

Fourier Transforms Infrared Spectroscopy Study on *Shadguna Makaradhwaja* - An Ayurvedic Mercurial Drug Compound

Vaibhav Charde*, Kishor Patel¹, Rohit Gokarn², Dhirajsingh Rajput³,
Biswajyoti Patgiri⁴, Pradeep Kumar Prajapati⁵

*¹Ayurveda Practitioner, Ashwinau Ayurveda & Panchakarma hospital, Ahmedabad, Gujarat, India,

²Assistant Professor, Division of Ayurveda, CIMR, Manipal University, Manipal, Karnataka, India, ³Assistant Professor, Department of Rasashastra and Bhaishajya Kalpana, Mahatma Gandhi Ayurved College Hospital & Research Centre, Salod (H), Wardha, Maharashtra, India, ⁴Professor, Department of Rasashastra and Bhaishajya Kalpana, ⁵Professor, Dept. of Rasashastra and Bhaishajya Kalpana, Institute for Post Graduate Teaching & Research in Ayurveda, Gujarat Ayurveda University Jamnagar, Gujarat (India), ⁵Professor, Dept. of Rasashastra and Bhaishajya Kalpana, All India Institute of Ayurveda, New Delhi, India

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Abstract

Background: *Shadguna Makaradhwaja* (SM) (a sublimated mercurial preparation with gold, sulphur and mercury in the ratio 1:8:48) is a well known and frequently used formulation especially in the treatment of diabetes mellitus and impotency. Ayurvedic classical texts advocates preparing of SM from thin foils of gold. However there is a significant quantity of gold which remains at the bottom of glass bottle as residue which can be utilized again to prepare SM. **Aim:** To identify the functional group and organic ligands present in samples of SM prepared from gold foils and from residual gold by using Fourier transforms infrared spectroscopy (FTIR). **Material and methods:** SM prepared from *Swarna Varkha* (thin foils of gold) (SM V) was taken from previous research of Shraddha Dhundi et. al. and one sample of SM prepared from bottom residue (*Apakwa Talastha Swarna* powder) (SM R) were subjected to FTIR analysis. **Results:** The analysis of both samples showed large number of peaks showing presence more functional groups. Intensity of peaks in SM R was slightly higher than SM V which may be due to repeated heating as it is prepared from bottom residue. **Conclusion:** Both samples contain few functional groups with complicated structure. SM R showed more peaks in double bond region which indicated its higher stability compared to SM V. Presence of different functional groups indicates their difference in pharmacological action.

Keywords: Ayurveda, FTIR, Gold, Mercury, *Shadguna Makaradhwaja*, Sulphur

Introduction

Shadguna Makaradhwaja (SM) is a *Kupipakwa Kalpa* (formulation prepared in glass bottle by following specific heating pattern). It is a metallic & mineral sublimated compound of *Swarna* (processed gold), *Parada* (mercury) and *Gandhaka* (sulphur) in 1:8:48 proportion by using herbal levigating media i.e. *Karpasa Pushpa Swarasa* (flower of *Gossypium arboreum* Linn) and *Ghritha Kumari Swarasa* (*Aloe vera* juice) ^[1]. According to Ayurvedic point of view, the materials utilized for levigation impregnate their properties on the material to which levigation was done. In modern view it can be correlated with chemical reaction between material and chemical constitute present in levigation media. The process of levigation may leads to incorporation of organic molecules and FTIR is useful in determining organic ligands and functional groups ^[2]. In other word FTIR can be used to assess the effect of levigation procedure which is widely utilized in Ayurvedic pharmaceuticals.

During the preparation of SM, significant quantity of gold remains at the bottom of glass bottle in the form of residue and reutilization of such residue can reduce the expense as gold is a costly metal. Although the difference between SM prepared from purified gold and gold residue is not known yet. The present study was planned to know the difference in terms of presence of functional groups in SM V (*Shadguna Makaradhwaja* prepared from *Swarna Varkha*) and SM R (*Shadguna Makaradhwaja* prepared from gold residue) by using FTIR.

Corresponding address:

Dr. Vaibhav Charde
Ayurveda Practitioner,
Ashwinau Ayurveda & Panchakarma Hospital,
Ahmedabad, Gujarat, India,
[Email: vaibhavayudoc@gmail.com](mailto:vaibhavayudoc@gmail.com)

Material and Methods

SM was prepared in the department of *Rasa Shastra* and *Bhaishajya Kalpana*, IPGT & RA, Gujarat Ayurveda University, Jamnagar (India). SM prepared from *Swarna Varkha* (thin foils of gold) (SM V) was taken from previous research of Shraddha Dhundi et. al.^[3] and one sample of SM was prepared from bottom residue (*Apakwa Talastha Swarna* powder) (SM R) in the ratio 1:8:48 [*Swarna* (Au): *Parada* (Hg): *Gandhaka* (S)].^[4] Both samples were levigated with same herbal liquid media viz. *Karpasa Pushpa Swarasa* and *Kumari Swarasa* and subjected to *Kupipakwa* method through vertical electrical muffle furnace for 36 hours.^[1] Both samples were analyzed for their functional groups by using FTIR at Sophisticated Analytical Instrument Facility (SAIF), Mumbai, India.

FTIR imaging is a complimentary Imaging tool and is a very versatile analytical technique for spectrochemical imaging. The advantage of chemical imaging compared to other sensor technologies is the ability to analyze the spatial distribution of the component materials in blends, granules or finished dosage forms. Many analysts need to obtain the molecular information from an area of a sample to see a picture of the distribution of molecules or functional groups. FTIR spectroscopy is well proven as a sensitive, rapid technique for material characterization of various samples for molecular species in a broad range of materials. By coupling the FTIR to a microscope accessory, measurements on small sample can be routinely carried out, down to few micron areas. In the present study FTIR spectrometer Perkin Elmer Spectrum GX Range of range 30-15600 cm^{-1} was used with specifications such as Focal plane array: 128 x 128, range: 4000-900 cm^{-1} , single point detector range 7500-450 cm^{-1} , analysis area 128x128 in 2D format on the sample plane 300 x 300 m, Grazing angle and spatial resolution with 15x.

Observations and Results:

FTIR analysis of *Makaradhwaja* samples was performed to detect the presence of functional groups or organic ligands.

FTIR spectra of SM V in the work of Shraddha Dhundi et. al. were observed in the region of 3566.15 to 580.3 cm^{-1} . General overview of SM V indicates presence of large number of functional groups. Total of 19 peaks were obtained which includes 6 peaks in Hydrogen stretching region (4000 to 2700 cm^{-1}), 2 peaks in Triple bond region (2700 to 1950 cm^{-1}), 2 peaks in Double bond region (1950 to 1550 cm^{-1}) and 4 peaks in Fingerprint region (1500 to 700 cm^{-1}). 5 peaks are obtained below known region of frequencies. Three medium peaks obtained above 3000 cm^{-1} are due to stretching vibrations between N-H bonds of Secondary amines. Strong peak at 3421.35 and 1630.77 cm^{-1} wavelength is also due to N-H stretching vibrations but it indicates presence of primary amines. Medium to strong C-H stretching vibrations obtained at 2930.23 and 2854.33 cm^{-1} are representative of Alkyl functional groups specifically Methylene bond. Strong peak obtained at 1630.77 cm^{-1} can be assigned to either benzene ring (Dienes) or to primary amines as the nature and intensity of this peak fall intermediately in the range of benzene and primary amines. Two peaks in the range of 1100-1300 cm^{-1} are raised due to C-O stretching vibrations and are assigned to two bands of Esters which are distinct from ketones, which do not possess a C-O bond. Two overlapped peaks obtained arised due to C-N stretching vibrations at 1053.33 and 1199.50 cm^{-1} are assigned to aliphatic amines. Strong peak at 670.46 cm^{-1} is indicates stretching vibrations between C-H bond and represent Vinyl functional group with specific bond of Cis-disubstituted alkenes as shown in table 3.

FTIR analysis of *Makaradhwaja* prepared from residue (SM R) also showed presence of large number of functional groups. FTIR spectra of SM R were observed in the region of 3877.66 to 461.85 cm^{-1} (fig.2). General overview of SM R indicates presence of many similar functional groups which were detected in SM V. Total 19 peaks were obtained which includes 5 peaks in Hydrogen stretching region (4000 to 2700 cm^{-1}), 2 peaks in Triple bond region (2700 to 1950 cm^{-1}), 5 peaks in Double bond region (1950 to 1550 cm^{-1}) and 4 peaks in Fingerprint region (1500 to 700 cm^{-1}).

Table 1: FTIR Peaks obtained in SM V and SM R

Sr.n o.	Sample	No. of Peaks	Obtained Peaks
1	SM V	19	409.56, 440.93, 475.49, 510.44, 593.62, 746.79, 1051.23, 1199.50, 1383.27, 1630.77, 1805.58, 2063.03, 2343.67, 2854.34, 2930.23, 3260.51, 3421.35, 3566.15, 3754.96
2	SM R	19	461.85, 558.61, 662.58, 734.19, 1024.95, 1471.41, 1522.91, 1562.90, 1637.97, 1710.26, 1765.79, 1859.81, 2282.81, 2456.46, 3462.56, 3638.02, 3760.61, 3815.87, 3877.63

Table 2: FTIR Peaks of sample SM V and SM R obtained in different regions

Sam ple	Hydrogen stretching region (4000 to 2700 cm^{-1})	Triple bond region (2700 to 1950 cm^{-1})	Double bond region (1950 to 1550 cm^{-1})	Fingerprint region (1500 to 700 cm^{-1})	Unknown Region
SM V	2854.34, 2930.23, 3260.51, 3421.35, 3566.15, 3754.96	2063.03, 2343.67	1630.77, 1805.58	746.79, 1051.23, 1199.50, 1383.27 734.19,	409.56, 440.93, 475.49, 510.44, 593.62
SM R	3462.56, 3638.02, 3760.61, 3815.87, 3877.63	2282.81, 2456.46	1562.90, 1637.97, 1710.26, 1765.79, 1859.81	1024.95, 1471.41, 1522.91	461.85, 558.61, 662.58

Fig 1: FTIR graph for SM V

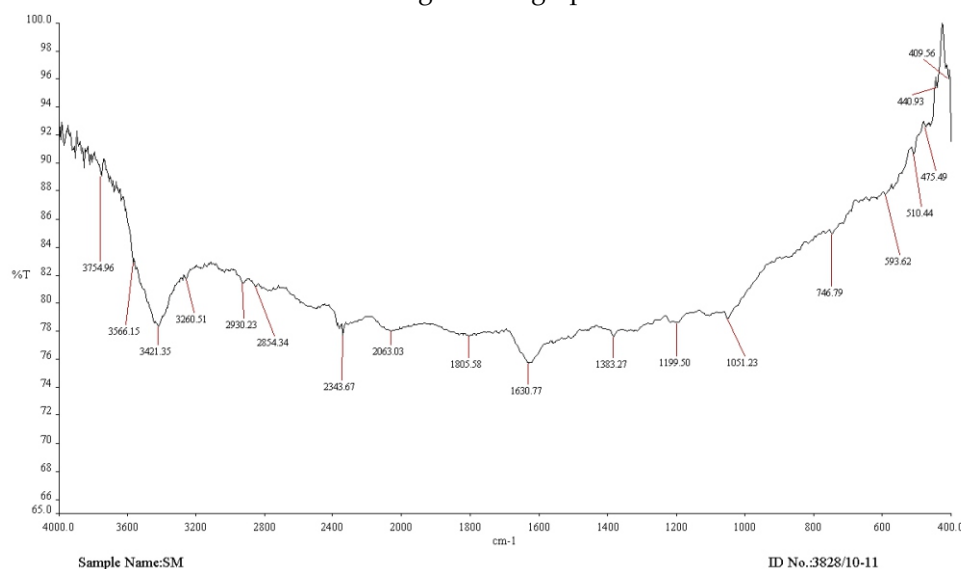
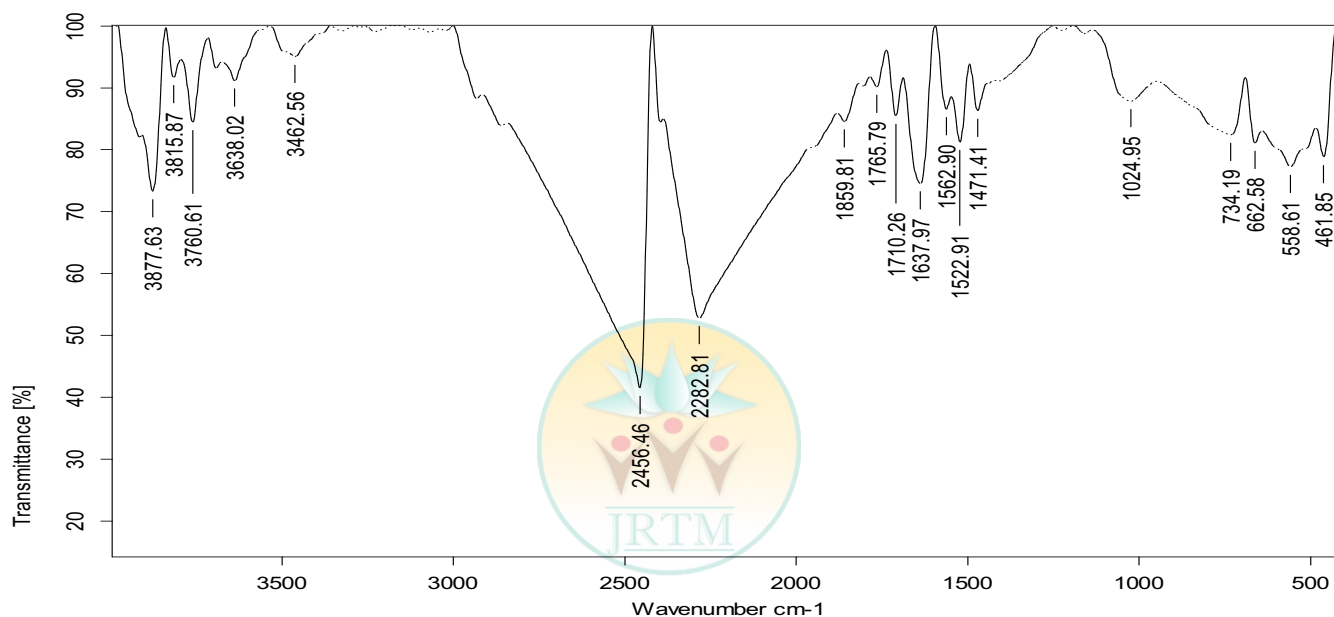


Fig 1: FTIR graph for SM R



3 peaks were obtained below known region of frequencies. Strong and sharp peaks obtained at 3462.56, 3638.02 and 3462.56 cm^{-1} indicate stretching vibrations between O-H bond and represent presence of alcohols or phenol functional group. Similar low intensity vibrations of O-H bond stretching were detected at 2456.46 cm^{-1} wavelength and assigned to carboxylic acids. Presence of aliphatic amine functional group was observed by a peak at 1024.95 cm^{-1} which is raised due to C-N stretching vibrations but the appearance of peak is not much clear and represent either primary or secondary amines.

Nitriles functional group was detected at 2281.81 cm^{-1} with a very sharp and broad peak of $\text{C}\equiv\text{N}$ stretching. $\text{C}=\text{O}$ stretching vibrations observed at 1710.26 and 1765.79 cm^{-1} are assigned to alpha-beta-unsaturated aldehydes. C-C bond stretching vibrations at 1637.97 cm^{-1} are indicative of functional group with aromatic ring. Sharp peaks obtained at 558.61, 662.58 and 734.19 cm^{-1} were raised due to C-X stretching vibrations and represent chloroalkenes and bromoalkenes respectively. Alkene functional group was also detected at 662.58, 734.19 cm^{-1} peak level which is due to $=\text{C}-\text{H}$ bends. Detail of assignment of peaks in various ranges of FTIR spectra of SM R is depicted in table 4.

Table 3: Various peaks obtained in FTIR analysis of SM V and their correlation

No.	Peak	Actual peak	Bond	Functional group	Appearance
1	>3000 cm^{-1}	3260.51, 3421.35, 3566.15, 3754.96	N- H	Secondary amines	Weak to medium
2	3400-3500 cm^{-1}	3421.35	N- H	Primary amines	Strong
3	2925 cm^{-1}	2930.23	C- H	Alkyl	Medium to strong
4	2850 cm^{-1}	2854.34	C- H	Alkyl	Medium to strong
5	1625 cm^{-1}	1630.77	C- C	Benzene ring	Strong
6	1560-1640 cm^{-1}	1630.77	N- H	Primary amines	Strong
7	1100-1300 cm^{-1}	1199.50	C- O	Esters	Two bands (distinct from ketones, which do not possess a C- O bond)
8	1020-1220 cm^{-1}	1051.23, 1199.50	C- N	Aliphatic amines	Often overlapped
9	1000-1100 cm^{-1}	1051.23	C- X	Fluoroalkanes	

Table 4: Peaks obtained in FTIR analysis of SM R and their correlation

No.	frequency, cm^{-1}	Actual peak	Bond	Functional group	Appearance
1	3640-3610	3462.56, 3638.02	O-H	Alcohols, phenols	Strong and sharp
2	3500-3200	3462.56	O-H	Alcohols, phenols	Short and broad
3	3300-2500	2456.46	O-H	Carboxylic acids	Medium
4	2260-2210	2282.81	$\text{C}=\text{N}$	Nitriles	Very sharp and broad
5	1710-1665	1710.26, 1765.79	$\text{C}=\text{O}$	Alpha, beta-unsaturated Aldehydes, ketones	Short
6	1600-1585	1637.97	C-C	Aromatics	Medium
7	1550-1475	1471.41, 1522.91	N-O	Nitro compounds	Short
8	1250-1020	1024.95	C-N	Aliphatic amines	Medium
9	1000-650	662.58, 734.19	$=\text{C}-\text{H}$	Alkenes	Short
10	910-665	662.58	N-H	Primary, secondary amines	Short and broad
11	850-550	558.61, 662.58, 734.19	C-Cl or C-Br	alkyl halides	Medium

Discussion

FTIR analysis of SM V and SM R showed large number of various peaks obtained in Hydrogen stretching region, Triple bond region, Double bond region and Fingerprint region as shown in table 1 and 2. Two peaks in both samples are detected in triple bond region which indicate presence of complicated structure as well as strong bonding between various functional groups. Some peaks which are observed below fingerprint region are kept in unknown region as the standard classification of FTIR peaks regions is limited up to 700 cm⁻¹.

Both samples showed 19 peaks however intensity of peaks in SM R is slightly higher than SM V. This may be due to repeated heating as SM R was prepared from residue. It can be assumed that repeated heating cause stability which may result in absorption of higher energy and thus emission of energy at higher excited state. The differences in the emitted energy at different levels result in obtaining peaks at those levels. It means SM R has more stable nature than SM V. Functional groups raised due to O-H stretching are detected in SM R. Chloro-alkene and bromo-alkene are observed in SM R while fluoro-alkene are observed in SM V analysis. These differences in functional groups and intensities of peaks confirm that both samples are different in their chemical properties. This is also suggestive of difference in pharmacological action of both drugs.

Conclusion

The two samples of *Shadguna Makradhwaja* contain few functional groups with complicated structure. SM R showed more peaks in double bond region which indicated its higher stability compared to SM V. Presence of different functional groups indicates their difference in pharmacological action and hence the therapeutic usage of both of them should be used with caution.

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