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Comparative Physico-chemical and Stability Studies of Lyophilized and Simple Shunthi (Zingiber officinale Rosc.) Powder

Author: Priyanka K Patel[1]

Co Authors: Atanu Anil Bairagi[2]

[1,2]Upgraded Dept. of Rasa Shastra and Bhaishajya Kalpana Dept., Govt. Ayurved College, Vadodara, Gujarat, India

ABSTRACT

Introduction: *Shunthi* as ingredients is extensively used by Ayurvedic physicians and pharma companies as single drug or in formulations. India led in ginger production with 34% of the global production, 2017. *Shunthi* hold a unique place in global traditional treatments and food supplement hence economically very important. No study in respect of lyophilized *Shunthi* has been done yet. Hence the stability study of *Shunthi* would help to establish the use of Lyophilized powder in formulations. **Aim and Objectives:** To develop the analytical parameters of Lyophilized *Shunthi* powder and compare the stability with simple *Shunthi* powder. **Materials and Methods:** Both Lyophilized and Simple *Shunthi* powder were analysed separately for organoleptic characters, physico-chemical parameters like loss on drying, water soluble extractive, alcohol soluble extractive, total ash, and acid insoluble ash. Assay of 6 – Gingerol by HPLC, Limit test for heavy metals and microbial limit test was also done. Stability study was carried out in $30^{o} \pm 2^{o}$C tem. and 75 ± 5% RH at 0, 3, 6, 9, 12 months. **Result and Conclusion:** Lyophilized *Shunthi* powder has better extractive value than Simple *Shunthi* Powder. Assay of 6-Gingerol showed significant result. Lyophilized *Shunthi* powder was found more stable than Simple *Shunthi* powder.

Key Words *HPLC, Lyophilization, Physio-chemical parameters, Shunthi, Stability*

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INTRODUCTION

Shunthi (*Zingiber officinale* Rosc.) is a potential herbs having immeasurable beneficial quality in different aspect have been used by our ancestors. Ginger has been an important ingredient in Ayurvedic, Chinese, and Tibb-Unani herbal medicines. *Acharya Bhavmishra* said that *Shunthi* is *Ruchya* (add taste to the food), *Aamvatadhni* (Help in Rheumatoid arthritis), *Pachini* (Digestive), *Vrusya* (Increasing the quality of semen), *Svarya* (Promotes voice effect), *Kaphavatahara* (Balance kaphavata), *vibandhnut* (Relieves constipation) and used in *Vami* (vomiting), *Swasa* (Brethlessness), *Shula* (Pain), *Kasa* (Coughing), *Hridayamya* (Heart dieases), *Arsha* (Piles), *Shlipada* (Elephantitis), *Shotha* (Oedema), *Anaha* (Distended abdomen), *Udarvata* (Bloating)[1]. According to Indian Pharmacopoeia, *Shunthi* is carminative, antiemetic, anti-inflammatory, hepatoprotective

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and used in *Agnimandya*[2]. Because of such versatile uses, *Shunthi churna* is extensively used by Ayurvedic physicians and Ayurvedic pharma companies as single drug or in formulations. India led in ginger production with 34% of the global production, 2017. Nigeria, China, and Indonesia also had substantial production[3]. *Shunthi* hold unique place in the traditional herbs based remedies and also economic growth of the nation.

'*Kalpana*' is the new modulation made to enhance the potency of the drug. Nowadays people are very cautious about the taste that exists with powder. Moreover, in modern pharma the powder is absolute in dosage form. But considering the potent value & totality of the preparations, *Churna Kalpana* still remains as the most important form of Ayurvedic preparations.

Lyophilization or freeze drying is a process in which water is removed from a product after it is frozen and placed under a vacuum, allowing the ice to change directly from solid to vapour without passing through a liquid phase[4]. The basic idea of Lyophilization is to "lock in" the composition and structure of the material by drying it without applying the heat necessary for the evaporation process. Drying takes place at very low temperatures, so that decomposition and enzyme action is inhibited, particularly hydrolysis is minimized. The material is frozen, so that the final dry product is a network of solid occupying the same volume as the original material. Thus, there is no case-hardening and the product is light and porous and due to remaining more active principle the dose is lower than simple powder[5]. Lyophilized powder retains the active principles, phytochemical, enhances shelf life and stability, decreases the moisture content with minimum heat, product are easily dissoluble thus increases the bioavailability and helps to lower the dosage of the medicine.

As far as Ayurveda is concerned the powder formulation made with the help of Lyophilization has major advantage over traditional process of powder preparation. With the above advantages is it evident to bring this technology for the Ayurvedic formulation preparation.

Ginger has different chemical components like Amaldehyde, Gingerol, Shogaol, and Paradol etc[6]. Indian pharmacopoeia has admitted gingerol as an active ingredient in *Shunthi*. So HPLC was also done to measure it in both samples and other physiochemical parameters for quantitative and qualitative data.

Stability is necessary for development of a pharmaceutical product. So present study has been planned to conduct real time stability study by physiochemical parameter of simple and lyophilized *Shunthi* powder at 0, 3, 6, 9, 12 months.

AIMS AND OBJECTIVES

To develop the analytical parameters of Lyophilized *Shunthi* powder. To compare the stability between Lyophilized *Shunthi* powder and standard *Shunthi* powder.

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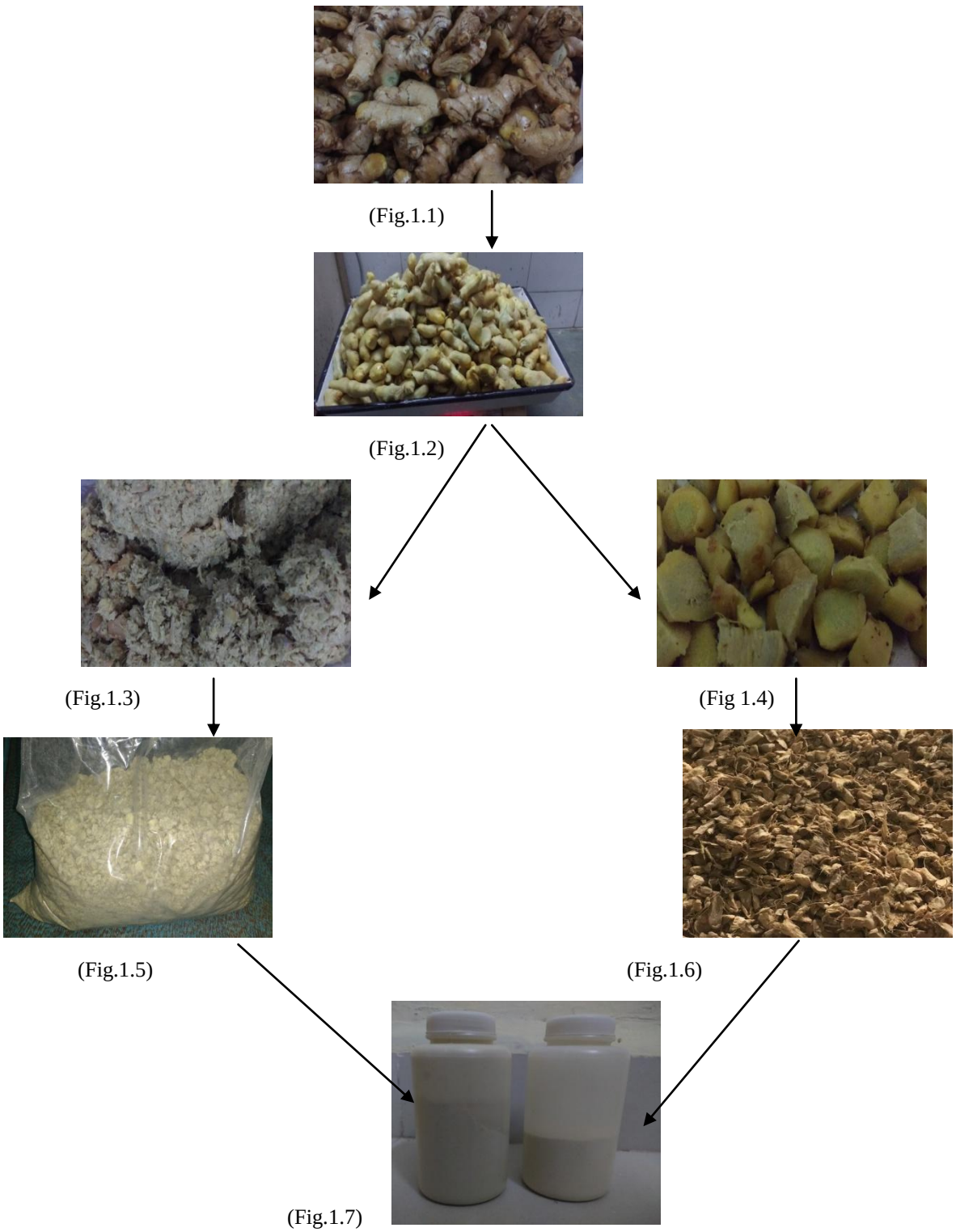

Figure 1 *Shunthi* Powder Preparation

MATERIALS AND METHODS

Procurement of Drug:

Raw *Adraka* was procured from Khanderao local market of Vadodara and authenticated by subject expert in Pharmacognosy division of Food and Drugs Laboratory, Vadodara. The study was carried out during February 2018 to March 2019.

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Preparation of drug: (A Concise Step by Step Methodology) [Figure 1]

Simple *Shunthi* powder (SSP) preparation:

First the manual sorting of *Adraka* was done and separated from foreign unwanted matters Then it was subsequently washed with lukewarm water and kept over a porous tumbler for water to drain out. Skin of *Adraka* was peeled and it was cut into small pieces with the help of a knife. These pieces were dried under sun for 12days. After a complete drying, it was pulverized. This Simple powder was sieved with 85 no. mess and packed in airtight LDPE containers. Containers of 50gms powder were then labeled and stored in control chamber for stability studies.

Lyophilized *Shunthi* powder (LSP) preparation:

For lyophilized *Shunthi* powder, first manual sorting of *Adraka* was done and separated from foreign unwanted matters. Then it was subsequently washed with lukewarm water and kept over a porous tumbler for water to drain out. After washing, skin of *Adraka* was peeled with the help of a knife. After peeling, *Adraka* was put in a grinder for size reduction. *Adraka* paste was collected after grinding, very similar to *Adraka Kalka*. This *Adraka Kalka* was finally subjected to lyophilization in the Freeze Dry Machine at Freeze Dry Systems Private Ltd, Savli, Vadodara. After the Freeze Drying was completed, this Lyophilized *Shunthi* was obtained and subjected for pulverization in grinder. Sieve the lyophilized powder with 85 no. mess and packed in airtight LDPE containers. [Table No.1]

Table 1 Powder Preparation.

Procedures		Sorting and cleaning	Grinding	Freeze/Sun drying	Grinding and sieving	Final Results
Time taken	SSP	3.30 hrs	-	12 days	90 min	14 days
	LSP	4 hrs	35 min	6 hrs	75 min	3 days
Wt. Before (Kgs)	SSP	10	-	8.478	3.034	10
	LSP	10	8.398	8.176	1.668	10
Wt. After (Kgs)	SSP	8.478	-	3.034	2.846	2.846
	LSP	8.398	8.176	1.668	1.543	1.543
Wt. loss (%)	SSP	15.22	-	64.21	6.19	71.54
	LSP	16.02	2.64	79.59	7.49	84.57

ANALYTICAL STUDY

Both Lyophilized and Simple *Shunthi* powder were analyzed with various analytical parameters. Stability study was carried out in 30^{o} ± 2^{o} C temperature and 75 ± 5% Relative Humidity at 0, 3, 6, 9, 12 months.

Organoleptic Characters

Color, Odour, Taste

Physio-Chemical Parameters

Loss on drying (LOD)[7], Water Soluble Extractive (WSE)[8], Alcohol Soluble Extractive (ASE)[9], Total Ash (TA)[10], Acid-Insoluble Ash (ASA)[11]

Assay of 6 - Gingerol by HPLC[12]

Microbial Limit Test[13]

Heavy metal Analysis by Atomic Absorption Spectroscopy (AAS)[14]

Assay of 6 – gingerol by HPLC:

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Preparation of Standard Solution(S): 10.0 mg of standard 6 gingerol was weighed accurately in a 5 ml volumetric flask. After that menthol HPLC grade was dissolved up to mark. 1 ml of above solution was dissolved in 2 ml volumetric flask with the help of menthol HPLC grade. It was sonicated for 5 minutes. Then it was filtered using 0.22 micron syringe filter and used for HPLC analysis. **Preparation of Sample Solution (T):** 1.0 gm of sample powder was weighed accurately in a 100 ml volumetric flask and menthol HPLC grade was dissolved up to mark. Then it was sonicated for 5 minutes. It was filtered using 0.22 micron syringe filter and used for HPLC analysis. **Chromatographic Conditions:** HPLC system: Shimadzu UFLC Modular, Stationary Phase: C18**,** Column Temperature: 40^0C**,** Filtering system: 0.22 µ syringe filter**,** Run time: 60 minutes**,** Sample Application Volume: 20 µL**,** Mobile phase (MP): Acetonitrile: Water (55:45)**,** Flow Rate: 1.3 mL/min**,** Wavelength: 280 nm**,** Mode of Separation: Isocratic

RESULT

Organoleptic characters (colour, odour and taste) of SSP and LSP were remained same during stability period. There were no major changes in it. Pungency and colour of LSP were approximated as *Adraka*. Study result of Simple *Shunthi* powder and Lyophilized *Shunthi* powder at different months were given in Table no. 2,3,4,5.

Table 2 Result of Physico - Chemical Parameters of SSP and LSP

Sr. No	Physico-Chemical Parameters of SSP		0 month	3 month	6 month	9 month	12 month
1.	LOD (% w/w)	SSP	7.70	7.94	9.05	9.90	13.07
		LSP	1.51	3.01	6.61	10.51	10.59
2.	WSE (% w/w)	SSP	12.29	11.76	11.19	10.52	9.92
		LSP	19.45	20.71	20.75	20.77	20.77
3.	ASE (% w/w)	SSP	5.75	6.10	6.84	6.85	6.95
		LSP	9.11	9.17	9.43	9.73	9.91
4.	TA (% w/w)	SSP	4.17	4.17	4.22	4.19	4.10
		LSP	5.40	5.60	5.65	5.51	5.31
5.	AIA (% w/w)	SSP	0.93	0.97	0.94	0.91	0.88
		LSP	0.81	0.75	0.74	0.79	0.73

Table 3 Result of Assay (%) of SSP and LSP

Month	SSP	LSP
0 month	0.200 %	0.970 %
12 month	0.025 %	0.571 %

Table 4 Result - Microbial limit test of SSP and LSP

	Month	SSP	LSP
Total plate count	0 month	189cfu/gm	115cfu/gm
	12month	618cfu/gm	29 cfu/gm
Total Yeast &Mould Count	0 month	237cfu/gm	80cfu/gm
	12 month	Absent	Absent
E. coli	0 month	Absent	Absent
	12month	Absent	Absent
Salmonella	0 month	Absent	Absent

Table 5 Result - Heavy metal analysis of SSP and LSP

Heavy Metal Content	Permissible Limits[15]	SSP	LSP
Lead	10 ppm	Not Detected	Not Detected
Cadmium	0.30 ppm	Not Detected	Not Detected
Arsenic	3 ppm	Not Detected	Not Detected
Mercury	1 ppm	Not Detected	Not Detected

DISCUSSION

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Result of Physico-chemical parameters are mentioned in Table No. 2. According to IP, LOD should not more than 12% w/w. LOD of SSP was 13% w/w at 12th month so it didn't fulfil the quality parameter at that month. LOD was rapidly increased in LSP than SSP because of hydroscopic nature of LSP. So lyophilized powder should be packed in airtight containers. LOD of LSP was found lesser than SSP. So due to more moisture SSP spoiled earlier than LSP. According to API, WSE of *Shunthi* should not less than 10% w/w. Both powders fulfill the criteria at initial stage but at 12-month SSP didn't comply quality criteria. WSE of SSP decreased gradually with time while LSP maintained its value during stability period. WSE of LSP was almost double than SSP so we can make effective *kalpanas* out of lyophilized powder. No more difference found in ASE of SSP and LSP with time. According to API, ASE should not be less than 3.0% w/w. So, both powders complied the quality criteria. The result of Total ash and Acid insoluble ash on both SSP and LSP suggested that there were no more inorganic impurities in samples than API required criteria. According to API, Permissible limit of Total ash is not more than 6.0 % w/w and Acid insoluble ash is not more than 1.5% w/w for *Shunthi*.

Assay of 6-Gingerol by HPLC shows that SSP's result (0.27% w/w) was under limit (Table No. 3) so it was unproductive when LSP had good amount (0.97% w/w) in compare to IP standard (Not less than 0.8% w/w) at initial finding. At 12 month 6-Gingerol in SSP was found 0.025% w/w and in LSP 0.571% w/w. With these finding, said that LSP retained much 6-Gingerol than SSP.

Table No. 4 shows that in SSP Total Plate Count was 189 cfu/gm, Total Yeast & Mould Count was 237cfu/gm and other E. coli, Salmonella, S. aureus, P. aeruginosa were absent at initial month where as in LSP Total Plate Count was 115 cfu/gm, Total Yeast & Mould Count was 80 cfu/gm and other microbes were absent. At 12 month in SSP Total Plate Count was 618 cfu/gm, Total Yeast & Mould Count, E. coli, Salmonella, S. aureus, P. aeruginosa were absent while in LSP Total Plate Count was 29 cfu/gm and Total Yeast & Mould Count, E. coli, Salmonella, S. aureus, P. aeruginosa were absent. Total plate count was increased in SSP with time, so according to this data we can say that SSP would have shortly crossed the standard limit and became useless due to microbial growth. Where as in LSP, Total plate count was decreased with time, may be due to controlled temperature, at that temperature they couldn't grow.

Plants are the main link in the transfer of heavy metals from the contaminated soil to humans. So, it is necessary to test heavy metal in herbs which are going for medicinal use. Heavy metal analysis of SSP and LSP was performed initially by Atomic Absorption Spectroscopy. Table No. 5 shows that Heavy metals (Lead, Cadmium, Arsenic, and Mercury) were not detected in both samples.

CONCLUSION:

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Lyophilized *Shunthi* powder preparation consumed less time than traditional sun drying. 64.21% of weight lost found during Sun drying and 79.59% of weight lost during lyophilisation process. So 15.43% yield of SSP and 28.46% yield of LSP were obtained.Values of physico-chemical parameters were better in Lyophilized *Shunthi* powder and were found to be more stable than Simple *Shunthi* powder that will help in minimizing the dose, increase the effect and quicker the action.

Further scope of study:

In future clinical studies for efficacy, pharmaceutical studies for extraction & isolation and pharmacological studies for dose reduction can be proposed for lyophilized *Shunthi* powder.

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