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A comparative study of structural properties and Antacid activity of some commercial samples of *Shauktika bhasma*

Research Article

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Abstract

Backgroud: *Shauktika Bhasma* is a traditional ayurvedic medicine which is an excellent remedy for a number of stomach disorders, hyper acidity and ulcers. It is prepared from mother pearl by subjecting the powder of mother pearl to traditional ayurvedic processes. To get a worldwide recognition to such supreme ancient medicine, it is necessary to reinvestigate it on the basis of modern analytical techniques.In the present work a comparative study of some commercial samples of *Shauktika bhasma* is undertaken to understand the current status of this *bhasma* and to explore its acid neutralization capacity using back titration method. Materials and Methods: Samples from renowned pharmacies are collected and subjected to chemical and structural investigations using analytical techniques like XRD, IR and UV. Back titration method is applied to find acid neutralizing capacity of these *bhasma* samples. Conclusion:The study reveals that *Shauktika bhasma* is chemically calcite form of calcium carbonate. Antacid activity of the samples is reported for these samples using simple titration method which is useful to estimate number of moles neutralized for a quick comparison. The antacid activity of the samples is found to be inversely proportional to the crystallite size of the *bhasma*.

Keywords: *Shauktika Bhasma,* XRD, IR, Back Titration, Antacid activity.

Introduction

Shauktika bhasma which is also known as *Shukti bhasma* is prepared from mother pearl and is a marin based biomineralized material and is a rich source of calcium. Shells of mother pearl are available in tremendous amount of quantity in sea having natural origin is used as starting material of *Shauktika bhasma*. *Sauktik bhasma* is used as effective medicine to treat hyper acidity and many other stomach disorders. It is believed that the whole process of preparation of the *bhasma* (*bhasmikarna*) eliminates unwanted and toxic elements of the material and imparts medicinal value to it. Due to process of *bhasmikarana* the *bhasma* becomes easily assimilable to the body.This property of *bhasmikarana* was recognized by ancient *Ayurvedicians* and even now days, ayurvedic pharmacies are preparing *Shauktika bhasma* following the *bhasmikarana* methods mentioned in standard ayurvedic texts (1).

Reinvestigation of *Ayurvedic bhasmas* is becoming an area of keen interest for researchers(2-6). Investigations on *Shauktikaa bhasma* are reported previously by ketkar *etal* (7-8) and Dubey etal (9).The effect of *bhasmikaran* process on chemical, structural properties of *bhasma* and its correlation with the drug action is a challenging task and this area is still open for the scientists.

The present study aims to investigate, the current status of *Shauktika bhasma*,for which four samples of reputed pharmacies are selected. Their chemical and structural characterization is carried out and the results are compared with each other. *Shauktika bhasma* is used particularly to neutralize the excess harmful acid in the stomach and maintain the pH and to keep away from damage due to free radical production. To verify this property, using analytical technique, the antacid activity of these samples is determined and compared with each other and also with standard calcium carbonate. Commercial samples under present study, exhibit calcite form of calcium carbonate.The correlation between antacid capacity of *Shauktikaa bhasma* (using back titration method) and its crystallite size is also reported.

Materials and Methods

Four samples from different reputed pharmacies were collected for the present study and are named as R-1, R-2, R-3 and R-4. The fifth sample R-5 is AR grade Calcium carbonate purchased from Aldrich.The percent calcium from each of the sample was calculated by titrating it against Standardized ethylene diammine tetraacetic acid(EDTA) using Erichrome black T indicator, following standard procedure of complexometric titration(10).The antacid activity of each sample was obtained using back titration method. All chemicals used were of AR grade. The XRD spectra

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were recorded on Bruker model from angle, 2θ= 20 to 80. The IR spectra of *Shauktika bhasma* samples were recorded on ThermoScientific (Nicolate iS5) IR spectrometer in the FT IR range.The electronic spectra of the bhasma samples were recorded in KBr disc in the region 200-1100 nm keeping pure KBr disc as the reference, on Shimadzu UV double spectrophotometer.

Results and Discussion

Determination of Percent Calcium using Complexometric Titration

Shauktika bhasma is calcium carbonate based *bhasma* which is supposed to contain calcium as the main constituent. Therefore all four samples are analyzed using standard complexometric titration method (10) for the estimation of of percent calcium present in the samples. The weighed(W) amount of each bhasma sample was dissolved in standardized hydrochloric acid(HCl).The solution was then titrated against Standardized ethylene diammine tetraacetic acid (EDTA) using Erichrome black T indicator. From the constant burette reading(CBR) the percent calcium was determined as follows.

First the amount of calcium(gm) is determined using the correlation:

1000 ml of 1M EDTA = 40 gm of Calcium
[(CBR of EDTA for bhasma solution) × (Exact molarity of EDTA)] × 40/ 1000= X gm of Calcium in the bhasma solution used for the titration.

From X gm of Calcium, amount of Ca (A gm) in the W gm (weighed sample) of bhasma was estimated .
% Calcium in the bhasma sample = (A × 100) / W

The % calcium for all samples is depicted in the (Table-1).

Table 1: Percentage of Calcium From *Shauktika Bhasma* samples

Sr.No	Name Of Sample	Percentage (%) of Calcium	Percentage (%) of $CaCO_3$
1	R1	37.33	93.32
2	R2	34.66	86.65
3	R3	42.66	100
4	R4	34.60	86.60
5	R5 ($CaCO_3$)	40.00	100

The standard AR grade calcium carbonate contains 40% calcium in it. Complexometric titration revels that percent calcium in the *Shauktika bhasma* samples is in the range of 30 to 40 %.This indicates that *Shauktika bhasma* is mainly composed of calcium carbonate. The sample R 3 show 42.66 % of calcium in the sample which indicates that the samples may contain a mixture of calcium carbonate (100%) along with other compounds of Ca such as calcium oxide(0.66 %). The samples with less percentage of calcium carbonate may contain some organic matter which is thermally stable.

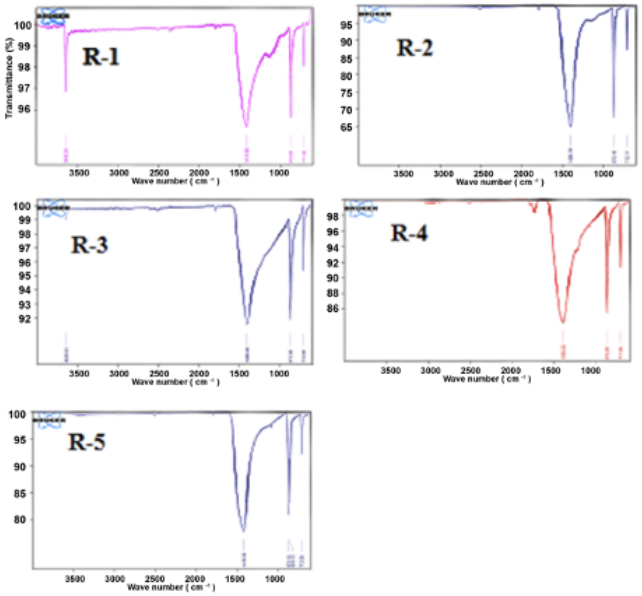
Infrared Spectroscopic studies

The IR spectra of of *Shauktika bhasma* were recorded on IR spectrophotometer of make ThermoScientific (Nicolate iS5) in the mid IR region 4000-450 cm^{-1}.Each sample for recording IR was prepared by mixing 1mg *bhasma* powder with 100 mg KBr and making the transparent pellet under pressure. This sample was then subjected to obtain its IR spectrum. (11). The spectra are presented in Fig-1 and important IR peaks of samples and Calcium carbonate are depicted in the Table-2.

Table 2: Infrared peaks of *Shauktikaa Bhasma* Samples

Sr. no	Name of the sample	Peaks of CO_3^{-2}(cm^{-1})
1	R1 (Bhasma-1)	1410, 873, 711
2	R2 (Bhasma-2)	1406, 873,712
3	R3 (Bhasma-3)	1408, 872, 710
4	R4 (Bhasma-4)	1392, 872, 711
5	R5 ($CaCO_3$)	1418, 872, 854, 712

Figure 1: IR Spectra of *Shauktika Bhasma*



The peak of carbonate (CO_3^{-2}) in pure $CaCO_3$ is observed at 1418 cm^{-1}(12).All sample of *bhasma* show characteristic peaks around (1392 – 1415 cm^{-1}) which are assignable to (CO_3^{-2}) from $CaCO_3$.The peaks around 872 cm^{-1} are in plane bending mode of calcite. The peaks in the range (710- 712 cm^{-1}) are due to out of plane binding vibrations of (CO_3^{-2}) from $CaCO_3$ specifically for the calcite phase(13) .Therefore IR spectra confirms that the main constituent of *Shautik bhasma* is calcite phase of $CaCO_3$. No peaks of calcium hydroxide were observed for the samples under study. Therefore IR spectra confirms that the main constituent of *Shautik bhasma* is $CaCO_3$.

UV Spectroscopic studies

The electronic spectra of *Shauktika bhasma*, were recorded on Shimadzu UV double spectrophotometer in the uv-visible region (200-800 nm). The sample was

prepared by mixing 1.0 mg *bhasma* powder with 100 mg KBr and making the transparent pellet of 0.1 mm thickness under pressure and it is subjected to obtain its electronic spectrum.(14).In the spectra of *bhasma* weak peaks are observed at 400.0, 345.0 304.0, 213.0 nm which specify the possibility of presence of organic matter in the bhasma sample (15). It might have introduced in the sample due to use of plant juices during the preparation of *bhasma* sample.

Powder XRD studies

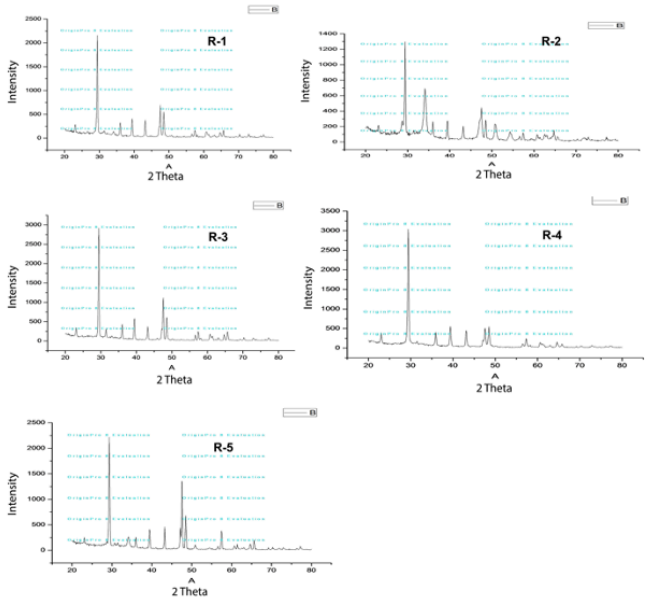
X ray spectroscopic studies are very useful for phase determination of calcium carbonate based drugs. The XRD patterns of *Shauktika bhasma* were recorded on Bruker D8 Diffrractometer. The compound was mortared to make fine powder and 10-20 mg sample was used to record the pattern in range 2θ = 5 to 60 0. The source used was Cu K alpha X rays with wavelength of 1.540 A^0 The X ray patterns are presented in Fig-2. The Full Length at Half Maxima (FWHM) was measured for the peaks at the 2θ angles with maximum intensity which are characteristic peaks of calcium carbonate. The measurement of FWHM and crystallite size was done using software available with the XRD instrument. The correlation between FWHM and the Crystallite size of the sample is given by Scherrer equation (16-17):

$$\text{Crystallite Size} = K\lambda / \beta\cos\theta$$

Here K is the Scherrer constant, λ is wave length of the X-ray beam used (1.54,184 Å), β is the Full width at half maximum (FWHM) of the peak for θ which is the Bragg angle.

The results are summarized in Table- 3.

Figure 2: XRD Patterns of *Shauktika Bhasma*



The Sharp nature of peaks indicates that all samples are crystalline in nature.The diffraction peak at (2θ= 29.40°) in all samples of *bhasma* support the 'Calcite phase of the $CaCO_3$ in the sample(18).

Table 3:The 2θ values and Crystallite Size from XRD of the *Shauktika Bhasma* samples

Sr. No	Sample Name	FWHM	Angle(2θ)	Crystallite Size (nm)
1	R1	0.2268	29.40	36.22
2	R2	0.2681	29.39	30.64
3	R3	0.2711	29.32	30.30
4	R4	0.2546	29.42	32.31
5	R5	0.2793	29.42	29.41

The starting material of *Shauktika bhasma* is *Shauktika* powder which an aragonite form of $CaCO_3$. In the process of *bhasmikarana*, $CaCO_3$ (aragonite phase) is converted into $CaCO_3$(calcite phase) as given in the following reactions.

$CaCO_3$ (Aragonite) Heating→ $CaO + CO_2 \uparrow$ …… (1)

$CaO + H_2O \rightarrow Ca(OH)_2 + CO_2 \downarrow \rightarrow CaCO_3 \downarrow$ (Calcite)----- (2)

The reaction 1 and 2 are taking place in consecutively in closed earthen pot giving final product as calcite form of calcium carbonate. In this process the impurities are eliminated and pure form of calcium carbonate is formed.

The crystallite size calculated from XRD patterns falls in the range (31 to 32 nm) confirms the nanometric nature of the sample. The crystallite size of all *bhasma* samples is greater than synthetic $CaCO_3$. The order of crystallite size of the *bhasma* sample is as follows. R3 < R2 < R4 < R1.

Determination of Antacid Activity of *Shauktika Bhasma*

The acid neutralizing capacity of a compound is, the amount of hydrochloric acid (HCl) that it can neutralize. This capacity can be determined by a technique called back-titration (19).A known amount of *Shauktika bhasma* is dissolved in an excess of HCl, and then the excess acid is back-titrated with standardized sodium hydroxide (NaOH) solution. For determination of antacid activity of each *bhasma,* finely grinded powder of the *bhasma* sample (0.250 gm) was taken in the conical flask. Then 15 ml of standardized 0.5M HCl was added in it and the solution was boiled. Phenolphthalein indicator was used for detecting the colour change at the end point. The solution in the flask was titrated against standard 0.5M NaOH till the end point of the titration is obtained. The constant burette reading is recorded for each of the *bhasma* sample which is used to calculate the antacid activity i.e. Moles of acid neutralized (Table-4). Formula used to calculate the activity is given in Eq.(1).

Moles of acid neutralized = (moles of HCl added) – (moles of NaOH required for back-titration) = (M_{HCl} X V_{HCl}) – (M_{NaOH} x V_{NaOH})……. Eq(1)

Where M = Molarity and V = volume in liters.

Table 4: Antacid activity of *Shauktika bhasma* (Moles of acid neutralized by *Bhasma* samples)

Sr. No	Name of Sample	Volume of NaOH required to neutralize excess of HCl in back titration	Moles of acid Neutralized (Antacid Activity)
1	R1 (Bhasma)	7.7 ml	3.65 moles
2	R2 (Bhasma)	5.5 ml	4.75 moles
3	R3 (Bhasma)	5.2 ml	4.90 moles
4	R4 (Bhasma)	6.7 ml	4.15 moles
5	R5 ($CaCO_3$)	7.8 ml	3.60 moles

Following reactions take place when, the *bhasma* neutralizes the acid.

$$CaCO_3\ (Bhasma) + 2HCl(acid) \rightarrow CaCl_2 + CO_2(g) + H_2O(l)$$

The excess acid remaining after above reaction, is back titrated by NaOH and is neutralized at the end point.

(Excess HCl) + (NaOH) → Neutral solution (end Point)

Antacid activity of all *bhasma* sample under study is comparable with synthetic $CaCO_3$. Among all *bhasma* samples antacid activity of sample R3 is highest and hence this sample is best antacid than other samples. The antacid activity is observed in the order of R3 > R2 > R4 >R1, which is inversely proportional to the crystallite size of the *bhasmas*. As the size of particle decreases, the surface area increases and it also promotes dissolution of calcium carbonate in the solution. This increases the percent carbonate ions in the solution and neutralization process is improved which results in neutralization of larger numbers of moles of acid. The time of neutralization is also decreased in this process.

The acid in the stomach is in the form of hydrochloric acid (HCl). Therefore present work of antacid activity could be useful for a quick preliminary study of acid neutralization capacity of *Shauktika bhasma* which is based on a simple acid- base back titration method.

Conclusions

The current status of four samples of *Shauktika Bhasma* is studied with respect to their chemical and structural properties using complexometric titrations and modern analytical techniques.Their comparative antacid activity is also studied. The results obtained in this work lead to following conclusions:

The main constituent of *Shautika bhasma,* obtained from four reputed pharmacies is $CaCO_3$ (Calcite Phase).The percentage of calcium in all samples of *Shautika bhasma* is in the range of 34-42%. The more or less percentage of calcium could be due to presence of small percentage of calcium oxide or some organic matter remained during *bhasamikarana* process.

Crystallite size of *bhasma* confirms nanometric nature of all *bhasma* samples which is greater than synthetic $CaCO_3$. The antacid capacity of all *bhasma* samples were comparable with synthetic $CaCO_3$ in spite of their higher crystallite size. In general antacid activity of all the *bhasma* samples under study, is seen to be inversely proportional to crystallite size of the samples calculated from XRD studies. Therefore it may be concluded that chemical, structural and antacid properties of all the samples of *Shauktika bhasma* prepared by different selected pharmacies are comparable with each other with small variations.

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