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Research Article

XPS STUDY FOR CHEMICAL BASIS OF SILVER BASED *BHASMAS*

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ABSTRACT

This review concerns the appropriate study of X-ray Photo-electron Spectroscopy to analyze oxidation states in the herbo-mineral drugs. It describes multivalent forms of silver and silver based drugs. Silver exhibits multivalent forms with various phases like AgO, Ag_2O and Ag_2O_3 etc. Silver *Bhasma* is one of the potent Ayurvedic drug from herbo-metallic combinations. This traditional preparation is used for treating various ailments such as disorders related to eye and nerve, brain functioning and tuberculosis etc. Mixture of silver metal and herbal ingredients passes through *Bhasmikarana* method and then gets converted into organo-metallic complex i.e. silver *Bhasma.* The preparation method of *Bhasmas* aims at removing injurious substances which are foreign to the body from metals. The process of *Bhasmikarana* transferred the material from its inorganic state to the organic; therefore the assimilation of the substances for their therapeutic use will be easier in the human body. Silver *Bhasma* plays an essential role in Ayurvedic therapeutics due to its versatile qualities. Therefore, if a *Bhasma* is to be considered as a standard one, X-ray photoelectron spectroscopy should be one of the essential methods. Scanning electron microscope (SEM) technique was used to detect morphology of silver based *Bhasmas* and Energy dispersive X-ray spectroscopy (EDAX) detected elemental analysis. X-ray photoelectron spectroscopy is the useful platform to detect important chemical constituents as per their required oxidation states which are essential part in therapeutic use.

INTRODUCTION

This research work may lead to new understanding of essential oxidation states for silver metal in preparation of silver *Bhasma* for medicinal use on the basis of X-Ray photoelectron spectroscopy. Metal based *Bhasma* should possess desirable chemical compounds which will be responsible for specific therapeutic activity. Modern technique like SEM-EDAX is useful for detection of chemical composition and particle size.

Basic unit of a human body is a cell which needs essential minerals for the proper functioning. For absorption of these minerals in their natural form is difficult task for human body. Hence these minerals are provided by plants in the form of organic minerals through food.[1] Active principles decide quality of drugs hence standardization of herbal formulations is important.[2] Twenty metallic based *Bhasmas* such as Calcium, Iron, Zinc, Mercury, Silver, Potassium, Arsenic, Copper, Tin and Gemstones analyzed to confirm their purity on the basis of Neutron Activation Analysis.[3] To enhance drug delivery, metal from Ayurvedic metallic preparations serve as a nano-carrier to treat a variety of chronic ailments and are always mixed with butter, honey, milk or ghee, which enhance their biocompatibility in human body by reducing adverse effects of metals.[4] The advanced analytical techniques will provide the most potent tool to ensure the best quality of herbo-mineral research on the basis of therapeutic efficacy, safety and shelf life which may secure marketing approval from regulatory authorities.[5] Ash of silver i.e., silver *Bhasma* possesses significant sedative hypnotic potential and its particular doses applied on

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a mouse which acts as a mild to moderate sedative property.[6] Anti-inflammatory activity of *Raupya bhasma* studied with protein denaturation bioassay for in-vitro evaluation.[7] The study of X-ray diffraction analysis confirmed bi-phased (Ag + Ag_2O) form of the Ag_xO film during deposition at low OFR values and X-ray photoelectron spectroscopy determined presence of Ag_2O as compared to Ag_xO film which synthesized at higher OFR values.[8] The Auger parameter used to identify silver oxidation states instead of considering absolute binding energy of $Ag3d_{5/2}$ during X-ray photoelectron spectroscopy study.[9]

MATERIALS AND METHODS

Five commercial samples, (which include one sample specially synthesized by us for research purpose) of Silver *Bhasma* are selected. [Table 1: Commercial Samples of Silver *Bhasma*]

X-ray photoelectron spectra were collected on a VG, scientific ESCA -3-MK-2 electron spectrometer with an Mg-Ka X-ray source (non-monochromatic) and the anode was operated at 14 KV, 10mA. All the XPS analysis was recorded on the basis of similar spectroscopic parameters. Silver based *Bhasma* samples were analysed using EDAX machine for obtaining the relative percentage of constituent elements on machine JEOL JSM – 6360A Analytical SEM. [Table 2: EDAX- Chemical composition of Silver *Bhasmas*]

RESULTS AND DISCUSSION

In this paper, silver based *Bhasmas* are selected for study where silver exists solely in the phase of Ag (I), the oxides AgO, Ag_2O and mixture of them with Ag_2S. On the basis of selected literature, presence of AgO in this silver samples observed in two different oxidation states i.e. Ag (III) and Ag (I). The positions of the $Ag3d_{5/2}$ components and respective assignments are presented in Table:1. It is worth to mention that the doublet assigned to Ag (I) in Ag_2O and in AgO have different characteristics from the points of Ag5d core level spectra, which confirm the chemical state of silver samples after completion of *Bhasmikarana* process. [Table 3: Binding energies of Silver *Bhasmas*]

Silver shows multi-oxidation states in various phases like AgO, Ag_2O etc. AgO and Ag_2O are most stable forms with different crystalline structures. This study results in a shift of $Ag3d_{5/2}$ peak to a higher BE. On the basis of the references, silver and its oxides are probably present in the region of BE from 367.4 to 377.4 eV. This conclude that the presence of silver in the multi-oxidation states. In this published work, oxides of silver reported as $Ag3d_{5/2}$ from 367.6 to 368.5 eV for Ag_2O; and from 367.3 to 368.1 eV for AgO.[10] The Binding energies of $Ag3d_{5/2}$ peaks for Ag_2O and AgO, are in the regions from 367.7 to 368.4 eV and 367.3 to 368.1 eV respectively on the basis of NIST XPS Database.[11] Resolution of spectra is complicated task because silver metal atoms and its oxide forms show close spectral characteristics by XPS method.[12] But Auger parameter will be applied to get better resolutions in the spectra of silver and its complex forms.[13] Wagner introduced the Auger parameter concept which proved essential method for identifying oxidation states of various elements.[14] Electrical conductivity difference is essential factor for atoms and its compound form which provides significant signals for silver metal atoms and its compounds by XPS method. The BE of $Ag3d_{5/2}$ and $Ag3d_{3/2}$ peaks 367.73 and 373.71 eV; reported for the presence of Ag_2O state in a sample.[15-20] *Bhasmikarana* is the significant step in the preparation of *Bhasmas* which removes toxicity from metallic oxide state and helps to enrich medicinal properties into it. This process involves conversion of the metallic state to higher oxidation state.[21]

The SEM photographs shows morphology of selected samples of silver based *Bhasmas*. In S_1 agglomeration is observed and the big particles are covered by small dusty particles. The particle size was about 50 micron-meter. In S_3 sample, the particle size was about 40 micron-meter and it is composed of highly porous agglomerates. In S_2 also agglomeration is observed and the big particles are covered by small dusty particles as that of for S_1. The particle size about 30 micron-meter. The S_4 sample shows particles of about 40 micron-meter. S_0 sample is composed of porous agglomerates and particle size is about 30 micron-meters with loss of their grain boundaries. For the preparation of silver based *Bhasmas*, higher degree of agglomeration may be a consequence of repeated number of *Putas*.[22] Energy dispersive X-ray spectroscopy (EDAX) is used for elemental analysis of silver based selected *Bhasma* samples. EDAX spectra suggested main constituent as silver oxide with traces of other elements Ca, C, Si and S etc. However use of medicinal herbs in synthesis of *Bhasmas* is associated with the presence of these elements.[22-23] Fig.1 The EDAX spectra of Silver *bhasma* [S_1]

Table 1: Commercial Samples of Silver *bhasma*

S.No.	Sample No.	Name of the Pharmacy
1.	S_0 *Bhasma*	Silver *Bhasma* in *Kaduindravana*
2.	S_1 *Bhasma*	*Baidynath*
3.	S_2 *Bhasma*	*Dhootpapeshwar* Ltd.
4.	S_3 *Bhasma*	*Unza* Pharmacy
5.	S_4 *Bhasma*	*Ayurved Arkashala* Ltd.

Table 2: EDAX:- Chemical composition of Silver *bhasmas*

S.No.	Sample	Empirical composition
1	S_0 *bhasma*	Ag (28.82), O (39.98), C (18.49), Ca (4.44), Si (2.02)
2	S_1 *bhasma*	Ag (36.76), O (37.31) , C (9.81), S (7.37)
3	S_2 *bhasma*	Ag (41.47), O (30.57), C (16.75), S (8.54)
4	S_3 *bhasma*	Ag (10.82), O (53.29), C (5.61), Si (6.53), S(1.45)
5	S_4 *bhasma*	Ag (18.41), O (9.93), C (20.53), S (30.55)

Table 3: Binding energies of Silver *Bhasmas*

Binding Energy (eV) Ag $3d_{5/2}$					
S_0	**Ag_2S**	**Ag_2O**	**AgO**	**Mix**	**Assignments**
	-----	-----	371.49	370.34	Ag (III) saturated
	-----	-----	-----	373.09	Ag (III) saturated
	-----	-----	-----	375.49	Ag (III) saturated
	-----	-----	-----	377.49	Ag (III) saturated
S_1	-----	-----	367.43	368.72	Ag (I)
	-----	-----	-----	374.04	Ag (III) saturated
S_2	367.1	367.73	-----	-----	Ag(I)
	-----	-----	-----	373.01	Ag (III) saturated
	-----	-----	-----	373.36	Ag (III) saturated
S_3	-----	367.79	-----	368.67	Ag (I)
	-----	-----	-----	368.80	Ag (I)
	-----	-----	-----	370.22	Ag (III) saturated
	-----	-----	-----	370.39	Ag (III) saturated
	-----	-----	-----	376.94	Ag (III) saturated
S_4	-----	-----	369.94	370.3	Ag (III) saturated
	-----	-----	-----	376.5	Ag (III) saturated

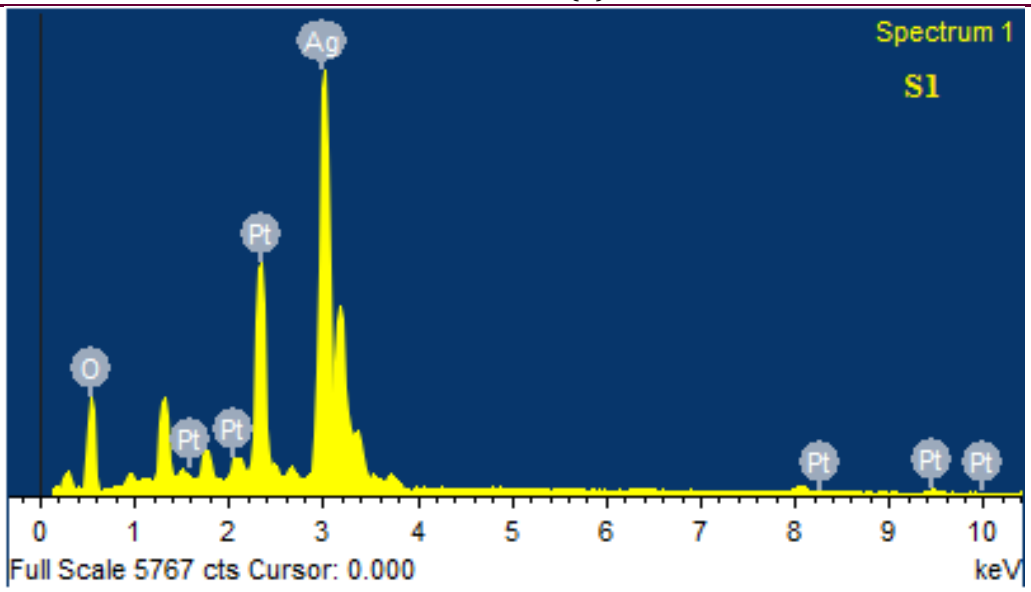


Fig.1 The EDAX spectra of Silver bhasma [S1]

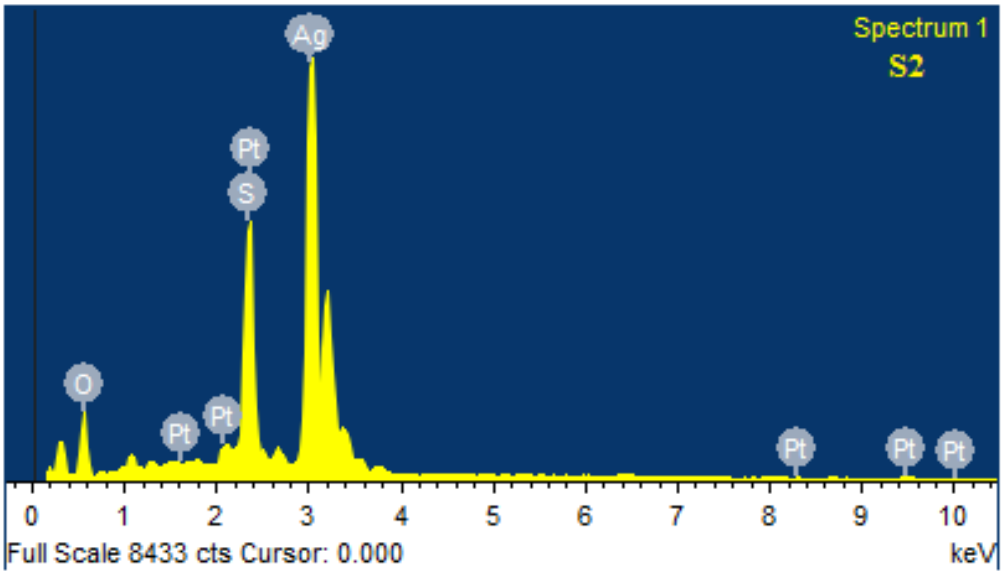


Fig.2 The EDAX spectra of Silver *bhasma* [S_2]

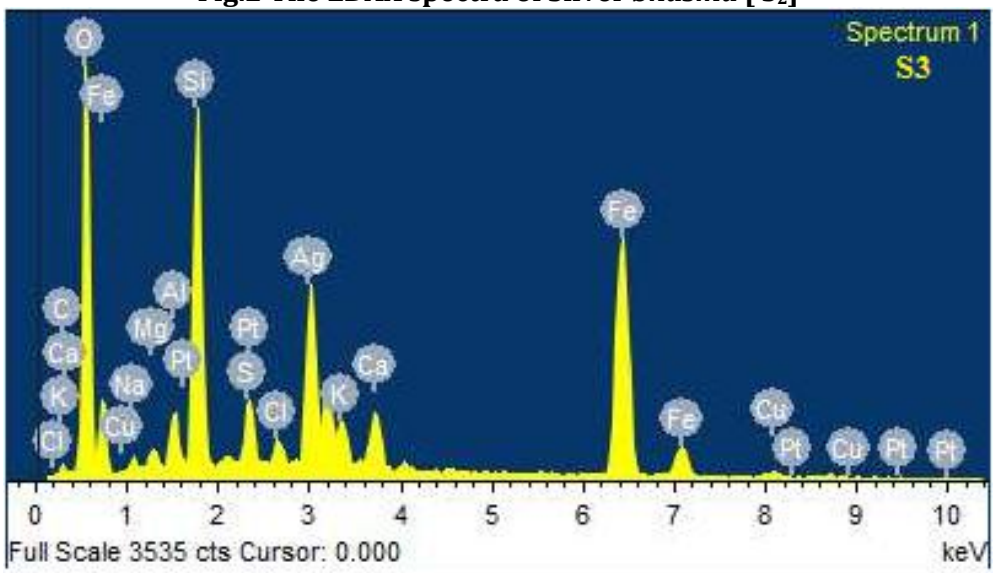


Fig.3 The EDAX spectra of Silver *bhasma* [S_3]

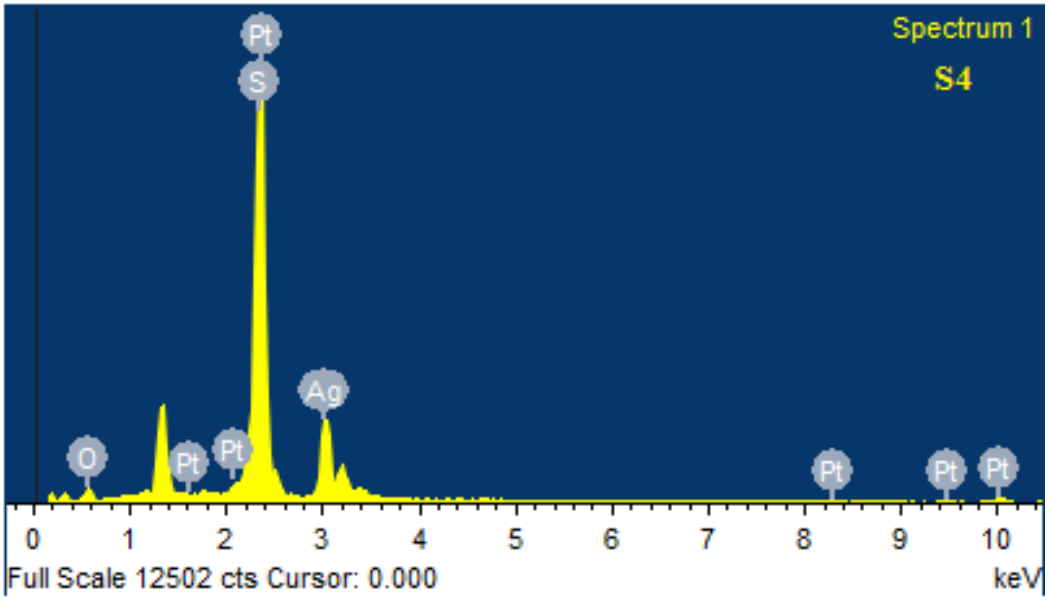


Fig.4 The EDAX spectra of Silver *Bhasma* [S_4]

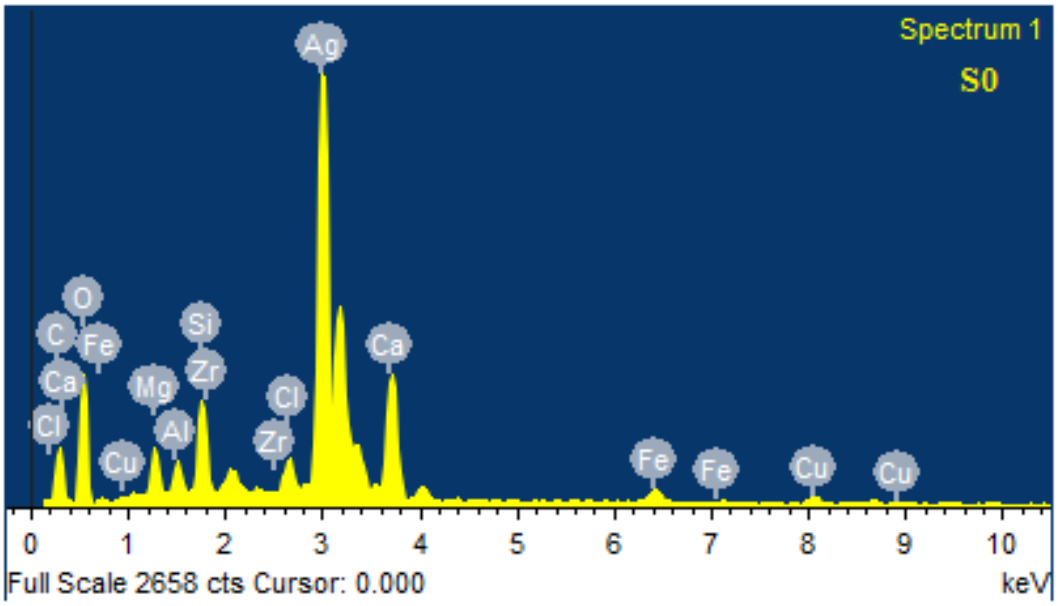
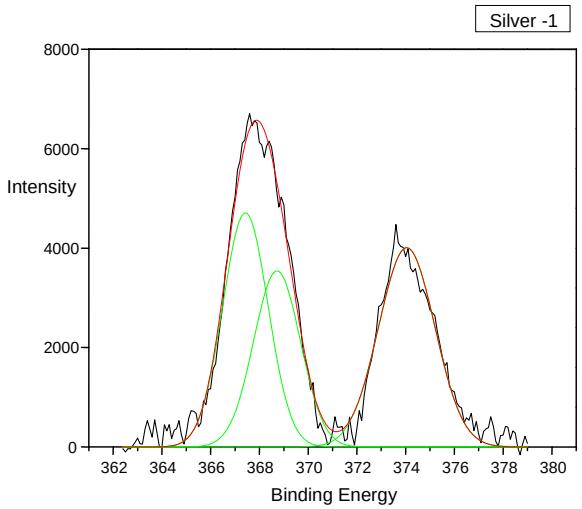


Fig.5 The EDAX spectra of Silver *bhasma* [S_0]



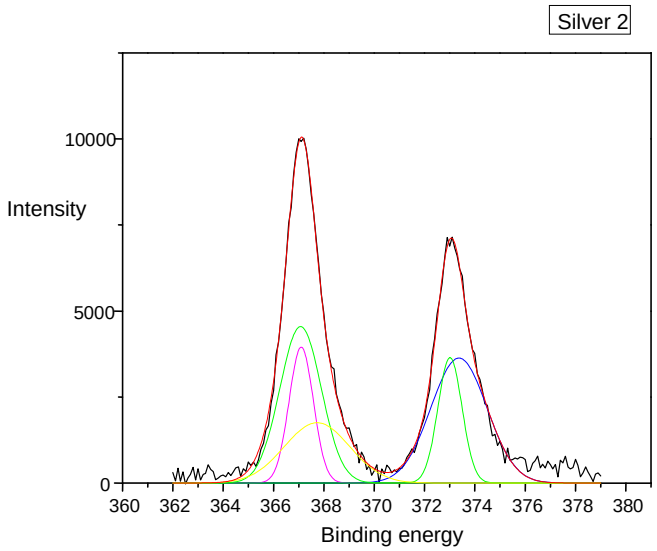
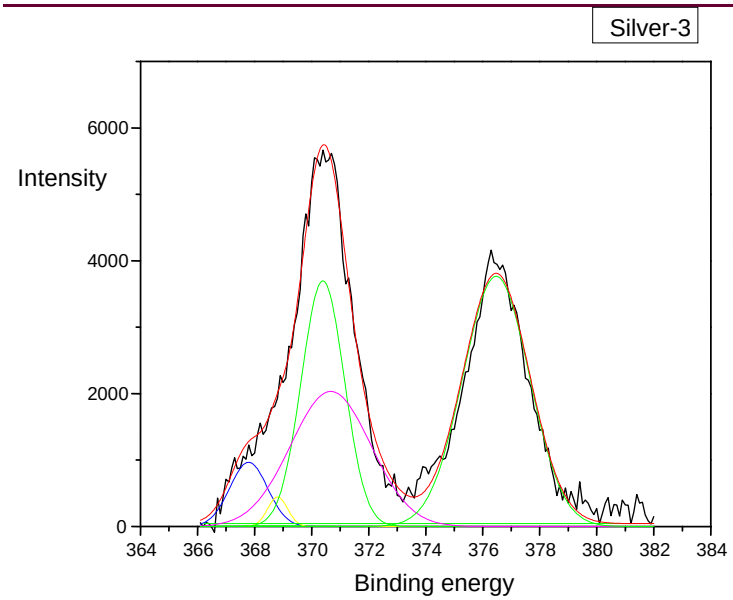


Fig.1 The XPS spectra of Silver bhasma [S1] **Fig.2 The XPS spectra of Silver bhasma [S2]**



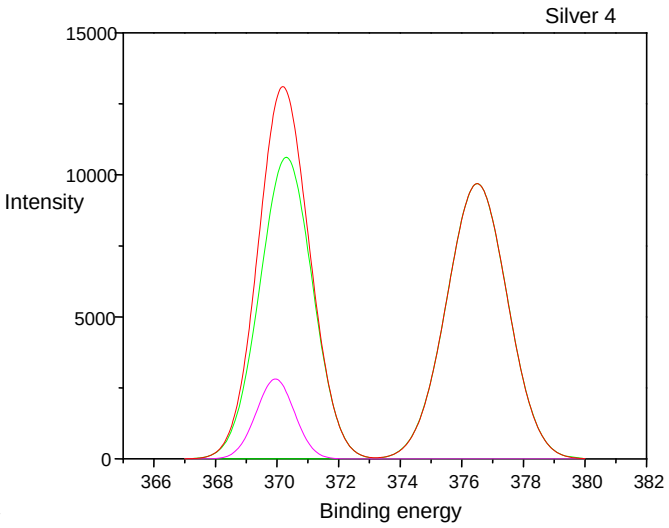


Fig.3 The XPS spectra of Silver bhasma [S3] Fig.4 The XPS spectra of Silver bhasma [S4]

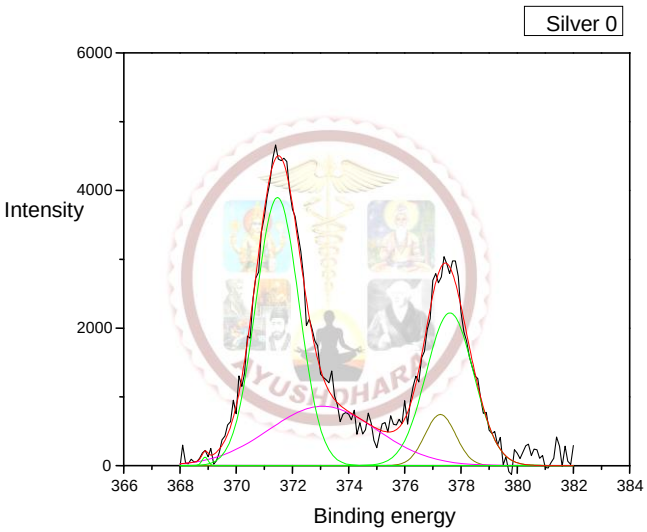


Fig.5 The XPS spectra of Silver bhasma [S0]

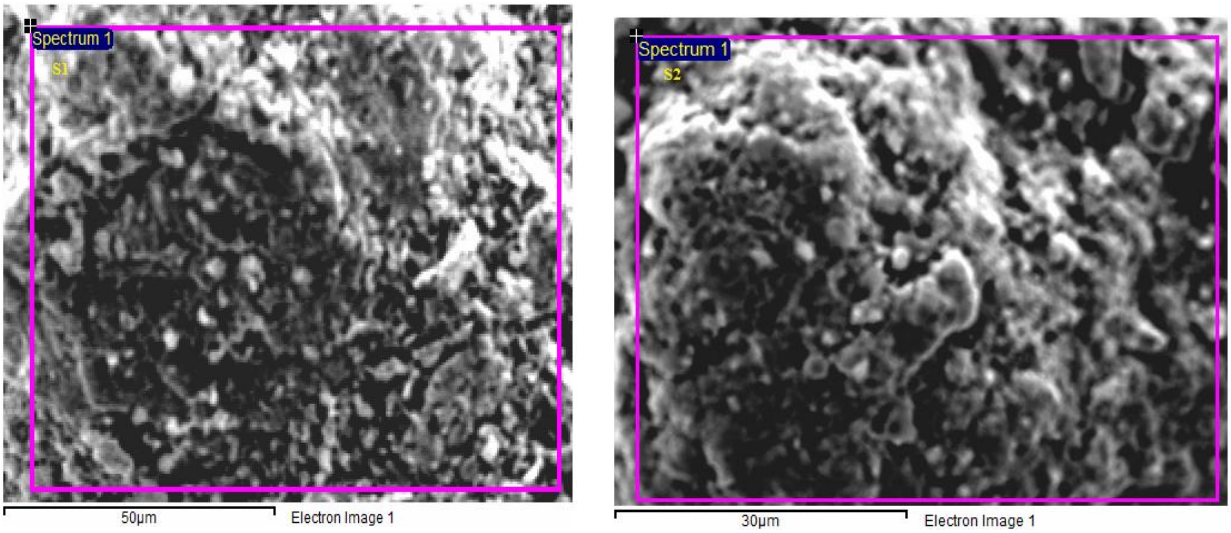


Fig.2 The SEM photograph of Silver *bhasma* [S_2] Fig.3 The SEM photograph of Silver *bhasma* [S_3]

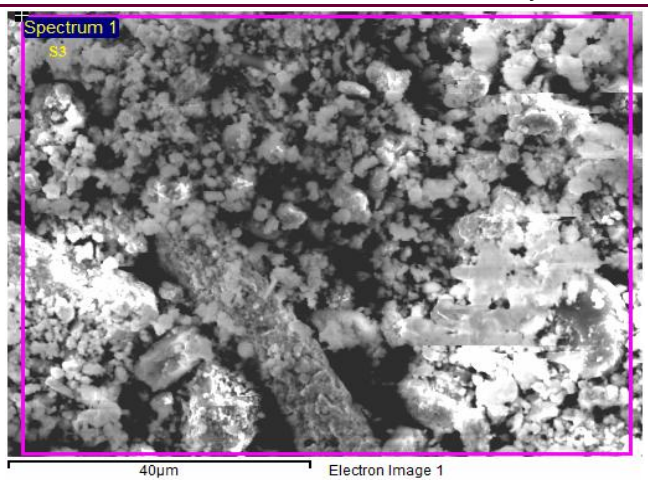


Fig.4 The SEM photograph of Silver *bhasma* [S_4]

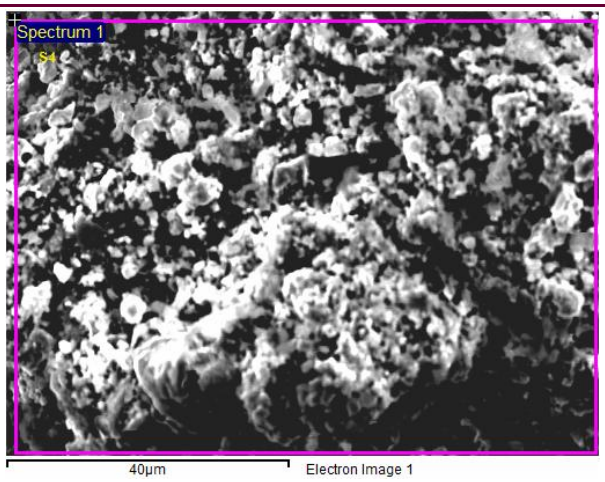
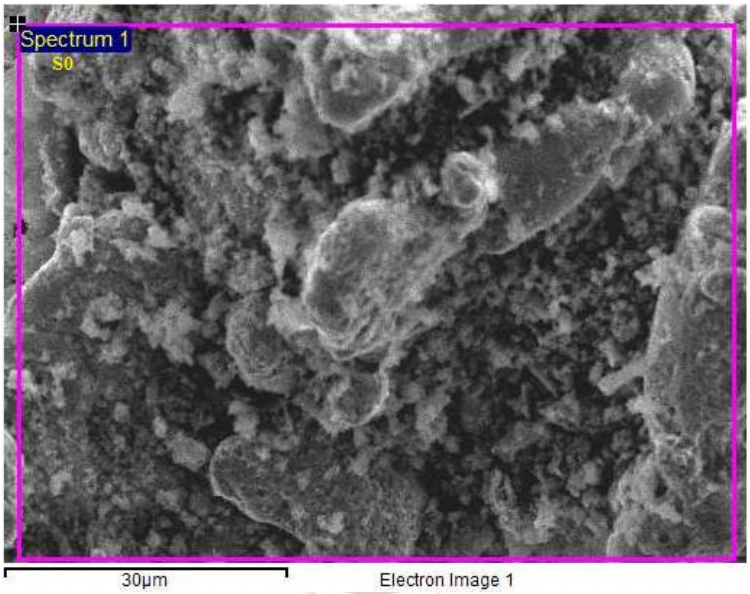


Fig.5 The SEM photograph of Silver *bhasma* [S_0]



CONCLUSION

XPS have been used to study the nature of silver states in the selected samples. This paper presents quantitative analysis of chemical composition of silver based Ayurvedic *Bhasmas* with lines of photoelectrons by X-ray photoelectron spectroscopy. The validity of procedures used for *Bhasmikarana*, will be proved on the basis of considering results of chemical compositions of *Bhasmas*. Conventional methods for preparation of Ayurvedic drugs may show variation in composition and properties in desired products. For quality assurance, standardization is an essential prerequisite to ensure safety with efficacy to Ayurvedic drugs. Standardization of mineral based *Bhasmas* include data of crude mineral, mineral origin authentification, identification of chemical constituents by various parameters like dosage with their stability, self life, chemical compositions and toxicity assessment. EDAX and SEM have been used for elemental analysis and to determine particle size. This article deals with XPS technique for developing authentic analytical method which will be helpful for quality control and standardization of Ayurvedic drugs.

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