IJPSR (2018), Volume 9, Issue 6 (Research Article)

INTERNATIONAL JOURNAL OF PHARMACEUTICAL SCIENCES AND RESEARCH

Received on 27 September, 2017; received in revised form, 30 November, 2017; accepted, 06 March, 2018; published 01 June, 2018

METALLURGICAL AND SOLUBILITY STUDY OF ASHODHIT AND SHODHIT SROTONJANA

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Keywords:

Anjana, Ashodhit, Bhavana, Shodhana, Srotonjana

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ABSTRACT: The mineral Srotonjana (antimony sulfide) is being used by Indians since ancient times. In Ayurvedic texts, it has been directed that Srotonjana should be used only after shodhana. Shodhana is a process to remove the unwanted physical, chemical impurities and toxic material and makes it suitable for medicinal use. Metallography is an advanced scientific study which is used in the study of the microstructure and identification of the parent metal particles along with the nature of compound formed during the shodhana. The difficulty of low solubility and less dissolution rate of metal and minerals has received broad academic and industrial awareness. So the aim of present study was to generate metallographic and solubility in the different solvent study as a tool (fingerprint) for ashodhita and shodhita Srotonjana for quality assessment and standardization. In the present study, Srotonjana was shodhit by seven bhavana (levigation) with bhringraj swaras and with triphala kwath. The finding results revealed that after shodhana Srotonjana broke into smaller particles and became homogeneous in both shodhit the samples but no change in their chemical composition. The shodhit samples of Srotonjana are soluble in the organic solvent which indicates the presence of organic material in Srotonjana.

INTRODUCTION: The eye has been privileged as the most important sense organ because its loss of function leads to the severe disability of man by keeping him in the darkness [1]. Anjana (collyrium) is a medicine of traditional technique which protects the eye form various diseases. The collyrium is mentioned as a daily routine to the eyes [2, 3]. According to most of the Rasa Shastra literature, five types of collyrium (Anjana) has been described like sauviranjana, rasanjana, srotanjana, pushapanjana, and nilanjana [4].

QUICK RESPONSE CODE

DOI: 10.13040/IJPSR.0975-8232.9(6).2579-84

Article can be accessed online on: www.ijpsr.com

DOI link: http://dx.doi.org/10.13040/IJPSR.0975-8232.9(6).2579-84

Stibnite or antimony sulfide (Sb_2S_3, Srotonjana) is the most ancient variety of collyrium (Anjana) recommended as preventing the deterioration of eyes as well as for its maintenance and beauty [5]. It is commonly found in America, Britain, China, and Japan.

In India, it is found in Bihar, Karnataka, Andhra Pradesh, Punjab, and Jammu. Srotonjana appears like black, as freshly prepared termite house and on breaking it gives lead gray with a blue tinge [6]. Most of the rasa drugs (herbo-mineral) obtain from mines, and they cannot be used as they are found. Srotanjana is found at a low temperature of hydrothermal veins, associated with realgar, orpiment, galena, pyrite, cinnabar, and quartz. Realgar, orpiment, galena these such types of minerals cause toxic in the human body [7]. All types of collyrium should be used only after shodhana [8].

To make them fit for therapeutic administration requires a certain process like shodhana and marana *etc*. Shodhana is a purification of the material by subjecting to the required process like swedana (boiling), mardana (trituration), prakshalana (washing), nirvapa (heating and dipping), bhavana (levigation), and bharjana (roasting), *etc*. [9] The concept of shodhana in rasa shastra not only covers the process of decontamination/detoxification of physical and chemical impurities although it also covers the reduction of side effects and enhances potency/ therapeutic efficacy of the shodhita drugs [10]. Although several studies have been carried out on ayurvedic anjana but less studies on shodhana of Srotonjana.

In the present research work, Srotonjana has been analyzed with a focus on preparing standard operating procedure. Metallography is an advanced scientific study which is used in the field of ayurveda to confirm the tests in a scientific manner. The metallographic study reveals the structural characterizations of metal and mineral. These studies also provide knowledge about phase orientations of an alloy and its physicochemical properties.

In this study, I have characterized the microstructure of the preparation, identification of the parent metal particles, and nature of compound (during purification process) by the metallographic study [11, 12]. Metal and minerals are not soluble in gastrointestinal tract due to low dissolution rate, limited oral bioavailability. The difficulty of low solubility and less dissolution rate of metal and minerals has received broad academic and industrial awareness. There are several methods (nanoparticles) undertaken to improve their dissolution and drug bioavailability [13, 14]. The present research work was concluded to generate metallographic and solubility study as a tool (fingerprint) to distinguish between ashodhita and shodhita Srotonjana for their quality. This can also be used to generate standardization parameters of the finished material.

MATERIAL AND METHODS:

Authentication of Raw Material: The raw material was purchased (300 gm.) from the local market and identified by the subject expert.

Chemical and Reagent: Geosyn Cold Mounting (GCM) compound and liquid, Grease, aluminium powder, nitric acid, acetic acid, ferric chloride.

Apparatus Requirement: Mounting socket, plane glass plate, glass rod, spatula, petri dish, cotton cloth, applicator stick, grinding machine with emery belt, polishing machine and metallurgical electronic microscope with the attached computer.

The following Samples were used for the Metallographic Study: Ashodhita Srotonjana (AS), Shodhit Srotonjana with bhringraja swarasa (SSBS), Shodhit Srotonjana with triphala kwath (SSTK).

Shodhana of Srotonjana: In the present study, the shodhana process of Srotonjana was done by two methods. In the first method, seven bhavana was given in the juice of *Eclipta alba* (Bhringraja Swarasa) and seven bhavana in triphala kwath. The processing of the shodhana of Srotonjana was done according to rasa texts [15, 16]. The first sample (150 gm.) of Srotonjana was subjected to shodhan (bhavana process) with bhringraja swarasa and triphala kwath separately. The trituration was done to till the Srotonjana was dried completely. The same process was repeated for another six times. The shodhit drug material of both samples was powdered and kept in airtight containers separately for analytical process.

Preparation of Metallographic Specimen:

Mounting: 500 mg of all three samples (AS, SSBS, SSTK) of srotanjana separately were taken in a petri dish and mixed separately with 1 gm of GCM. All the mixture of samples was poured into the copper mounting socket having a greased surface. After that adequate amount of GCM liquid was poured over the mixture and left for 5 minutes. After 5 min all the material became solid and removed it from the socket. The solid materials were cleaned with soap and water and then subjected for pulverizing.

Pulverizing: Pulverizing of the sample is done for metallographic examination. During pulverization, samples were cut into small pieces, and they became scratch free. The rough surface of the sample was made plain and smooth by designed motor driven emery belt. Pulverizing was done till the flat appearance of the sample was found.

The sample was washed thoroughly with soap and water. After that intermediate and fine pulverization process was used. In both processes, high qualities graded emery papers with respect to size and uniformity of the emery particles were used. This process was completed when the finer scratches replace the coarse scratches. After that it was subjected to polishing.

Polishing: During the pulverization, some fine scratches remain on the surface of the sample. The samples became free from fine scratches with the help of polishing. In the polishing process, two steps were used in the first step samples got preliminary polishing and second step final polishing. In the first step, polishing of samples was execute on polishing laps which were made-up of bronze discs covered with cotton cloth. In the second step, sample was seized with modest pressure against the moving lap. After final step of polishing all the samples were washed with water. For removing of polishing materials, samples were cleaned with a wet cloth and rinsed gently by methanol and dried in hot air.

Etching: In this process, a special type of chemical reagent (etchant) was used to the polished metallographic surface to know about the metallographic structure. In the mounted samples different type of constituents with different orientation of the grains was present. They were reacted with the etchant substances and revealed different metallographic structures.

Pre-etching Treatment: Previous to etching, the mounting surface was cleaned with soap and water completely until it became free from tarnish (oil and grease) matters used through the mounting process, to make sure the uniformity and the samples were cleaned and dried by silvo with selvette cotton cloth.

Microscopic Study of the Prepared Sample: With the help of plasticine, the polished and etched sample was mounted on the glass slide. After that, it was placed under a spring-loaded sample leveling device and pressed to be fixed and leveled on the glass slide. The microscope was focused on the surface of the sample at first observed under low power (10X) and then under high magnification (200 X). The whole cross-section was observed minutely by moving the slide in X and Y direction. Then the image was transferred to the closed computer, and the photographs were taken. The sample was first examined in a polished condition and then with its etching condition [17, 18].

Solubility Studies: 50 mL of each different solvent (H_2O, Conc. HNO_3, HCl, 0.17- N HCl, 2N-NaOH and $CHCl_3$, CH_3OH, Dioxen and DMSO) system were mixed with all three sample (200 mg) separately in the different conical flask. The conical flask kept for overnight and heated gently on a hot plate. Meanwhile, some gooch crucibles were dried in an oven at 120 ºC and dehydrated in dissector. Crucibles were weighed in an electronic balance and noted in **Table 1, 2** and **3**.

The contents of the conical flask were filtered through the crucible by using a vacuum pump. Again the crucibles were dried in an oven and dehydrated in dissector and finally weighed with the residue. The final weights of the different sample were recorded in **Table 1, 2** and **3** and calculated the solubility in the different solvent [19].

RESULTS:

TABLE 1: SHOWING THE SOLUBILITY (IN PERCENTAGE) OF AS

Name of Solvent	Total weight of sample	Weight of residual (mg)	Amount of dissolution (mg)	Solubility in percentage (%)
H_2O	200mg	196	04	02
C_6H_6	200mg	190	10	05
CH_3Cl	200mg	190	10	05
CH_3OH	200mg	188	12	06
Dioxen	200mg	186	14	07
DMSO	200mg	186	14	07
HCl (0.17N)	200mg	168	32	16
HCl (Conc.)	200mg	146	54	27
HNO_3 (Conc.)	200mg	146	54	27
NaOH (2N)	200mg	176	24	12

The amount of dissolution = Total weight of the sample weight of residual.
The weight of crucible = 332.200 gm, weight of AS = 200 mg.

TABLE 2: SHOWING THE SOLUBILITY (IN PERCENTAGE) OF SSBS

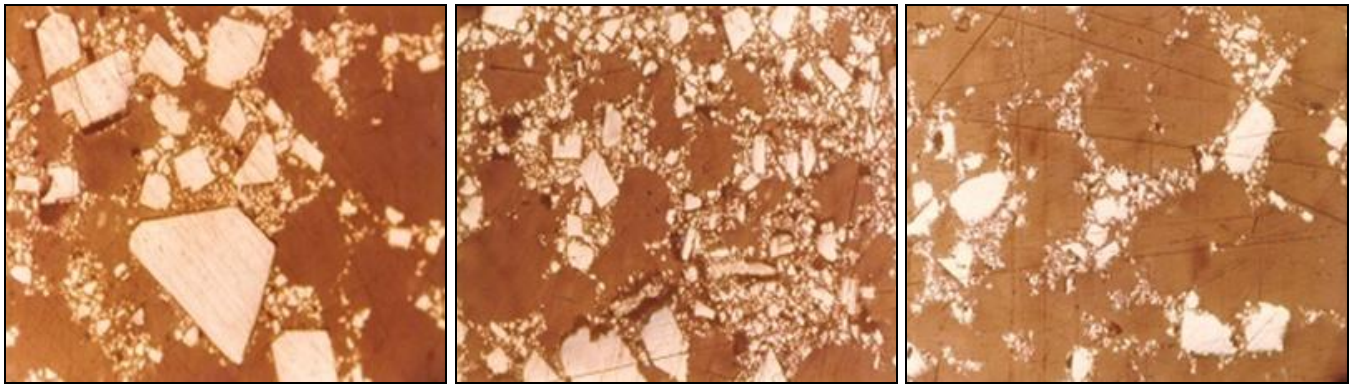
Name of Solvent	Total weight of sample	Weight of residual (mg)	Amount of dissolution (mg)	Solubility in percentage (%)
H_2O	200mg	192	08	04
C_6H_6	200mg	188	12	06
CH_3Cl	200mg	188	12	06
CH_3OH	200mg	188	12	06
Dioxen	200mg	182	18	09
DMSO	200mg	180	20	10
HCl (0.17N)	200mg	176	24	18
HCl (Conc.)	200mg	120	80	40
HNO_3 (Conc.)	200mg	118	82	42
NaOH (2N)	200mg	160	40	20

The amount of dissolution = Total weight of the sample weight of residual.
The weight of crucible = 332.200 gm, weight of SSBS = 200 mg.

TABLE 3: SHOWING THE SOLUBILITY (IN PERCENTAGE) OF SSTK

Name of Solvent	Total Weight of sample	Weight of residual (mg)	Amount of dissolution(mg)	Solubility in percentage (%)
H_2O	200mg	192	08	04
C_6H_6	200mg	180	20	10
CH_3Cl	200mg	182	18	09
CH_3OH	200mg	182	18	09
Dioxen	200mg	178	22	11
DMSO	200mg	178	22	11
HCl (0.17N)	200mg	160	40	20
HCl (Conc.)	200mg	110	90	45
HNO_3 (Conc.)	200mg	112	88	44
NaOH (2N)	200mg	160	40	20

The amount of dissolution = Total weight of the sample weight of residual.
The weight of crucible = 332.200 gm, weight of SSTK = 200 mg.



A **B** **C**

FIG. 1: (A) SHOW CLASSICAL STRUCTURE OF AS (B) SBSS (C) SSTK AT (Mag x 200)

DISCUSSION: Metallography is the study of the composition of metals and metal alloys from side to side the examination of samples with a metallurgical microscope [20]. It is the imaging of topographical or micro-structural features on prepared surfaces of materials. In this method, planar surfaces are prepared to achieve a polished finish. Chemical or other etching techniques are frequently used to describe macrostructure and microstructure features. Samples for microstructure evaluation are usually encapsulated in a plastic mount for handling during sample preparation. Sample preparation consists of pulverizing and then polishing using successively finer abrasives to obtain the desired surface finish. Etchants are particularly formulated for the precise sample material and assessment objectives. Sampling for metallography can be a random segment to evaluate representative mass properties or a section in an precise location to distinguish localized objects conditions [21].

The classical structure of AS (antimony sulfide) is shown in **Fig. 1A**. In this figure, a particle of antimony sulfide was dull in color and having a large size. In another **Fig. 1B, 1C** clearly revealed that after shodhana, a particle of antimony sulfide breaks into small particles with shining.

In the metallographic study, while preparing the samples, every metal responds differently, especially during polishing and etching. Antimony sulfide, being a soft metal, it has been observed that even polishing by using silvo with selvette cloth, it is not possible to get rid of the scratches on the surface. Another alternative has been used for polishing, in this method, a solution of methanol mixed has proved beneficial in polishing in getting rid of the scratches. A specific etching solution prepared by mixing nitric acid, acetic acid and water has proved useful and differentiated the metallic structure of antimony from compounds and other materials.

On etching, the metallic phase appears the dark in the shade. This has been observed clearly in the AS. While in shodhit Srotonjana of both samples of antimony sulfide were broken into smaller particles and found homogeneous in both the sample. The solubility of Srotonjana (AS, SBSS and SSTK) in 0.17-N solution of hydrochloric acid gives an idea that the percentage of solubility in gastric juice. The percentage of antimony absorbed less in solution so that it could hardly be toxic and yet effective. The dissolution test of above samples done in H_2O, C_6H_6, $CHCl_3$, CH_3OH, dioxane and DMSO organic solvent to check the soluble organic material present in the samples which may also reduce the toxic effect of antimony.

CONCLUSION: Thus this study shows that Ayurvedic shodhana process is effective to decrease the toxicity of Srotonjana, but there is no considerable change occurs in a chemical constituent of ashodhita or shodhita Srotonjana. The finding results revealed that after shodhana Srotonjana broke into smaller particles and became homogeneous in both shodhit the samples but no change in their chemical composition.

The solubility test of Srotonjana, all three samples are soluble in 0.17 N solution of hydrochloric acid (up to gastric juice concentration of HCl). While SSTK were found more soluble than AS and SSBS. Both shodhit samples of Srotonjana are soluble in organic solvent which indicate the presence of organic material in Srotonjana. The organic material reduces the toxicity of antimony as well as enhances the medicinal property.

ACKNOWLEDGEMENT: The authors are thankful to Department of metallurgy IIT, BHU, Varanasi for providing the metallographic facility.

CONFLICT OF INTEREST: Nil

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How to cite this article:
Gautam DNS: Metallurgical and solubility study of Ashodhit and Shodhit Srotonjana. Int J Pharm Sci Res 2018; 9(6): 2579-84. doi: 10.13040/IJPSR.0975-8232.9(6).2579-84.