pH value (5% aqueous solution)	4.24	4.52
Loss on drying at 110°C (%w/w)	0.67	0.54
Ash value (%w/w)	96.66	91.48

$$2CuFeS_2 + 7.5O_2 \rightarrow 2CuSO_4 + Fe_2O_3 + 2SO_2$$

$$2CuFeS_2 \rightarrow CuS + 2FeS + S$$ [24]

At early stages of *Shodhana,* cracks were seen on the surfaces of SM flakes, and finally, some coarse powder was observed. Repeated heating and cooling of SM flakes causes disruption in compression-tension equilibrium and leads to cracks on the flake surface (stress corrosion theory).[25] During red hot state, different compounds such as pyrrhotite ($Fe_{1-x}S$), pyrite (FeS_2), bornite (Cu_5FeS_4) and chalcocite (Cu_2S) will be formed on the surface of SM flakes.[26] Theory of thermal expansion states that expansibility differs from metal and mineral to compound on heating. In general, expansibility of compound is less than metal.[27] Hence, repeated heating leads to breaking of SM flakes into coarse to fine powder. Copper and iron are converted to oxide form at red hot state by reacting with atmospheric oxygen.[28] Studies reported oxidized iron and copper compound as Fe_2O_3 (ferric oxide) and CuO (cupric oxide), and rest sulfides are FeS_2 (iron disulfide), $FeSO_4$ (ferrous sulfate), Cu_2S (cuprous sulfide) and CuS (copper sulfide).[29] Raw SM may contain physical impurities such as unwanted rock or gangue minerals, typically silicate minerals, or oxide minerals for which there is often no value.[30] These visible impurities were removed by hand picking method before *Nirvapa*. SM is often found with a variety of other trace elements such as cobalt (Co), nickel (Ni), manganese (Mn), zinc (Zn) and tin (Sn) substituting for copper and iron (Fe), selenium (Se) and arsenic (As) substitute for sulfur and trace amount of silver (Ag), gold (Au), platinum (Pt), lead (Pb), vanadium (V), chromium (Cr), indium (In), aluminum (Al) and antimony (Sb).[31,32] It is most likely that many of these elements are present in finely intergrown mineral within the SM (chalcopyrite). During the process of red hot in iron pan on high temperature, chemical impurities such as extra sulfur in the form of sulfur dioxide (SO_2) and arsenic may be get vaporized[13] while other get oxidized and after heating, it was instantly quenched in the TK. It facilitates the media to enter inside the drug easily by which the remaining blemishes will get separated or dissolved in the liquid media as well as the therapeutic property of TK introduced into SM. Instant quenching is important because repeated immediate cooling after heating leads to breaking of the material. Yellowish and golden shining of SM was lost completely after *Shodhana* process and turned into dark black coarse powder.

An average temperature of SM in red hot stage was noted as 425.28°C. Average time required to get red hot was noted around 24.81 min. There was a continuous decrease in the period of reaching to the red hot stage of SM during first to seventh process of *Nirvapa*, which may be due to the conversion of oxides and sulfides of SM. At a later stage of *Shodhana*, surface area of SM was increased due to repeated heating and quenching. Hence, SM took less time to become completely red hot [Table 1]. An average loss of SM was noted as 68 g (11.33%) after *Nirvapa*. After seventh *Nirvapa*, fine particles of SM were found settled to the bottom of vessel. Loss was observed during *Nirvapa* as some fine particles were lost through filtering and a fraction might have entered into TK. Documentation of weight changes is an essential part for the drug standardization. Assessment of final weight with relation to initial weight will give an idea to choose the weight of the initial material to procure the desired quantity of finished product. Large-scale production will substantially decrease the expenditure in relation to the quantity as the expenditure on labor is not going to change by increasing quantity up to some level.

The formation of oxides scales is also responsible for increase in pH after every purification steps. Oxides (Fe and Cu oxides) are mainly basic in nature, so it can raise the pH after every

Table 6: Equipment and their specifications in the process of SM *Shodhana*

Equipment and their specifications	Value
Iron pan/Kadhai (for *Swarna Makshika*)	
Depth	25 cm
Diameter	100 cm
Circumference	250 cm
Weight	4.5 kg
Stainless steel vessel (for *Triphala Kwatha* during quenching)	
Depth	21 cm
Diameter	17.5 cm
Circumference	55 cm
Capacity	5 l
Stainless steel vessel (for *Triphala Kwatha* preparation)	
Depth	45.72 cm
Diameter	58.42 cm
Circumference	194.31 cm
Capacity	50 l
Thermocouple	
Ceramic made with 1200° with digital display	
Iron ladle	
Length	106.68 cm
Stainless steel ladle	
Length	30 cm
Cotton cloth	
Length	100 × 100 cm
Weighing balance	
Maximum capacity	25 kg
Minimum capacity	10 g
Measuring jar	
Maximum capacity	2 l
Stainless steel tray	
Length	20 cm
Breadth	20 cm

quenching. Specific gravity of TK before *Nirvapa* was 1.057 and after *Nirvapa* was 1.074. Specific gravity is defined as the weight of a given volume of the liquid at the stated temperature as compared with the weight of an equal volume of water at the same temperature.[17] Increase of specific gravity in TK after *Nirvapa* is due to the presence of SM particles in TK. Total solid content of the TK before *Nirvapa* was 15.5 and after *Nirvapa* was 20.57. The total solids are the measure of the combined content of all inorganic and organic substances contained in a liquid. The soluble content determines the amount of constituents in a given sample of drug.[18] Total solid content of the media after *Shodhana* procedure were increased slightly just because of the fine particles of SM which came into it during *Shodhana* procedure. Analysis of the media revealed that increase in pH, specific gravity, and total solid contents [Table 4] justifies this. An average 60.09 ml (5%) loss was observed in TK, the loss which might be due to the intense heat of the material at the level of which some fraction of moisture gets evaporates. As fuel, an average 2-kg soft coal and 1-kg hard coal for each process of *Nirvapa* is needed. Average temperature of coal during red hot stage was noted as 829.95°C.

Role of *Triphala*

Triphala mainly consists of tannins, gallic acid, chebulinic acid, ascorbic acid (Vitamin C) and phenolics.[33] Ascorbate (one of a number of mineral salts of ascorbic acid [Vitamin C]) has been known to antagonize the intestinal absorption of copper. More recent studies have characterized a post absorption role for ascorbate in the transfer of copper ions into cells. The vitamin reacts directly or indirectly with ceruloplasmin, a serum copper protein, specifically stabilizing the bound copper atoms and facilitating their cross-membrane transport. Ascorbate at physiological levels and above impedes the intracellular binding of copper to Cu, Zn and superoxide dismutase. The mechanism is unclear but nonetheless suggests both positive and negative regulatory functions for ascorbate in copper metabolism.[34,35] Ascorbic acid increases the bioavailability of Fe by converting Fe^{+3} to Fe^{+2}, while phenolics can reduce the bioavailability of Fe by binding, for example, tannins. Excess of ascorbic acid and a lack of dietary tannins have both been suggested as contributing to clinical/pathological Fe storage disease.[36] *Triphala* is a mild laxative and thereby counteracts the constipating property of iron and copper. Thus, it is mentioned in maximum *Shodhana* procedures of various metals and minerals.

Precautions

1. Procurement of raw SM should be done as per the classical characteristics of acceptable variety
2. Preparing crystals of around ½–1 cm will facilitate *Nirvapa* procedure
3. Protective measures should be taken to avoid contact of red hot particles of SM coming out of the iron pan
4. Mouth and nostrils are to be covered with a mask to avoid sulfur fumes. Eyes are also to be covered appropriately (protective glasses)
5. On observing red hot stage, contents are to be quenched immediately without delay
6. While quenching, maximum care should be taken to avoid loss and accidents
7. Level of liquid should be sufficiently maintained to immerse the mineral completely
8. Collection of SM should be done carefully after quenching. Fresh liquid should be used in each experiment
9. All observations should be recorded carefully during each experiment.

Conclusion

Process standardization and quality control is a mandatory task and an essential requirement for good manufacturing practices to assure the quality and quantity of final product. Repeated quenching in the decoction of *Triphala* imparts some microelements that contribute to nullify possible toxic nature of raw material and help in increasing therapeutic attributes of SM. Average 88.67% yield was obtained after

Shodhana. Results of the present study ensure the uniformity of the operative procedures, and those can be followed in SM *Shodhana* successfully.

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Conflicts of interest

There are no conflicts of interest.

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