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Comparative Pharmaceutico Analytical Study of *Madhyama Paka* and *Khara paka* in *Sneha Paka* WSR to *Katupila Taila*

Samip Shah[1]*, Prashant Bedarkar[1], V. J. Shukla[2], B. J. Patgiri[1], Mehul Mehta[3]

Abstract

Introduction: Different types of *Snehapaka* have been mentioned in *Sarangdhar Samhita* such as *Mrudu Paka, Madhyama Paka, Khara Paka, Dagdha Paka, Ama Paka, and Gandha Paka*. Further, different uses of *sneha* according to its *paka* had been mentioned in which *Madhyama Paka* and *Khara Paka* are stated to be used for internal (*Pana*) and external (*Abhyang*), respectively. **Aim:** The present study was carried out to discriminate type of *paka in Katupila taila* on the basis of classical and modern parameters, namely, *Paka pariksha,* organoleptic-physicochemical parameters, and chromatography. **Results:** Average 934.8 g (93.48% yield) and 845.33 g (84.53% yield) of *Katupila Taila madhyama paka* (KPTM) and *Khara paka,* respectively, was obtained from batch size of, 1 L of *Tila Taila* with 212 g of *Kalka dravya* and 4 L of *Kwatha* of *Katupila* stem. Temperature reached up to 92°C and 97°C, respectively, for completion of *Madhyama Paka* and *Khara Paka*. Heating was carried out for 3 consecutive days for both *Paka*. **Discussion:** Average duration between initiation of *Madhyama Paka* and end of *Khara Paka* was 1 h and 44 min. Here, temperature and duration of heating play a major role in the type of *Paka*. It showed difference in the values of physicochemical parameters of both *Paka*. KPTM and *Khara paka* were having specific gravity (0.9179 ± 0.0050 and 0.9294 ± 0.0067), refractive index (1.449 ± 0.009 and 1.474 ± 0.015), acid value (2.908 ± 0.0538 and 1.809 ± 0.0472), iodine value (108.075 ± 2.9793 and 72.875 ± 3.2548), saponification value (169.66 ± 8.9132 and 226.64 ± 6.1567), and BTTT value (19.6 ± 0.3, 28.5 ± 0.5), respectively.

Keywords: *Katupila taila, Khara paka, Madhyama paka, Securinega leucopyrus, Snehakalpana, Snehapaka*

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Introduction

Traditional Indian medicine is as old as human civilization. In India, the skill of drug making has been well known since ancient times as evident from *Rigveda*. Standard drug making was of deep concern in Ayurveda since ancient time.[1] Herbal medicinal products are quite popular and have a long history of practice. The major hurdles in their official recognition and prevalence as established medical systems are inadequate quality control aspects.[2] *Sneha Kalpana* is one of the most common preparations in *Bhashjya Kalapana*. Different types of *Paka* can be done in *Sneha Paka,* namely, *Mrudu Paka*, *Madhyama Paka,* and *Khara Paka*. Utilization of each *Paka* is varying according to the ancient classics. Most commonly *Madhyama Paka Taila* is used internally, whereas *Khara Paka* is used externally.[3] An attempt has been made to record pharmaceutical manufacture and differentiate *Madhyama Paka* and *Khara Paka*on the basis of temperature, duration of *Paka*, physicochemical parameters, BTTT value, and high performance thin layer chromatography (HPTLC). Standardization of *Katupila Taila* is desirable for its greater recognition and acceptance. The standardization study of formulation never be achieved by one or two parameters and hence is preferable to achieve it in a multidisciplinary way. With this in mind, the study has been undertaken to develop pharmaceutical standardization with six batches of *Katupila taila madhyama paka* (KPTM) and *Khara Paka.* Temperature pattern, duration of heating, organoleptic characters, and physicochemical parameters were recorded and evaluated in all six batches of KPTM, *Khara Paka,* and *Tila Taila* (TT) (Sesame oil). HPTLC fingerprinting was carried out for *Madhyama Paka* and *Khara Paka Katupila Taila*.

Aims and Objectives

The objectives of the study are as follows:

1. To develop standard manufacturing procedure of KPTM and *Katupila Taila Khara Paka* (KPTK)
2. To develop analytical profile of KPTM and KPTK.

[1]Department of Rasashastra and Bhaishajya Kalpana, Institute for Post Graduate Teaching & Research in Ayurveda, Gujarat Ayurved University, Jamnagar, Gujarat, India

[2]Department of Pharmaceutical Chemistry, Institute for Post Graduate Teaching & Research in Ayurveda, Gujarat Ayurved University, Jamnagar, Gujarat, India.

[3]Department of Quality Control, Indian Institute of Ayurvedic Pharmaceutical Sciences, Gujarat Ayurved University, Jamnagar, Gujarat, India.

Corresponding Author: Samip Shah, Department of Rasashastra and Bhaishajya Kalpana, Institute for Post Graduate Teaching & Research in Ayurveda, Gujarat Ayurved University, Jamnagar, Gujarat, India. E-mail: sam6453@gmail.com

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Materials and Methods

Pharmaceutical Study

Total six batches of KPTM and *Khara Paka* were prepared.

Collection and authentication of raw drugs

Katupila stem was collected at its fruiting stage from Nana Thavaria Villege, near Vijarkhi dam, Jamnagar (Gujarat) in June month [Figure 1a].

Katupila (*Securinega leucopyrus*) was authenticated in Department of Pharmacognosy, IPGT and RA, Gujarat Ayurved

University; bearing Herbarium Voucher's Specimen no. 6317 [Figure 1b].

TT (FSSAI grade) was procured from the pharmacy, Gujarat Ayurved University. Formulation was prepared in Department of Rasa Shashtra and Bhaishajya kalpana laboratory, I.P.G.T and R.A, Jamnagar.

Preparation of Katupila Kwatha (KPK)

Physical impurities from raw material were removed manually. *Yavkuta* of *Katupila* stem was prepared in pharmacy, Gujarat Ayurved University. Then, it was soaked in water overnight for 15 h. Next day, it was subjected to heat over *Mandagni* to *Madhyamagni* (80–100°C) until half part is reduced. *Kwatha* was filtered in warm condition through cotton cloth and stored in stainless steel vessel for further process [Table 1 and Figures 1d-f].

Preparation of Katupila Kalka

Katupila Yavakuta was taken in grinder machine to prepare fine powder. Fine powder was triturated with just sufficient quantity of water to convert in to paste form. Boluses of *Kalka* were kept in stainless steel vessel for further process [Table 2 and Figures 1c].

Figure 1: Pharmaceutical steps of *Katupila Taila* preparation. (a) Herbarium of *Katupila* (*Securinega leucopyrus*). (b) Preparation of *Kalka*. (c) Soaking of *Katupila* stem *yavakuta* in water. (d) Preparation of *Katupila Kwatha*. (e) Filtration of *Katupila Kwatha*. (f) Heating of *Tila Taila*. (g) Addition of *Katupila Kalka* boluses. (h) Addition of *Katupila Kwatha* in mixture of *Kalka* and *Tila taila*. (i) Phenodgam stage. (j) *Mardanevartikotpatti Madhyama Paka*. (k) Burnt/Charred *Kalka* of *Kara Paka*. (l) Filtration of *Katupila Taila*

Preparation of KPTM and Khara Paka

Katupila Taila was prepared as per the general preparation of *sneha paka* [Table 3]. TT in the mentioned quantity was taken in a stainless-steel vessel and heated over mild flame (100°C for 8 min) till complete evaporation of moisture and then heating was stopped. After that boluses of *Kalka* were added to TT at 80°C. After mixing of *Kalka* into *Taila*, the 4 L of KPK was added and the mixture was heated over a mild to medium heat. Heating was continued maintaining the temperature in between 87°C and 101°C with intermittent stirring. The mixture was left undisturbed after discontinuation of direct heating till next heating, thus intermittent heating was carried out over 3 days (Total 13 h (*Madhyama paka*) and 13 h and 30 min (*Khara Paka*) of active heating and 52 h (*Madyama paka*) and 54 h (*Khara paka*) of cooling to room temperature). Mixture was stirred continuously to avoid the settling of solid contents and overheating due to possibility of settling down. Heating was continued on 3rd day until *Sneha Siddhi Lakshana* of each *Paka* was obtained. After obtaining desired *Sneha Siddhi Lakshana*, heating was discontinued and oil was filtered immediately through two folded cotton cloth in hot stage. After cooling, the prepared oil was kept in a vessel for a day for sedimentation allowed for settlement of suspended particles and filtered again and stored in labeled air tight, non-reacting bottle, at room temperature, in dark place till analysis [Figure 1g-l].

Analytical study

Analytical evaluation of KPK, KPTM, and KPTK was carried out to develop standards for the reproducibility of product. The samples were analyzed on the basis of organoleptic characters. It includes sensory characters of drug, that is, *Sparsha* (Consistency and texture), *Rupa* (Color), and *Gandha* (odor). Physicochemical parameters such as specific gravity @ 25°C, refractive ndex, pH, total solid contents, acid value, iodine value, saponification value, Bellier's turbidity temperature test,[4] and HPTLC were done for analysis of *Katupila Taila* at pharmaceutical chemistry laboratory of I.P.G.T and R.A, Jamnagar.

HPTLC

Sample preparation.[5]

5 g of sample of *Katupila Taila* was accurately weighed and introduced in a 250 ml flask filled with a reflux condenser. Solution of 2 g of potassium hydroxide in 40 ml alcohol was added and heated on a water bath for 1 h. After shaking frequently, the contents of flask were transferred with addition of 100 ml hot water while the liquid is still slightly warm shook carefully with three successive quantities each of 100 ml of solvent ether. Three extracts were combined in the separating funnel containing 40 ml of water and swirl gently. The lower layer was allowed to separate and rejected. The ether layer was washed with two successive quantities each of 40 ml of water and with three quantities each of 40 ml 3% w/v solution of potassium hydroxide. Each treatment was being followed by a wash with 40 ml of water. Finally, ether layer was washed with successive quantity each of 40 ml of water until the aqueous layer is no longer alkaline to phenolphthalein. Ether layer was transferred to a weighed flask. Separating funnel was washed out with solvent ether and distilled off with aid of gentle air current, cooled, and weighed. Then, it was diluted with diethyl ether and stored.

Sample application was done using CAMAG linomat 5.

HPTLC of both the *Katupila Taila* was carried out using solvent system Toluene: Diethyl ether (93:7 v/v). HPTLC study was performed for the normal phase separation of components of product [Figure 2].

Chromatographic study (HPTLC) was carried out at 254 nm and 366 nm to establish fingerprinting profile. Derived plates were visualized at 254 nm and spots at 366 nm before spray and after spray [Table 4, Figure 3].

Observation and Results

Yield of dried *Katupila* stem from 22.100 kg quantity of fresh stems was 17.170 kg, that is, 77.69%. While preparing *Yavakuta* of *Katupila* stem, 15.500 kg quantity of *Yavakuta* was obtained with 70.13% yield. Total 2.090 kg (92.88%) of fine powder was derived from 2.250 kg of *Yavkuta* with 92.88% yield and 7.12% tailing. Mean diameter and length of stem were 0.49 ± 0.19 cm and 3.4 ± 1.1 cm, respectively. Average weight of *Kalka* was 212 g after mixing with water [Table 5]. Prepared *Kalka* was light brown in color, having characteristic smell. During heating of TT (Sesame oil), foaming was observed which was probably due to moisture content of *Taila*. When temperature of oil reached at 75°C, boluses of *Kalka* were added and fried for around 10–12 min. The *Kalka dravya* was added while discontinuation of heating to avoid overheating of *Kalka dravya*. After mixing of *Kalka* into *Taila* and 4 times of KPK, (4000 ml) was added and again heating process was done with maintaining temperature in between 87°C and 101°C with occasional stirring [Table 6]. After 15 min of heating, bubbling was visible with experience of peculiar odor of *Katupila*. After half an hour of heating, contents became dark brownish in color and upper layer of mixture was light brown in color with more viciousness of mixture and frothing. After 1 h of heating, light brown color of mixture was changed to dark brown having excessive frothing was observed during the process. The *Taila* was heated intermittently maintaining temperature in between 87°C and 101°C for 3 days. On the 3rd day, after 11 h of heating process, *Phenodgama* (appearance of excessive foam) was observed. *Kalka* was examined at regular intervals. *Mridu Paka* and *Madhyama Paka* stages were observed at specific temperature which presented in Table 7. Consistency of *Kalka* became sticky to touch with no moisture and was able to make *varti* (*Mardanevartikotpatti*) and smooth in texture. At this stage of *Madhyama Paka*, *Taila* was filtered through cotton cloth without squeezing and in hot stage to get maximum yield of final product. On an average, total duration and yield for *Madhyam Paka Katupila Taila* and *Khara PakaKatupila Taila* were observed to be approximately 13 h, 939 ± 2.6457 ml and 13.5 h, 845.33 ± 22.50 ml, respectively after 3 days.

Table 1: Observations of six batches of KPK

Batches >	*KPK A*	*KPK B*
Initial Quantity of *Katupila* stem (g)	1000	1000
Size of the pieces (cm)	3.4±1.1	3.4±1.1
Diameter of the pieces (cm)	0.29±0.19	0.29±0.19
Total Quantity of water (ml)	8000	8000
Total time for soaking (h)	15 h	15 h
Temperature (after 1 h)	100.75±0.6	100.75±0.6
Duration (min)	153.16±6.52	138.83±3.18
Obtained *Kwatha* (ml)	4281.66±84.71	4160.83±58.25
Fresh residue (g)	1898.16±35.17	1718±62.67

Table 2: Physicochemical characteristics of KPK

Parameters	*KPK A*	*KPK B*
Specific gravity	0.9855±0.0039	0.9869±0.0021
Refractive index	1.333±0.0074	1.324±0.0036
pH	5.63±0.0103	5.54±0.0097
Total solid contents (%w/v)	0.2916±0.0055	0.2989±0.0067

KPK: Katupila Kwatha

Table 3: Formulation composition of *Katupila Taila*

No.	*Ingredients*	*Latin/English name*	*Part used*	*Quantity*
Kalka dravya				
1	*Katupila churna*	*Securinega leucopyrus* Muell.	Stem	167 g (1/6 part)
Sneha dravya				
2	*Til Taila*	*Seasamum indicum*	Seed oil	1000 g (1 part)
Drava dravya				
3	*Katupila Kwatha*	*Securinega leucopyrus* Muell.	Stem	4000 g (4 part)

Specific Observations

Organoleptic characters: The organoleptic characters of KPTM and *Khara Paka* are presented in Table 7.

Physicochemical parameters: Physicochemical parameters of KPK are presented in Table 2.

Comparative physicochemical parameters of *Til Taila* (Sesame oil), KPTM, and *Khara Paka* are presented in Table 8.

Discussion

As *Sneha Paka* is carried out with *Kwatha*, quantity of *Kalka* was taken as 1/6th as that of oil.[6]

Temperature reached up to 92°C during completion of *Madhyama Paka* and 97°C for *Khara Paka*. Heating was carried out for 3 consecutive days for both *Paka*. Total duration of preparation of *Katupila Taila* was 13 h and 13 h and 30 min for *Madhyama Paka* and *Khara Paka*, respectively. Duration between initiation of *Madhyama Paka* and end of *Khara Paka* was 1 h and 44 min. Specific Sneha *Paka Pariksha* of *Kalka* was done for *Paka* in which *Mardanevartikotpatti* (formation of wig while rolling the *Kalka* between index finger and thumb) was observed in *Madhyama Paka*. While in *Khara*

Table 4: HPTLC of *Katupila Taila Madhyama Paka* and *Khara Paka*

Katupila Taila Madhyama Paka				
Solvent system	*Wavelengths*	*No of spots*	*Rf value*	*Area under curve*
Toluene (9.3) : Diethyl ether (0.7)	254 nm	3	0.03, 0.13, 0.51	71.91, 13.53, 14.56
	366 nm	2	0.03, 0.13	80.96, 19.04
Katupila Taila Khara Paka				
Solvent system	*Wavelengths*	*No of spots*	*Rf value*	*Area under curve*
Toluene (9.3) : Diethyl ether (0.7)	254 nm	3	0.03, 0.14, 0.52	50.68, 23.09, 26.23
	366 nm	3	0.03, 0.14, 0.48	55.98, 38.05, 5.98

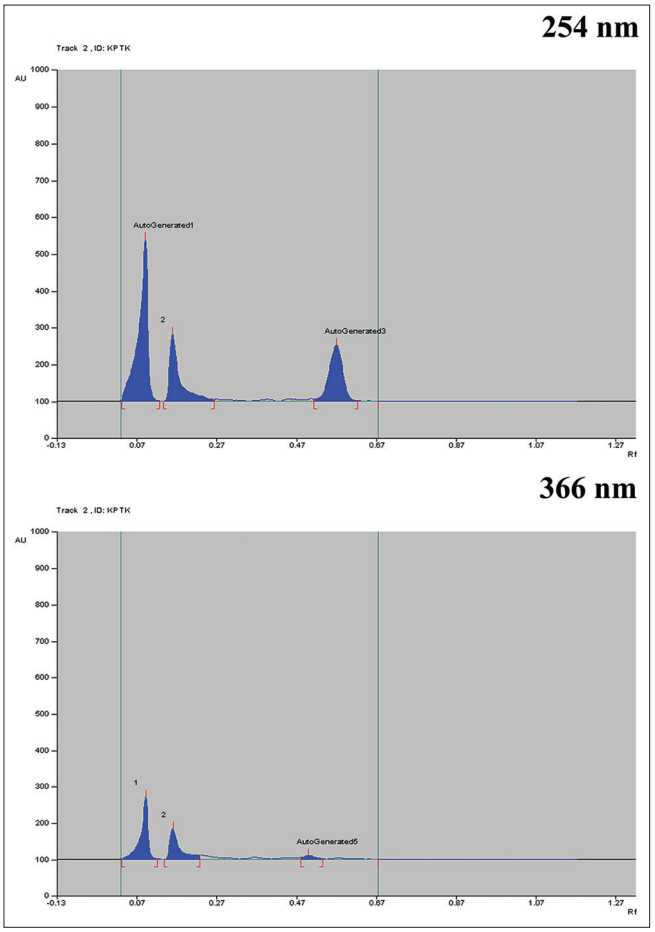

Figure 2: HPTLC of *Katupila Taila Khara Paka*

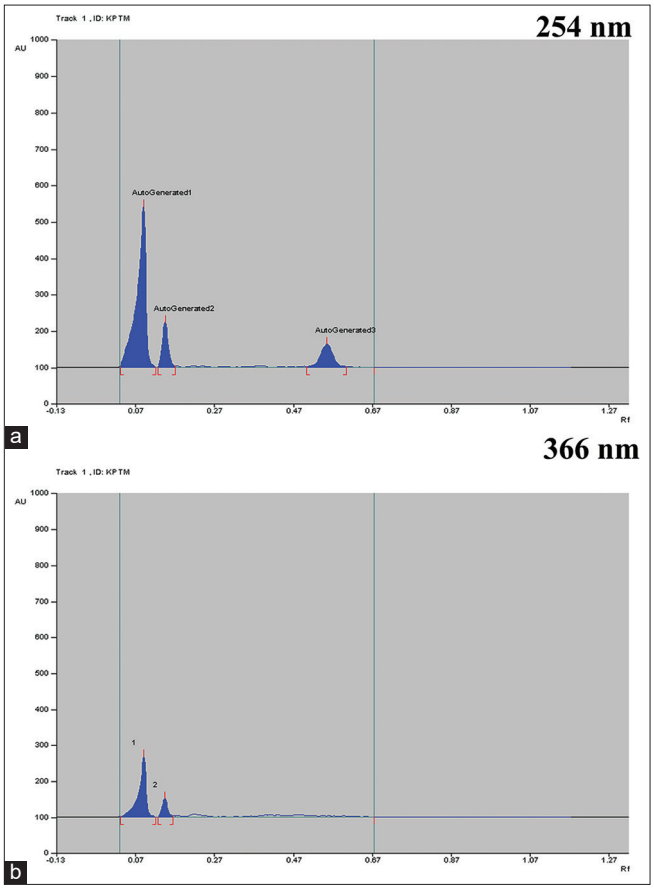

Figure 3: (a-b) HPTLC of *Katupila Taila Madhyama Paka*

Paka, *Kalka* was getting powdered while rubbing and *varti* could not be formed. Minute quantity of *sneha* was observed in *kalka* of *Madhyama Paka* and burnt smell was observed in the *kalka* as well as *taila* in *Khara Paka*. An average 6.51% ± 0.70 and 14.25% ± 2.42 of loss was found during preparation of *Katupila Taila Madhyam Paka* (KPTM) and *Khara Paka,* respectively. Comparatively, more loss in *Khara Paka* than *Madhyama Paka* may be due to more absorption of *Taila* in to *Kalka* and complete evaporation of the aqueous contents, evaporation of formed volatile fatty acids, and other volatile compounds during successive additional heating as that of *Madhayam paka* 30 min more duration and temperature.

As per tabled data [Table 8], the standards (all analytical physicochemical parameters) of TT chosen for the present study are within limits for sesame oil prescribed by regulatory authorities, which matches with API,[7] BIS,[8] and CODEX[9] standards. Parameter values of TT, KPTM, and KPTK were recorded and compared. Refractive index of samples, that is, KPTM (1.449 ± 0.009) and KPTK (1.474 ± 0.015) were minor different. More refraction of light in KPTK compared to KPTM may be due to presence of more suspended particulate matter, and change in concentration of extracted chemical moieties, and addition/extraction of different chemical constituents into sesame oil or neo formation of different chemical constituents into sesame oil.[10] Specific gravity may also get altered (increased) due to some logically relevant reasons in KPTK (0.9294 ± 0.0067) as that of KPTM (0.9179 ± 0.0050) which may be more suspension of solid particulate matter in to prepared *Sneha*, new generation of complexes with extracted constituents of formulation ingredients or their derivatives having comparatively more density.

Comparatively, lower acid value of KPTK (1.809 ± 0.0472) as compared with KPTM (2.908 ± 0.0538) suggests formation of more complexes of generated free fatty acids (FFA) and trans fatty acids (TFA) with phytoconstituents of drug *Katupila*.[6]

More saponification value in KPTK (226.64 ± 6.1567) as that of KPTM (169.66 ± 8.9132) may suggests formation of more shorter chain fatty acids, with lower molecular weight and thus supporting fast and better absorption of generated phytoconstitutes of oil.[11] Iodine value is a measure of the amount of unsaturation (number of double bonds) in a fat.[12] Increased iodine value observed in KPTM (108.075 ± 2.9793) as that of KPTK (72.875 ± 3.2548). BTTT values were 19.6°C ± 0.3 and 28.5°C ± 0.5 for KPTM and KPTK, respectively.

Acid value and iodine value should get increased with increase in duration of heating and or intensity of heating. However, in the present study, they are found reduced. Heating leads to auto oxidation, generation of FFA, and unsaturation. However, contrary to this during deep frying, it is documented that in general, deep-fat frying decreases the content of unsaturated fatty acids in frying fat and oil. Researchers (Tynek *et al*., 2001 and Cuesta *et al*., 1991) have found a relative loss of the C18:2 fatty acid and a decrease in the iodine value of oil after heating due to more intensive thermo-oxidative transformations that occur compared to heated oil containing food. The decrease in the iodine value can be attributed to the destruction of double bonds by oxidation, scission, and polymerization.[13] Iodine value is measure of degree

Table 6: Observation of six batches of *Katupila Taila* at different temperature

Sr. No	*Observation*	*KPT M*		*KPT K*	
		TEMP (°C)	*Time*	*TEMP (°C)*	*Time*
1.	Starting Temp.	27	0 min	29	0 min
2.	On addition of *Kalka*	88	21 min	87	15 min
3.	On addition of *Kwatha*	84	30 min	81	25 min
4.	During *Phenodgama*	87	11 h	87	11 h 10 min
5.	During *Mrudu Paka*	89	12 h	90	12 h
6.	During *Madhyama Paka*	91	13 h	92	12 h 50 min
7.	During *KharaPaka*	-	-	97	13 h 30 min
8.	Duration between initiation of *Mrudu Paka* and end of *Madhyama Paka*	-	72 min	-	58 min
9.	Duration between initiation of *Madhyama Paka* & end of *KharaPaka*	-	-	-	104 min
10.	Duration (start from *MruduPaka*) for appearance of *Kalka Varti Pariksha*	-	75 min	-	105 min
11.	Duration (start from *MruduPaka*) for disappearance of sound upon burning of wick	-	75 min	-	105 min
12.	Total duration of heating (Heating)	-	13 h	-	13 h 30 min
13.	Total Time for the Process (Heating-Cooling)	-	52 h	-	54 h
14.	Total days required for process	-	3 Days	-	3 Days

Table 7: Results obtained during preparation of KPT

Parameters	*KPT M*	*KPT K*
Quantity taken of *Tila Taila* (g)	1000	1000
Quantity obtained of *Katupila Taila* (g)	939±2.6457	845.33±22.50
Total loss (g)	65.16±7.02	142.50±24.22
Loss in (%)	6.51±0.70	14.25±2.42
Color of *Kalka*	Brown	Dark Brown
Form of *Kalka*	Paste	Charred powder
Color	Dark Yellow	Dark Brown
Smell	Characteristic	Burnt
Touch	Oily	Oily

KPT: *Katupila Taila*

Table 8: Physicochemical characteristics of formulations

Parameters	*Tila Taila*	*KPTM*	*KPTK*
Specific gravity	0.9103	0.9179±0.0050	0.9294±0.0067
Refractive index	1.386	1.449±0.009	1.474±0.015
Acid value	3.263	2.908±0.0538	1.809±0.0472
Iodine value	109.860	108.075±2.9793	72.875±3.2548
Saponification value	161.32	169.66±8.9132	226.64±6.1567
BTTT value	21	19.6±0.3	28.5±0.5

of unsaturation which suggests quantity of double or triple bond between C compounds (with single bonds in FFA).[11] However, a complex series of reactions such as hydrolysis, oxidation, polymerization, isomerization, and cyclization takes place during the deep-fat frying. Under the influence of temperature, fat and oils are susceptible to oxidation primarily leading to the formation of hydroperoxides. Due to their high reactivity, these hydroperoxides especially at high temperatures rapidly react with secondary oxidative products, for example, aldehydes, ketones, peroxides, and hydrocarbons as well as cyclic compounds.[14] The effects increase in iodine value, acid value, and other parameters like peroxide value are temperature dependent (intensity and duration of heating). These reactions result in the formation of volatile and non-volatile compounds affecting the sensory, functional, and nutritional qualities of the frying oil. Thus, results of parameters of fats and oils for medicated *Sneha kalpana* are particular for specified formulation prepared with mentioned protocol (including type of *Snehapaka*). In present study, also similar results as mentioned above are derived. Iodine value and acid value in KPTK (*Katupila taila kharapaka*) are comparatively less as like in deep frying against KPTM where these parameters are expected to increase in view of heating. In present study of preparation of *Madhyam Paka* and *Kharapaka Katupila taila*, as there was neither significant change in range of highest temperature given for *Snehapaka*, nor significant change in duration of *Snehapaka*, denying their major role in exhibition of observed difference in results of these tests for oil (Acid value and iodine value) but supporting significance of ayurvedic principle of stages of *Shnehapaka,* for example, *Madhyama Paka, Khara Paka* in the present case and the difference may be just attributed to further additional duration (30 min) and intensity of heating (5°C more than highest temperature given for *Madhyama Paka*) of prepared formulation *Katupila taila Madhaym Paka* (KPTM) leading to its *Kahara Paka,* that is, KPTK. Thus, very little additional heating (not only the intensity of temperature but duration of heating) after *Madhyam Paka* can change the nature of prepared *Snehapaka;* hence, classical texts has mentioned different types of *Snehapaka* (e.g., *Ama* (*Chikkana*), *Mrudu, Madhyam, Khara, Dagdha* (*Vishoshi*) *Paka,* etc.) along with their *Siddhi Lakshana*[15,16] (parameters to identify optimum end points for state of *Snehapaka*, chief desirable characteristics for process completion) for different types of *Snehapaka* individually.

Conclusion

Adopted method of *Snehapaka* for preparation of *Katupila Taila* can be considered as standard. An average 939 g ± 2.6457 (93.90% yield) of KPTM and 845.33 g ± 22.50 (84.53% yield) of KPTK was obtained from batch size of, 1 L of TT with 212 g of *Kalka Dravya* and 4 L of *kwatha* of *Katupila* stem prepared over 3 days with total duration of actual heating of 13 h with maximum temperature of 92°C for *Madhyama Paka* and 13 h and 30 min with the highest temperature of 97°C for *Khara Paka*. Both *paka* have different route of administration/application. Hence, as per the indication of *sneha*, *Madhyama paka* and *khara paka* can be obtained deliberately by means of temperature and duration of heating.

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