Research Article

www.ijrap.net

(ISSN Online:2229–3566, ISSN Print:2277–4343)

PHARMACEUTICAL AND ANALYTICAL STUDY OF KAJJALI WITH SPECIAL REFERENCE TO DURATION OF TRITURATION

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Received on: 13/6/25 Accepted on: 28/7/25

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DOI: 10.7897/2277-4343.164129

ABSTRACT

Kajjali is Sagandha, Niragni murchana of Parad. In the middle of all Khalviya Rasayana, Kajjali is enclosing chief importance as it forms the base to several Parad Yoga. Apart from this there is no clear explanation of duration of trituration for Kajjali preparation. In the present work, Samgungandhak Kajjali was prepared, and samples of Kajjali were taken at different intervals of time for analyzing on classical as well as modern parameters viz. XRD and SEM. Kajjali was triturated for 120hrs but the luster of Parad still present in it. With XRD analysis free mercury got reduced to greater extent and also the size of particles gets reduced with hours of trituration and found in nano-particle range by SEM analysis.

Keywords: Kajjali, Parad, XRD, SEM.

INTRODUCTION

Parad (Mercury) is one of the chief constituents of Rasaushadhis[1-4]. The elemental form of Parad could not be used for therapeutic purposes. In the classical text, various procedures are described to convert it into compound form known as Rasa Bandha[5]. Among these, one is Kajjali Bandha.[6] Kajjali is a fine black powder obtained from pounding of Sudha Parad, Sudha Gandhaka, Dhatu (metals), other substances, without addition of any liquid substance is known as Kajjali.[7,8] Kajjali prepared with Sudha Parad and Sudha Gandhaka is used both, individually or in compound form in various diseases.[9,10] Beside this, it also used for marana of Rajata, Tamra and Lauha.[11,12,13] It is main ingredient of various Rasa Yogas.[14] It cures all types of diseases with the specificity of sahapana and anupana.[10] Kajjali can be prepared as dwiguna, triguna, chaturguna, shadguna, ashtaguna, shodashguna (i.e. the ratio of Parad and Gandhaka can be 2, 3, 4, 6, 8, 16 parts).[15]

Acharyas specified that Kajjali becomes soft, smooth and lusterless (nischandra)[8] in final phase of preparation but there is no explanation that reveals the time limit for trituration of Kajjali. Besides this, on studying the earlier research work concerning Kajjali, there is difference in duration of trituration.[16,17]

In the present study, samples of Kajjali were taken at different intervals of time and examined for classical tests and also for modern analytical tests viz. XRD and SEM. So, by this study an attempt has been made to set a time limit for the trituration of Kajjali.

MATERIALS AND METHODS

Pharmaceutical Study

Procurement of Raw Material: Asudha Parad and Asudha Gandhaka were purchased from Hans Ayurved Bhawan Pharmacy Haridwar. All the drugs and godugdha (cow's milk) were purchased from local market of Haridwar.

Steps Involved in Preparation of Kajjali
Samgungandhaka Kajjali was prepared as per reference of Rasa Tarangini.[15]
Kaanji preparation- Kaanji was prepared as per reference of Rasa Tantra Saar and Siddha Prayoga Samgrah.[18] (Table 1)
Shodhana of Parad- Rasendra Saar Samgrah[19]
Shodhana of Gandhaka- Rasa Tarangini[20]

Shodhana of Parad: Asudha Parad (450g) was taken and triturate with Nagavalli svarasa (60ml of juice of betel leaf), Lahsuna svarasa (60ml of juice) and Triphala kwatha (60ml) in a clean Khalva yantra (mortar and pestle) for eight hours daily for three days. The Parad was washed with lukewarm water several times until clean and clear Parad is obtained. After that Parad was washed with Kanji till the color of Kanji remains unchanged and allowed to dry. Filter Parad with four folded Markin cloth. Shudha Parad collected after the shodhana process was 448gm (98%). (Table 2)

Shodhana of Gandhaka: Gandhaka (450gm) was powdered (sieve no. 40) and pour into a cauldron containing Katu taila (mustard oil) (112ml) and melted over mridu agni (Max 114^0 C). Boiled Godugdha (2 litres) was placed in another stainless-steel vessel. A piece of Markin cloth was tied over the mouth of steel vessel containing Godugdha. When Gandhaka completely melted, it was poured into Godugdha through the cloth. After

cooling Gandhaka was taken out in tray and washed out by hot water and allowed to dry. The same procedure was repeated two more times and at the end of the procedure Gandhaka was washed carefully with hot water and allowed to dry in open air at room temperature. (Table 3)

Kajjali Preparation: 300g of Sudha Parad and 300g of Sudha Gandhaka were taken in a Khalva yantra and triturated for a specific time period till a fine black lusterless powder was obtained. As time proceeds, mixture changes to blackish yellow color in which particles of Parad were visible. After some time, the whole mixture gets converted into black compound. As trituration process continues, mixture converted into fine, black, smooth, Kajjal sadrusha (Collyrium) powder. During the process, Kajjali was tested for Rekhapurnatva (filled between finger lines), Varitaratva (floats on the surface of water) and Nischandra (lustureless) tests. The trituration continued up to 120 hours and examined for nischandra test but the luster of Parad did not disappear completely. Samples of Kajjali were taken at 4, 22, 94, 100, 110 and 120 hours of trituration. Net quantity of Kajjali obtained was 500gm (83.3%).

Analytical Study
Samples of Kajjali were taken at 4, 22, 94, 100, 110 and 120 hours of trituration and analyzed for physical changes. Also, the XRD analysis to find the composition with structure of Samgungandhaka Kajjali and SEM (Scanning electron microscopy) for particle size of the samples (at 22, 94, 120 hours) have been performed.

Physical Parameters: It includes appearance, color, touch, smell and luster.

XRD Analysis
Powder X-Ray diffraction (XRD) study was carried out in Institute of Instrumentation Centre, IIT, Roorkee, India.

X-Ray diffraction (XRD) is a versatile, non-destructive technique that reveals detailed information about the chemical composition and crystallographic structure of natural and manufactured materials. The basic principle of the phase analysis using powder XRD technique lies in the presence of diffraction peaks corresponding to various interplanar (dhkl) spacings which are the characteristics of a given material.[21]

The samples were exposed to X-ray beam of intensity 45-55KV and 40-50mA. Pattern was recorded for the angle (2θ) ranging from 5^0- $119^{0.}$

Scanning Electron Microscopy[22,23]
Scanning Electron Microscopy study was carried out in Institute of Instrumentation Centre, IIT, Roorkee, India. SEM analysis was done only of samples taken at 22, 94 and 120 hours of trituration.

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The instrument used for the study have model name Carl Zeiss AG Supra 40 WDS Manufacture by Zeiss Gemini, carl Zeiss SMT, Oberkochen (Germany). The operating parameters were Energy resolution – 15 KV, working distance- 10mm, Chamber pressure set to- $< 2.2\times10^{-4}$torr, Tilt angle- 0^0c and accumulation times- 50 sec. The software used during study was Noran System Sin.

RESULTS AND DISCUSSION

The comparative analysis between properties of asudha and sudha Parad and Gandhaka were depicted in Table 4 and 5. Alteration in the parameters of Kajjali with trituration were found to be significant and illustrated in Table 6.Table 7 pertaining the data of XRD analysis of different samples of Kajjali. SEM analysis of Kajjali samples at 22, 94 and 120 hours of trituration were exemplified in Table 8.

Various procedures of Parad and Gandhaka shodhana have been described in our classical texts.[20,24-32] In the present work, shodhana was done by triturating Parad with Naagvalli swarasa, Lahsuna swarasa and Triphala kwatha because of good availability of drugs for shodhana and also the loss during the process was very minimum (percentage yield 98%). Gandhaka shodhana was done in katu taila. As Katu taila have laghu guna, katu rasa and vipaka with agni dipana karma[33,34] by which it may possibly help in pachana of Aamdosa, that may be the reason behind the use of katu taila shodhita Gandhaka in Amadosha vyadhi.

During Kajjali preparation, after 72 hours of trituration, the mixture completely turned into Kajjali and became Varitara and Rekhapurna but luster of Parad was still observed. After 94 hours entire powder became fine, smooth, black and luster of Parad decreased but did not disappear completely. So, the trituration continued to get nischandra Kajjali as the property of Kajjali only mentioned in Rasamritum. Although, the luster of Parad get reduced than before but has not disappeared completely till 120 hours.

The XRD pattern of all six Kajjali samples has been shown in Graph 1 to 6. The X-ray diffraction results of the Kajjali indicated that three numbers of sharp and intense X-ray diffraction peaks are present in diffraction images of all samples that showed the face centered cubic crystalline nature of β-HgS (Cinnabar) in Kajjali samples. XRD pattern shows β-HgS along with free Sulphur are the crystalline phase also present in all Kajjali samples. XRD of Sample1 represents the mixed phase crystalline structure of β-HgS with Hg and S impurities. In all Kajalli samples any broadness in the diffraction peaks is not seen that indicates good crystalline properties in material. The X-ray diffractogram of all the samples of Kajjali, the most prominent three peaks of X-ray diffraction were obtained having the crystal lattice plane of (111), (220) and (311) respectively that shown in Table 7.

With the analysis of XRD data of all Kajjali samples, it was found that free mercury is present in all the samples of Kajjali, but it gets reduced with trituration and found in comparatively minute quantity in sample 6 of 120 hours trituration. So, if the trituration is done for some more time, it may possibly result in complete diminution of free mercury.

SEM of all the Kajjali samples shows heterogeneous structure i.e. all the particles were not of equal size. Data concerning SEM analysis at 50KX magnification in Table 8 of Kajjali samples at 22, 94 h and 120 hours of trituration shows particle size between 185.1nm- 661nm, 158.3nm-600.7nm and 100.4 nm to 171.1nm respectively. The values show that the particle size going to decrease along with diminution in variation between the maximum and minimum size of particles with trituration.

Table 1: Kaanji Preparation

Weight of rice	2500 gm
Weight of water	35 kg
Weight of Manda	19 L
Total time taken in Manda preparation	90 min
Weight of kulatha	2500gm
Weight of water	35 kg
Weight of kulatha kwatha	9 L
Total time taken in kwatha preparation	5hrs
Weight of saindhava lavana	29gm
Weight of Raajika	29gm
Yield of Kaanji	28L
pH	2.91
Total time taken in Kaanji preparation	15days

Table 2: Parad Shodhana

Ingredients	Quantity
Ashudha Parad	450gm
Total quantity of Nagvalli swarasa	60ml
Total quantity of Lahsuna swarasa	60ml
Total quantity of Triphala kwatha	60ml
Total yield (%)	98%

Table 3: Gandhaka Shodhana

Temperature	Quantity of milk (ml)	Quantity of oil (ml)	Weight before shodhana (gm)	Weight after shodhana (gm)	Loss in weight (gm)	Loss %
114^0C	2000	125	500	438	62	12.4
115^0C	2000	109.5	438	389	49	11
115^0C	2000	97.25	389	359	30	7.7

Table 4: Properties of Ashudha and Shudha Parad

Parameter	Ashudha Parad	Shudha Parad
Appearance	Liquid	Liquid
Color	Grey	Bright grey
Touch	Smooth	Smooth

Table 5: Physical Properties of Asudha and Sudha Gandhaka

Parameter	Asudha Gandhaka	Sudha Gandhaka
Appearance	Crystal	Granular
Color	Yellow	Bright yellow
Touch	Hard	Smooth
Smell	Sulphurious	Typical smell of sulphur
Taste		

Table 6: Physical properties of Kajjali in different hrs of Trituration

Parameter	4 hours	22 hours	94 hours	100 hours	110 hours	120 hours
Appearance	Powder	Powder	Powder	Powder	Powder	Powder
Color	Grey	Grayish Black	Black	Black	Black	Black
Touch	Rough	Smooth	Very Smooth	Very Smooth	Very Smooth	Very Smooth
Smell	Sulphuroius	Sulphuroius	Sulphuroius	Sulphuroius	Sulphuroius	Sulphuroius
Luster	Easily appear mercury particle	Comparatively less	Appears only in sunlight	Comparatively less and appears only in sunlight	Appears only in sunlight	Appears only in sunlight

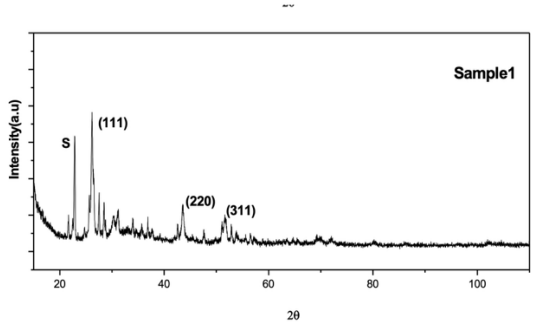
Table 7: XRD Analysis

Sample	Degree	d-values	Intensity (Relative)	Miller Plane (hkl)	Phase assignment
Sample 1	26.15	3.34334	100	(111)	Cub and Hex
	43.62	2.08352	28.1	(220)	Cub and Hex
	51.70	1.81430	19	(311)	Cub and Hex
Sample 2	26.38	3.39171	1oo	(111)	Cubic
	43.65	2.07723	29.4	(220)	Cubic
	51.68	1.74430	22	(311)	Cubic
Sample 3	26.35	3.37920	100	(111)	Cubic
	43.73	2.06835	29	(220)	Cubic
	51.77	1.76432	21.8	(311)	Cubic
Sample 4	26.39	3.41275	100	(111)	Cubic
	43.48	2.07930	31.1	(220)	Cubic

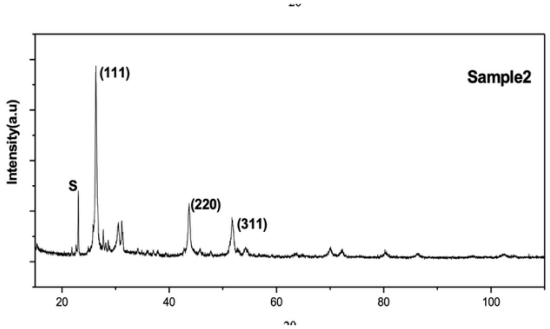
	51.54	1.77168	23.2	(311)	Cubic
Sample 5	26.33	3.38151	100	(111)	Cubic
	43.70	2.06954	29.7	(220)	Cubic
	51.76	1.76478	22.6	(311)	Cubic
Sample 6	26.20	3.39835	100	(111)	Cubic
	43.58	2.07510	29.7	(220)	Cubic
	51.64	1.76840	22.3	(311)	Cubic

Table 8: Particle size analysis of Kajjali Samples by Scanning Electron Microscopy

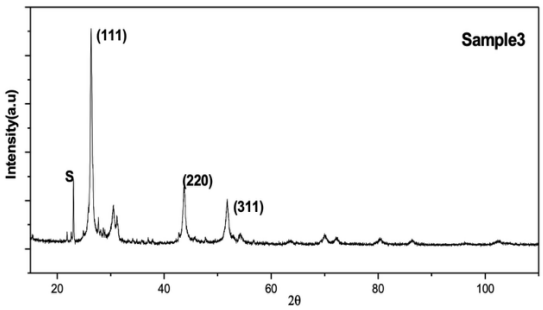
Sample	Particle size Range
Kajjali (22 hours)	185.1nm- 661nm
Kajjali (94 hours)	158.3nm-600.7nm
Kajjali (120 hours)	100.4 nm to 171.1nm



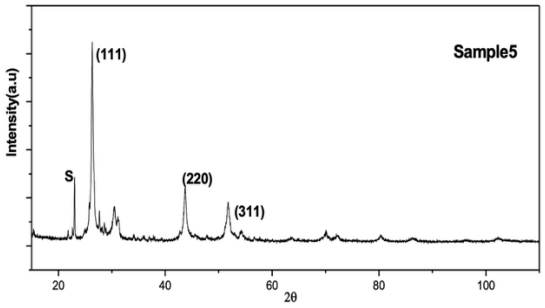
Graph 1: Analysis of Kajjali Sample1 (4 hours) by X-Ray Diffraction



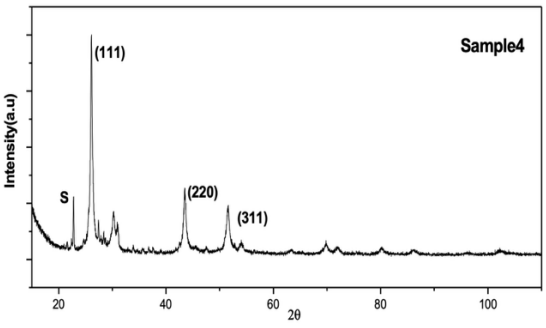
Graph 2: Analysis of Kajjali Sample 2 (22 hours) by X-Ray Diffraction



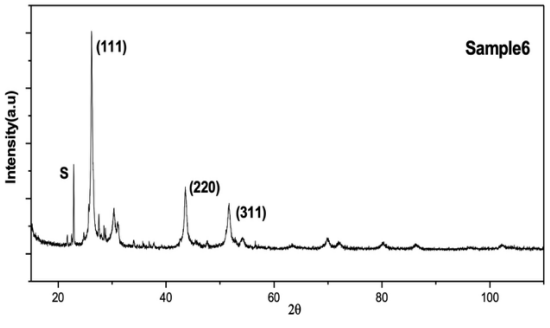
Graph 3: Analysis of Kajjali Sample 3 (94 hours) by X-Ray Diffraction



Graph 4: Analysis of Kajjali Sample 5 (100 hours) by X-Ray Diffraction



Graph 5: Analysis of Kajjali Sample 4 (110 hours) by X-Ray Diffraction



Graph 6: Analysis of Kajjali Sample 6 (120 hours) by X-Ray Diffraction

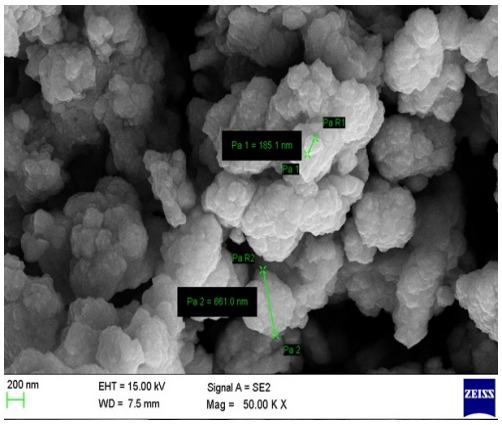


Figure 1: SEM Image of Kajjali (at 22 hours trituration)

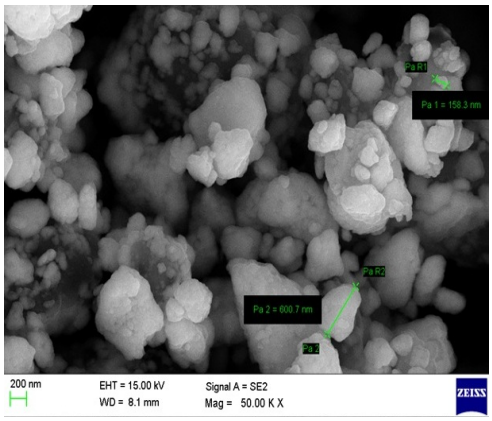


Figure 2: SEM Image of Kajjali (at 94 hours trituration)

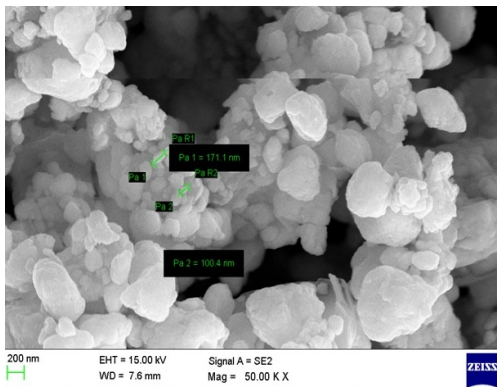


Figure 3: SEM Image of Kajjali (at 120 hours trituration)

CONCLUSION

From the present study, it can be concluded that Parad shodhana done as per reference of Rasendra Saar Samgrah is better in terms of yield. The luster of Kajjali got reduced to greater extent but did not disappear completely even after 120 hours trituration. But with trituration of Kajjali, quantity of free mercury got reduced and also the particle size reduced to nano- particle range. As Kajjali is ingredient in majority of Rasaushadhies, a standard operating procedure for Kajjali must be developed.

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Cite this article as:

Kestwal Kamini and Mitra Shuchi. Pharmaceutical and analytical study of Kajjali with special reference to duration of trituration. Int. J. Res. Ayurveda Pharm. 2025;16(4):60-65
DOI: http://dx.doi.org/10.7897/2277-4343.164129

Source of support: Nil, Conflict of interest: None Declared