https://doi.org/10.47552/ijam.v16i2.5727 ISSN No: 0976-5921

International Journal of Ayurvedic Medicine, Vol 16 (2), 2025; 411-415

IR, XRD and Antacid studies of some commercial samples of Eggshell based Ayurvedic medicine: *Kukkutandtwakbhasama*

Research Article

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Abstract

Kukkutandtwak bhasma (KB) is a chicken eggshell based Ayurvedic medicine which is well known for its use as calcium supplement, treatment on leucorrhea, anemia, diarrhea and stomach disorders. In the present communication IR and XRD spectra of some commercial samples of KB are studied to find out their major constituents. Comparative study of four samples under study confirms that the main constituents of KB is a mixture of Calcium carbonate and calcium hydroxide or only calcium carbonate. Characteristic peaks around 1406-1453cm^{-1}in IR spectra of all samples indicates presence of calcium carbonate,The peaks at 2θ=29.42 in XRD patterns support this observation. Among the four samples, three samples exhibit broad peaks at 3640 cm^{-1}indicative of presence of calcium hydroxide in addition to calcium carbonate. XRD peaks at 2θ=34 confirms the presence of calcium hydroxide in three samples.Calcium carbonate based antacids are known for their rapid acid neutralization action.Since the main constituent of KB is calcium carbonate, the antacid capacity of the KB samples are studied. The antacid capacity is determined using acid-base back titration method, expressed in terms of, number of moles neutralized by the *bhasma* samples.The number of moles neutralized by KB samples; containing a mixture of $CaCO_3$+$Ca(OH)_2$ are greater than the sample which contains only $CaCO_3$. Estimation of antacid capacity using simple acid-base back titration leads to a quick idea of antacid capacity of various commercial samples and is useful to develop the *Kukkutandtwak bhasma* as an effective antacid agent of natural origin.

Keywords: Eggshell, *Kukkutandtwak bhasma,* XRD, IR, Antacid capacity.

Introduction

Kukkutandtwak bhasma (KB) which is prepared from chicken eggshell is used for the treatment of white discharge, anemia, urinary tract infection and weakness(1-2).It is also used to treat diarrhea, vomiting, nausea and eye disorders(3).Apart from this there is an equally important property of this traditional drug which has been investigated in the present communication is its acid neutralization capacity.

The raw material used for preparation of KB is chicken eggshell which is available in large amount and is normally disposed as a waste material. KB is composed of calcium compounds and is prepared by following standard procedures mentioned in standard Ayurvedic texts(3-4). According to *Ayurveda*, when eggshell powder is subjected to the *bhasma* preparation process (*bhasmikarana*) process, it is transformed into medicinal formulation in which unnecessary or harmful matter to human health is eliminated and the process imparts therapeutic value to it. Analytical studies on the KB are reported previously by few researchers (5-7). Many pharmacies are preparing it on commercial base. The main aim of the present study is to perform a comparative study of IR and XRD patterns of commercial samples of KB, collected from reputed pharmacies and to explore their antacid capacity using acid-base titration method. Identification of different constituents of Ayurvedic formulations and its structure-activity correlation is still a difficult task as different methods are followed by different pharmacies to prepare the *bhasma*.This paper will facilitate the identification of main constituents of KB using IR and XRD analysis which is useful for the standardization of this traditional medicine for its more effective use. In addition to this, antacid activities of the samples are also reported which are correlated with their main constituents established from XRD and IR studies. As the KB is prepared from eggshells which have a natural occurrence, its use as antacid agent will be safer than the synthetically synthesized antacids.

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Materials and methods

Commercial samples of KB are purchased from four reputed pharmacies and are titled as KB1, KB2, KB3 and KB4. The samples are compared with AR grade calcium carbonate purchased from Aldrich. The phase determination is done by XRD peaks recorded for 2θ angle in the range of 20 to 80^0. The XRD patterns of KB are recorded using Bruker model of X ray diffractometer. The crystallite size of the samples is

obtained using automatic software available in the model. The Infrared spectra of KB and $CaCO^3$ are recorded on IR spectrometer of Bruker in the FT IR range, between 4000-450 cm^{-1}. Antacid Capacity of *Kukkutandtwak bhasama* (KB) is measured by using standard procedure of back titration(16).The antacid capacity of a substance is its power to neutralize the acid and is determined by calculating "number of moles" of an acid, neutralized by it. The weighed amount of KB is dissolved in an excess of HCl where the neutralization of specific amount of acid is completed by the *Bhasma* and then the HCl remained in the solution is titrated against the standard NaOH. The method is commonly known as back titration method. In the present study finely powdered KB sample is dissolved in 15 ml of 0.5M HCl in a conical flask and the mixture was heated till the complete dissolution of the *bhasma*.Following reactions take place in the solution:

For the sample K2 :

$$CaCO_3+2HCl \rightarrow CaCI_2+H_2O+CO_2 \quad \text{---- Eq (1)}$$

For samples K1 K3 and K4 :

$$CaCO_3+Ca(OH)_2+2HCl \rightarrow 2CaCl_2+H_2O+CO_2 \text{ -- Eq (2)}$$

Further the acid which remains after completion of above reaction is back titrated with 0.5 M NaOH till its neutralization. The volume required for acid neutralization is noted by colour change of the solution at the end point using phenolpthaline indicator. The constant burette readings (volume in ml) are substituted in the Eq(3) to obtain number of moles of acid neutralized by *bhasma* samples.

The antacid capacity is determined as follows

Moles of acid neutralized by *Bhasma* sample = (moles of HCl added in the bhasma sample) – (moles of NaOH required for neutralization of excess acid in back-titration)

= (M HCl X V HCl) – (M NaOH x V NaOH) -- Eq(3)

where M is molarity of HCl or NaOH and V is volume of HCl or NaOH.

Results and Discussion

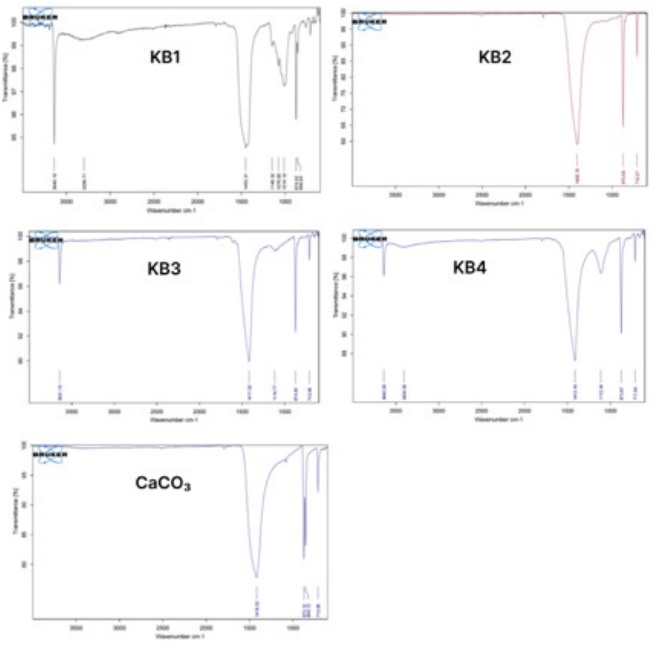
Infrared Spectral Studies of *Kukkutandtwak bhasma* (KB):

The FT IR spectra of (KB) samples are presented in Fig-1 and important frequencies are depicted in Table-1

Table 1: IR Peaks of *Kukkutandtwak Bhasama* (KB)

Sr. no	Peak-1	Peak-2	Peak-3	Peak-4	Peak-5	Peak-6
1	KB1	3640.10	1453.31	1076.60 1014-1049 b peaks	874.02	-
2	KB2	-	1406.16	--	873.58	712.27
3	KB3	3641	1417.38	1119	874.45	712.08
4	KB4	3640.28	1413.16	1112.38	873.87	711.97
5	$CaCO_3$	-	1418.32	--	872.12 854.13	712.06

Figure 1: FT IR Spectra of *Kukkutandtwak bhasama*



The pure $CaCO_3$ is used as standard to compare the IR spectra of the *bhasmas*. It should be noted that in IR spectra of pure $CaCO_3$, peaks around 3640$cm^{-1}$are absent. On the contrary, in KB1, KB2 and KB4 samples, very sharp peaks are observed around 3640 to 3641cm^{-1}which can be assigned to OH stretching frequency of $Ca(OH)_2$(8-9). In the spectra of KB2 ,this frequency is absent indicating absence of $Ca(OH)_2$.The second peak is observed in the range of 1406-1453 cm^{-1} in all samples of *bhasmas* is typical peak of carbonate in calcium carbonate (10-11). Except that of KB2, all samples show third and fourth peaks in range of 1014 to 1118 cm^{-1}(10). These peaks can be assigned to vibration modes of C-O from calcium carbonate. The absorption peaks 5 and 6 around 854-874 cm^{-1} and 711-712 $cm^{-1}$are attributable to in plane bending and out of plane bending vibrations of calcium carbonate (12-13).Therefore IR spectral studies specify that all the samples of KB are a mixture of $CaCO_3$ and $Ca(OH)_2$ except that of KB2 sample. The KB2 sample contains only $CaCO_3$ as the major constituent.

XRD Study of *Kukkutandtwak Bhasama* (KB)

The XRD patterns of KB are presented in Fig-2.The significant 2θ values, full length at half maxima (FWHM), Crystallite size and identified phases of the KB samples are depicted in Table 2.

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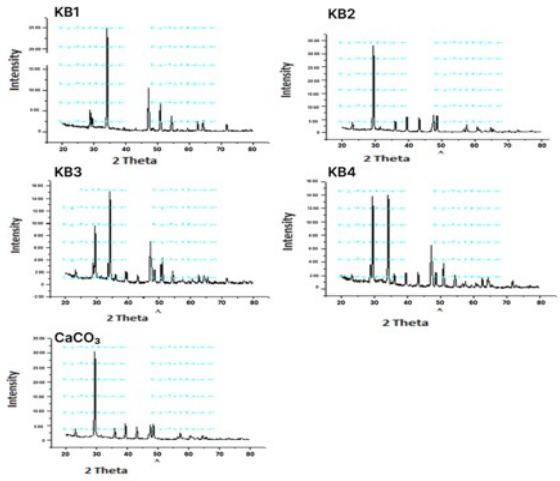
Table 2: XRD Parameters of *Kukkutandtwak Bhasama* (KB)

Sr. No.	Samples	Significant 2θ Values	2θ peak at highest Intensity	FWHM	Crystallite Size (nm)	Constituents of KB
1	KB1	29, 34.13,47, 52, 54, 63,65,72	34.13	0.2414	34.43	$CaCO_3$+**$Ca(OH)_2$**
2	KB2	22, 29, 36, 43,48, 49,57, 29.41(Calcite)	29.41	0.2554	32.17	$CaCO_3$
3	KB3	29, 34, 34.12, 36,38, 44, 47, 48, 52,54	34.12	0.2691	30.89	$CaCO_3$+**$Ca(OH)_2$**
4	KB4	29, 34, 37, 38, 47,48, 52, 54, 64,65,34.12	34	0.2797	29.72	$CaCO_3$+**$Ca(OH)_2$**
5	$CaCO_3$	29.42, 36, 43,48, 49,57, 29.41 (Calcite)	29.42	0.2793	29.41	$CaCO_3$

Figure 2: XRD Patterns of *Kukkutandtwak Bhasama* (KB)



KB1, KB3 KB4 show sharp peaks at 2θ=29.42 which are due to calcite phase of $CaCO_3$[10].They show additional sharp peaks at 2θ =34 with highest intensity. This value of 2θ is a characteristic value of $Ca(OH)_2$ (14,15).The XRD patterns of $CaCO_3$ and K2 samples are very similar. No peak for $Ca(OH)_2$ at 2θ =34 is observed in K2 sample. Therefore XRD studies revealed that K1, K3 and K4 samples contain a mixture of ($CaCO_3$ + $Ca(OH)_2$) while K2 contains only $CaCO_3$ as its major constituent. There are no significant peaks at 2θ =32, 37 and 44 showing that these samples do not contain any CaO(11) in them as a byproduct of the *bhasma* sample which could be formed during calcination cycles done during synthesis of the *bhasma* samples.

Table 3: Antacid Capacity of *Kukkutandtwak bhasama* (KB)

Sr No.	Bhasma Sample	Main Constituents	Crystallite Size (nm)	Constant burette reading (ml)	No of Moles Neutralized
1	KB1	$CaCO_3$+**$Ca(OH)_2$**	34.43	7.0 ml	4.0
2	KB2	$CaCO_3$	32.17	7.8 ml	3.6
3	KB3	$CaCO_3$+**$Ca(OH)_2$**	30.89	5.4 ml	4.8
4	KB4	$CaCO_3$+**$Ca(OH)_2$**	29.72	6.4 ml	4.3

The order of antacid activity of samples in terms of "Number of moles neutralized" is depicted in Table-3.The order of activity, under study is KB3(4.8)>KB4(4.3)>KB1(4.0)>KB2(3.6) as the number of moles neutralized by *bhasma* samples is in the same order as given in the parentheses. (Table-3). The samples K1, K3 and K4 having composition of mixture of calcium carbonate and calcium hydroxide show more antacid activity than sample K2 having major constituent only calcium carbonate.

Discussion

The IR and XRD study confirms that major constituents of *Kukkutandtwak Bhasama* (KB) are $CaCO_3$ and $CaOH_2$. KB is prepared by treating eggshell powder with certain medicinal plant juices and by heating it in close earthen pots. In a close system, eggshell powder which is chemically $CaCO_3$ (Aragonite) undergoes, following simultaneous reactions; Eq 4 and 5. (16).

$$CaCO3\,(Aragonite)\ Heating \rightarrow CaO + CO2\uparrow \ldots \text{Eq } 4$$

$$CaO + H2O \rightarrow Ca(OH)2 + CO2\downarrow \rightarrow CaCO3\downarrow (Calcite) \ldots \text{Eq } 5$$

Therefore when eggshell powder is subjected to *bhasmikarana* process, possible end products are CaO, $CaCO_3$ (Calcite) and $Ca(OH)_2$.The present study facilitates identification, presence or absence of these compounds from IR peaks and 2 theta values of XRD patterns.Both the spectral techniques confirm the presence of $CaCO_3$ and or $Ca(OH)_2$ and absence of CaO in the KB samples. Exact detection of constituents is supportive for standardization of *Kukkutandtwak Bhasama* on scientific basis.The acid neutralization capacity of the selected bhasmas (in terms of number of moles neutralized by bhasma samples) are in the range of 3.6 to 4.8 moles. This capacity can be correlated with constituents of the KB *bhasma*.

As $Ca(OH)_2$ is highly alkaline than $CaCO_3$, its presence in the mixture of calcium carbonate and calcium hydroxide imparts grater acid neutralizing capacity to KB, resulting more salt formation i.e $2CaCl_2$ (Eq-2). The exact ratio of $CaCO_3$ to $Ca(OH)_2$ in the

samples could not be found from the present investigations. But from (Table-3), it may predicted that sample K3 showing highest antacid activity (4.8) could have more amount of $Ca(OH)_2$ in the mixture than in the K4 (4.3) and K1 (4.0). This observation needs to be confirmed from pH metric studies of the *bhasma* solutions during neutralization.

Recent 'in-vitro' studies on antacid agents, show that calcium carbonate-based antacids are extensively used due to their capability of providing rapid relief by direct neutralization of excess gastric acid (HCl) in the stomach. $CaCO_3$ reacts with hydrochloric acid to form calcium chloride, water, and carbon dioxide. This leads to immediate increase in gastric pH (17). Studies on another antacid drug composed of $Al/Mg(OH)_2$ indicates that presence of magnesium hydroxide (chemically similar to $CaOH_2$) imparts efficient neutralization action on excess acid to reduce heartburn(18). *Kukkutandatwak bhasma* contains calcium carbonate and calcium hydroxide of "natural origin" and hence is more assimilative by human body as an antacid. In the current investigations its chemical action is studied against HCl using simple acid-base back titration method which is useful to check antacid capacity of any KB sample quickly, before their 'in vitro' investigations.

Conclusion

The IR and XRD investigations of commercial samples of *bhasma* are very useful to find out the major constituents of the *bhasma* sample.The commercial samples of KB are mainly composed of a mixture of ($CaCO_3$ + $Ca(OH)_2$) or of only $CaCO_3$ which can be identified from their IR peak positions and characteristic 2θ values in XRD. The acid neutralization capacity of *bhasma* samples is calculated in terms number of moles neutralized. The *bhasma* samples which are composed of mixture of ($CaCO_3$ + $Ca(OH)_2$) show greater antacid capacity(4.0- 4.8) than the sample which is composed of merely $CaCO_3$ (3.6). The study could be useful to develop the *Kukkutandtwak bhasma* (KB) as an antacid drug of natural origin.

Acknowledgement

The authors are thankful to In charge Principal Prof. (Dr) V. A. Rankhambe and Prof (Dr) A. B. Pawar Head Department of Chemistry, Y.M.College,Bharati Vidyapeeth (Deemed to be University) Pune for providing necessary facilities.

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