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Physico-Chemical Standardization of Tanaka (Borax): An Ayurvedic Mineral Drug

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Tanaka, which is one among the Kshatriyas has been used since very long time in Ayurveda. It has a wide range of therapeutic applications, including diseases like Vrana (ulcers), Shvasa (asthma), Kasa (cough), etc. The present study was conducted to generate a fingerprint for raw and purified Tanaka by using techniques like X-ray Diffraction (XRD), SEM, EDAX and Chemical analysis to find out the structural and chemical compositions. The raw Tanaka contains $Na_2B_4O_7$ $10H_2O$ (structure- Rhombohedral), $Na_2B_4O_7$ $5H_2O$ (structures- Orthorhombic), and purified Tanaka contains $Na_2B_4O_7$ $5H_2O$ (structures- Orthorhombic) by the XRD method. Scanning Electron Microscope (SEM) studies showed that Tanaka was uniformly arranged in agglomerates of size 10 microns as compared to the raw Tanaka which showed an arrangement of grains of size 3 microns. Quantitative chemical analysis showed that Purified Tanaka contains more Boran (13.48%) compared to raw Tanaka (10.08%). This study revealed that identification of physicochemical changes, standardized Tanaka carrying out during the purification procedures and it is useful for further research.

Keyword: Tanaka, Borax, Purification, XRD, SEM, Quantitative analysis

INTRODUCTION: Tanaka ($Na_2B_4O_7$ $10H_2O$) is composed of boric acid and soda. In the native state it exists as an impure saline incrustation of a dirty-white colour.

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It exists as crystalline tough masses or in the form of translucent irregular masses, and when exposed to air it becomes opaque [1]. Tanaka (Borax) is a salt of tetra Boric acid, an important compound of Boron, which is also known as sodium biborate [2]. Pandora (white coloured) is the best out of Sphatikabha, Gudaprabha and is useful in the procedures [3, 4].

In the text of Rasa shastra, Tanaka has its own important role because of its high therapeutic value and being used in many Parada Karmas.

Rasa Vaidyas have included Tanaka under the groups of Ksharatraya [5, 6], Ksharapanchaka [7], Dravaka gana [8, 9], Mitra panchaka [10], etc. In the procedure of Apunarbhava [11] test for Bhasma, Prada Vela Samskara [12], Vidadi mukhakara dravyas [13], Tanaka are also used to cleanse gold and silver [14].

Since, the impure Tanaka causes Vomiting and Giddiness [15, 16], purification of Tanaka is very necessary before the administration or adding into medicinal compounds.

Materials and Methods:

1. Purification of Tanaka
2. X-ray diffraction study of Tanaka
3. Scanning Electron Microscope (SEM)
4. Energy Dispersive X-Ray Analysis
5. Quantitative chemical analysis Tanaka

1. Purification of Tanaka (Borax):

Raw Tanaka is taken in a clean and dry Khalva yantra and pounded well to prepare powder. This powder is taken in to a Sharava (earthen pot) then it is heated in Mandagni (Mild heat), followed by Tivragni (maximum heat), until all the water content in the Tanaka is completely evaporated. Finally Tanaka is obtained as a white coloured puffy light substance [17] (fig.1). After three times of purification almost 50% of weight is reduced, because of the evaporation of water in the raw Tanaka (Table 1).

Table 1: Observations during Tanaka Sodhana process

S.No.	Parameters	Expt-1	Expt-2	Expt-3
1.	Raw Tanaka	250 g.	250 g.	250 g.
2.	Purified Tanaka	120 g.	110 g.	115 g.
4.	Loss	130 g.	140 g.	135 g.
5.	Time taken for purification	2 hours	1 hour 50 mints	1 hour 50 mints
6.	Date of experiment	03-11-2006	21-02-2007	18-01-2008

Figure 1: Purification of Tanaka (Borax)

2. X-ray diffraction study of Tanaka

Powder X-Ray diffraction (XRD) studies were carried out in the department of Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology (IICT), Hyderabad.

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 [18].

X-Ray Diffraction study is a powerful procedure for detecting the presence of various phases in a given sample. The basic principle of the phase analysis using powder XRD technique lies in the presence of diffraction peaks corresponding to various inter planar (dhkl) spacings which are the characteristics of a given material. The relative intensities of various peaks occurring at different'd' spacings are also different for different phases.

Preparation of Sample:

The sample preparation for the analysis was done using standard XRD procedure. The sample was powdered to 100 mesh size using mortar and

pestle, to get a homogenous powder mixture. The powder was then spread onto a double-side tape with a spatula, and then placed on a cavity with plastic holder. Care was taken to fill the powder uniformly in to the mount. It was exposed to x-ray beam of intensity 35KV and 20MA. All the peaks were recorded on the chart, and the corresponding 2θ values were calculated. Results are summarized in figure 2, 4 and Table 2, 3.

Raw Tankan (Borax):

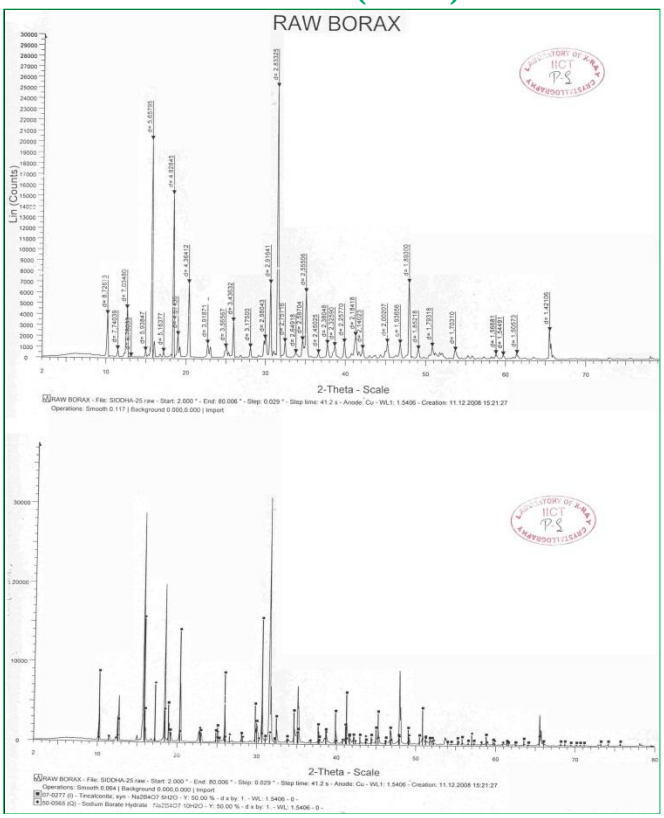

Figure 2: X-ray diffraction of the Raw Tanaka (Borax)

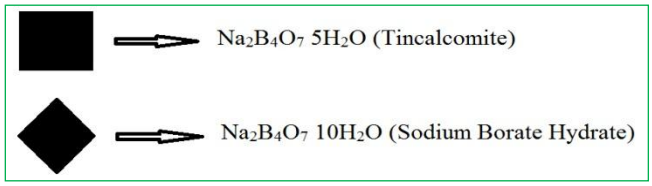

Figure 3: Structure of the Borax

Table 2: Showing 2θ vs 'd' spacing values Impure Tankana (Before purification)

S.No.	Position (2Th)	Height (cts)	d-spacing (A)	Chemical structure
1.	31.3	30000	2.83325	Orthorhombic
2.	15.9	24000	5.65795	Orthorhombic
3.	30.7	11000	2.91641	Rhombohedral
4.	20.2	10500	4.36412	Rhombohedral
5.	10.2	8000	8.72673	Rhombohedral
6.	26	7000	3.43632	Rhombohedral
7.	17.2	7000	5.16377	Orthorhombic
8.	41.2	6000	2.18418	Rhombohedral
9.	29.8	5000	2.98043	Rhombohedral
10	19	5000	2.58704	Rhombohedral
11	18.9	4800	4.67436	Rhombohedral
12	50.8	4700	1.79318	Rhombohedral
13	34.6	4000	2.25770	Rhombohedral
14	45.2	4000	2.00207	Rhombohedral

Pure Tankan:

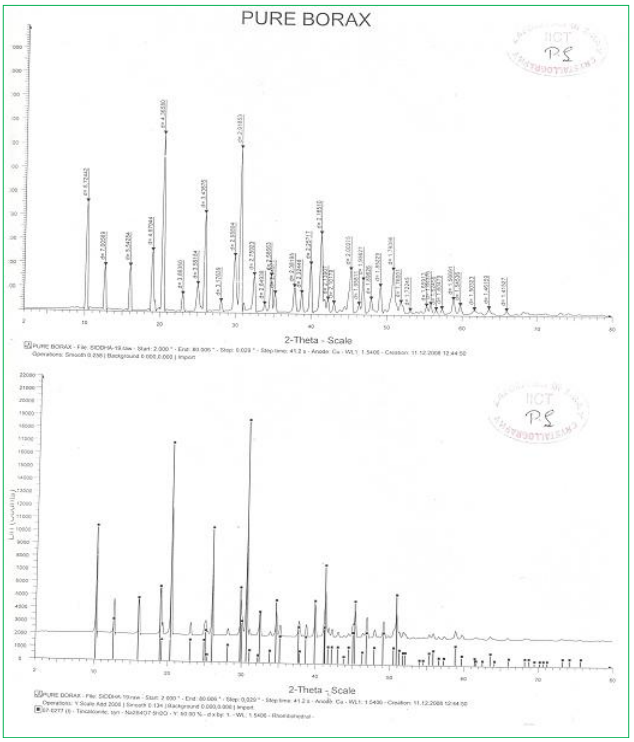

Figure 4: X-ray diffraction of the Pure Tanaka (Borax)

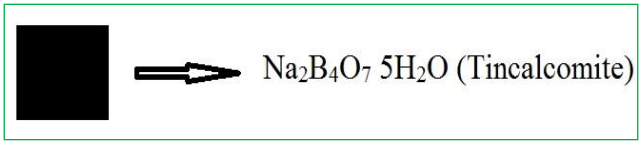

Figure 5: Structure of the Borax

Table 3: Showing 2θ vs 'd' spacing values Pure Tanaka (After purification)

S.No.	Position (2Th)	Height (cts)	d-spacing (A)	Chemical structure
1.	30.3	19000	2.91853	Rhombohedral
2.	20.2	17000	4.36580	Rhombohedral
3.	10.2	10500	8.72442	Rhombohedral
4.	26	10500	3.43876	Rhombohedral
5.	41.1	8000	2.18510	Rhombohedral
6.	19.9	6000	4.57944	Rhombohedral
7.	29.9	6000	2.98894	Rhombohedral
8.	50.8	5500	1.79396	Rhombohedral
9.	15.9	5000	5.54284	Rhombohedral
10.	34.6	5000	2.58663	Rhombohedral
11.	39.9	4500	2.25717	Rhombohedral
12.	45.2	4500	2.00275	Rhombohedral
13.	32.2	4000	2.75923	Rhombohedral
14.	25	2000	3.56184	Rhombohedral

RESULTS:

The XRD analysis can show the chemical structure of the borax before and after the purifications., In X-RD Graph d-spacing [Å] values of major peaks are compared with standard JCPDS card table for $Na_2B_4O_7$ $5H_2O$ (structure- Rhombohedral) and $Na_2B_4O_7$ $10H_2O$ (structures- Orthorhombic). Hence this card is studied for crystal structure after the purification $Na_2B_4O_7$ $10H_2O$ also converted into $Na_2B_4O_7$ $5H_2O$ (structures- Orthorhombic) because while heating the water molecules are evaporated and the final formula shows only $Na_2B_4O_7$ $5H_2O$. The variations of structures of raw Tanaka and purified Tanaka can be show in Figure 3, 5.

3. Scanning Electron Microscope

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. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. 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 SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). The design and function of the SEM is very similar to the EPMA and considerable overlap in capabilities exists between the two instruments [19].

During SEM inspection, a beam of electrons is focused on a spot volume of the specimen, resulting in the transfer of energy to the spot. These bombarding electrons, also referred to as primary electrons, dislodge electrons from the specimen itself. The dislodged electrons, also known as secondary electrons, are attached and collected by a positively biased grid or detector, and then translated into a signal. To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analysed, and translated into images of the topography being inspected. Finally, the image is shown on a CRT.

Chemical Characterization of Tankan by Scanning Electron Microscope (SEM) carried out in the Defence Metallurgical Research Laboratory (DMRL), Hyderabad, A.P.

Preparation of Sample:

The fine powder of Tanaka was placed inside the microscope's vaccum column through an airtight door, and then the air was pumped out. After the air was pumped out of the column, a beam of electrons was emitted by an electron gun from the top. This beam travels downward through a series of magnetic lenses designed to focus the electrons to a very fine spot. Near the bottom, a set of scanning coils made the focused beam to move

back and forth across the mounted sample, row by row.

As the electron beam hits each spot on the sample, secondary electrons are backscattered from its surface. A detector counts these electrons and sends the signals to an amplifier. The microscopic photographic image was built up from the number of electrons emitted from each spot on the sample.

Results:

The SEM pictures with 1.00 KX, resolutions are taken for the samples. The surface of the Tanaka grains is uniformly arranged in agglomerates, it seems to develop first at exposed edges and corners of fractured surfaces. (Fig. 6, 7).

Particle size analysis by SEM method:

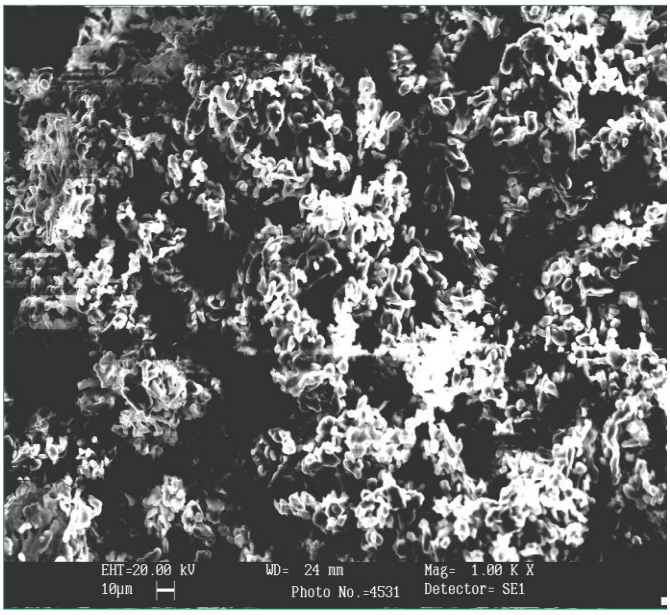

Figure 6: SEM image of raw Tankan 1.00 KX

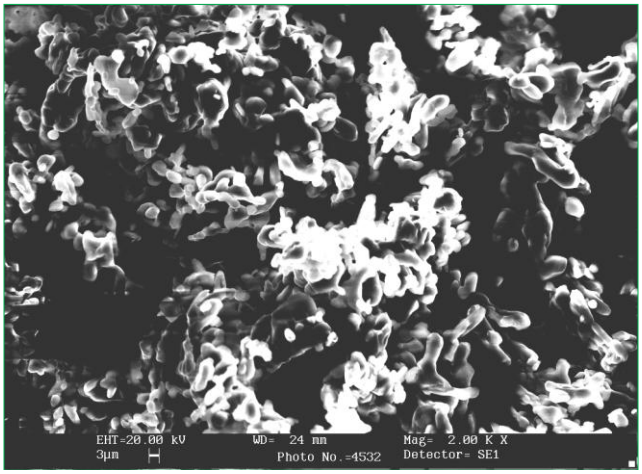

Figure 7: SEM image of purified Tankan 1.00 KX

4. Energy Dispersive X-Ray Analysis

A SEM may be equipped with an EDX analysis system to enable it to perform compositional analysis on specimens. EDX analysis is useful in identifying materials and contaminants, as well as estimating their relative concentrations on the surface of the specimen.

Chemical Characterization of Tanaka by Energy Dispersive X-Ray Analysis (EDAX) was carried out in the Defence Metallurgical Research Laboratory (DMRL), Hyderabad, A.P.

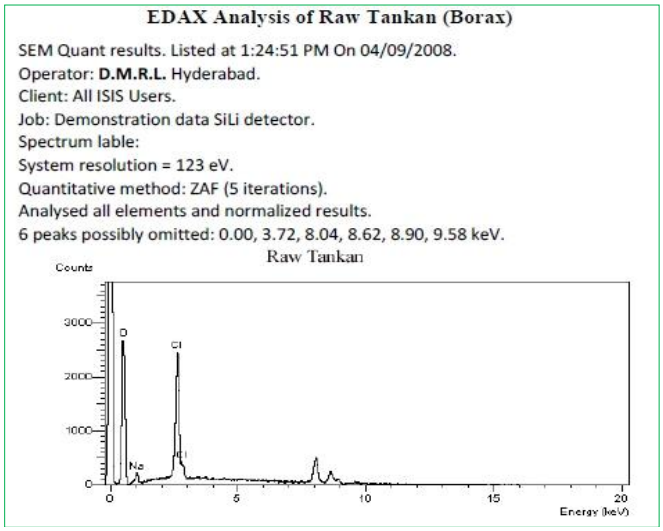

Figure 8: EDAX Graph of Raw Tankan

Standards:

O K	Quartz	01/12/93
Na K	Albite	02/12/93
Cl K	KCl	15/02/94

Results:

Elements	Spect. Type	Element %	Atomic %
O K	ED	77.43	87.58
Na K	ED	3.27	2.57
Cl K	ED	19.30	9.85

*=<2 Sigma

Figure 9: Element Composition of raw Tankan

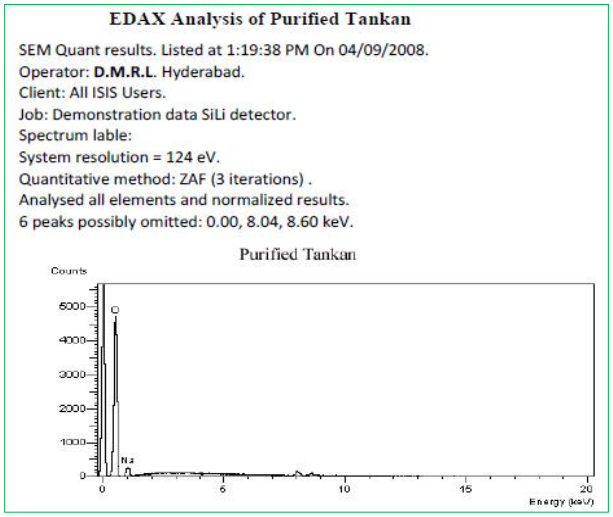

Figure 10: EDAX Graph of Purified Tankan

Results:

In the EDAX analysis, EDAX Graph was generated (Fig. 8, 10), the detected elements in raw Tanaka was Oxygen, Sodium and Chlorine 77.43%, 3.27%, 19.30% respectively (Fig. 9). In Purified Tanaka was Oxygen, Sodium 94.94%, 7.12% respectively (Fig. 11). The absorption of the soft x-rays are not detected of elements below an atomic number of 11 (Na) [20]. Because of this reason, Boran was not detected in both the samples.

Standards:

O K	Quartz	01/12/93
Cl K	KCl	15 /02/94

Results:

Elements	Spect. Type	Element %	Atomic %
O K	ED	94.94	94.94
Na K	ED	7.12	5.06
Total		100.00	100.00

*=<2 Sigma

Figure 11: Element Composition of purified Tankan

5. Quantitative chemical analysis of raw Tanaka and purified Tanaka

Tankan (Borax) by using classical method which is mentioned in Quantitative inorganic analysis by Vogal [21].

Procedure: weigh the sample, accurately 1 g. of the sample is transferred into a 250 ml. volumetric flask, and make up to the mark with distilled water and shake well till sample is completely dissolved. Titrate 25 ml. of the solution with standard 0.1N hydrochloric acid, using methyl orange as indicator.

To another 25 ml. of the solution, add the quantity of standard mannitol, and shake until dissolved; add a few drops of phenolphthalein and titrate with standard 0.1 N carbonate free sodium hydroxide solution to the first permanent faint pink colour. Add a further 0.5 g. of mannitol: if the solution becomes colourless, add more standard sodium hydroxide until the pink colour reappears. Repeat the process until the addition of mannitol has no effect up on the end point.

Calculate the percentage of Boran in sample from the Formula, given below

I ml 1n NaOH = 0.010811 g. of Boran

Test Results:

Table 3: Chemical analysis of raw Tanaka and suddha Tanaka

S.No	Test Parameter	Boran as B (% by mass)
1.	Raw Tankan	10.08
2.	Purified Tankan	13.48

Conclusion

The raw Tanaka contains $Na_2B_4O_7$ $10H_2O$ (structure- Rhombohedral), $Na_2B_4O_7$ $5H_2O$ (structures- Orthorhombic), and purified Tanaka contains $Na_2B_4O_7$ $5H_2O$ (structures- Orthorhombic) by the XRD method. After the purification $Na_2B_4O_7$ $10H_2O$ also converted into $Na_2B_4O_7$ $5H_2O$ (structures- Orthorhombic) because while heating the water molecules are evaporated and the final formula shows only $Na_2B_4O_7$ $5H_2O$. Scanning Electron Microscope (SEM) studies showed that the Tanaka was uniformly arranged in agglomerates of size 10 microns as compared to the raw Tanaka which showed an arrangement of grains of size 3 microns. Quantitative chemical analysis showed that Purified Tanaka contains more Boran (13.48%) compared to raw Tanaka (10.08%). This study revealed that identification of physicochemical changes, standardized Tanaka carrying out during the purification procedures and it is useful for further research.

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