INVESTIGATION OF TEMPERATURE PROFILES OF TRADITIONAL PUTA AND PREPARATION OF CHALCOPYRITE ASH FOR AYURVEDIC TREATMENT USING MUFFLE FURNACE

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Degree of Master of Science

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Thesis submitted in partial fulfillment of the requirements for the degree Master of Science in Sustainable Process Development

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DECLARATION

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Dr.(Mrs.) S.H.P.Gunawardena		Dr.(Mrs.) S.K.M.K.Herapathdeniya	
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Abstract

Bhasmas (ashes) are complex compound forms of metals, minerals or gemstones obtained after a series of ayurvedic pharmaceutical preparation processes; purification (*shodhana*), trituration (*bhavana*) and incineration (*marana*) in combination with various herbal extracts and other substances. These three processes play an important role in preparation of ashes. *Puta* is the specific quantum of heat required to get the desired quality ashes in the process of incineration (*marana*) and naturally available fuels like cow dung cakes are used to produce heat in the conventional *puta*. The amount of heat required to produce a specific ash is substance specific and described in terms of fuel burnt. The objectives of this research are to establish temperature profiles for *Maha Puta, Gaja Puta* and *Varaha Puta* and to verify temperature profile of *Varaha puta* using a muffle furnace. Temperature profiles were established using dried cow dung cakes with an average calorific value of 15.44 MJ/kg as the fuel and the maximum temperatures achieved for *Maha Puta, Gaja Puta* and *Varaha Puta* were 1380 ^{0}C, 1060 ^{0}C and 850 ^{0}C respectively. Then temperature profile of traditional *Varaha Puta* was matched with a muffle furnace and Chalcopyrite ash (*Swarna Makshika bhasma*) was prepared using both traditional method and electric muffle furnace. The ashes produced using both methods showed similar properties and hence the temperature profile obtained for traditional *Varaha Puta* using the muffle furnace was verified.

Key words : *Puta*, ash (*bhasma*), Chalcopyrite

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

1 INTRODUCTION

1.1. Background

In Ayurveda system of medicine, minerals, metals and gemstones are used to prepare drugs combining with various herbs for curing diseases. Converting metals and minerals into acceptable form for oral administration is done by following series of pharmaceutical processing methods. Metals and minerals processed with different herbal materials are converted into special form known as *bhasma* (ash) and these herbo-mineral preparations come under a special branch of Ayurveda called *Rasa Shastra*. It deals with the drugs of mineral origin, their varieties, characteristics, processing techniques, properties and their therapeutic uses.

In the process of preparation, metal or mineral is converted into a special form known as *bhasma* (ash form) and preparation process involves purification (*shodhana*), trituration (*bhavana*) and incineration (*marana*). Purification is a process through which the external and internal impurities of metals / minerals are removed. It involves elimination of harmful matter from the metal or mineral, modification and conversion of undesirable physical properties of the metal / mineral to desirable form and enhancement of the therapeutic action (Pal, Sahu, & Haldar, 2014). The second process, trituration is a process of wet grinding in which materials are ground with specific liquid media for a particular period and this process leads to unique and suitable physico-chemical changes and potentiate the efficacy of material. The final process, incineration is the process in which purified metals or minerals are made in to pellet form with herbal extracts and required other ingredients as prescribed in classical texts and then is subjected to fire treatment in closed earthen crucibles in a pit by burning dried cow dung cakes to obtain ashes of metal / mineral. This process is repeated as many times as prescribed in classical texts for each preparation. The quantum of heat required to produce specific ash is known as *Puta*. Amount of heat needed to produce specific ash it is described in terms of cow dung cakes to be burnt and temperature profiles are not defined for traditional *Puta*. The current favour is to produce ashes using alternative heating method and therefore identifying temperature profiles for different *Puta* systems is very important.

1.2. Objectives

- Establish the temperature profiles of three traditional *puta* systems; *Varaha Puta*, *Gaja Puta* and *Maha Puta*
- Verify the temperature profile of *Varaha Puta* by comparing the properties of Chalcopyrite ash (*Swarna Makshika bhasma*) prepared using traditional *Varaha puta* system and electric muffle furnace

2 LITERATURE REVIEW

2.1. *Bhasma*

Bhasma in *Ayurveda* has been defined as a substance obtained by calcination (incineration). *Bhasmas* are prepared by the process in which the metal or mineral is converted into ash. Metal or mineral are purified to remove impurities and treated by triturating and macerating in herbal extracts. The dough so obtained is incinerated to obtain the ashes.

Bhasma (residue after incineration) form along with appropriate herbs for treatment of critical ailments is a medicinal preparation in Ayurveda. The procedures for preparing these medicines are time-consuming and complicated (Mander & Liu, 2010).

These *bhasmas*, or lighter forms of metals or minerals, are contained in organo-metallic compounds that work as carriers. This means that they are able to carry the herbs mixed with them faster to the desired site and start the action immediately. They act as catalysts and increase the bioavailability of the herbs to the cell.

After performing the desired action, the *bhasmas* are eliminated through our excretory systems, specifically via urine and stool. Ayurvedic chemists have evolved various procedures like sublimation, oven treatment, controlled heat incineration, grinding, mixing, and powdering etc. to inculcate the therapeutic properties in the minerals for which many specific types of instruments are designed (Bose & Baghel, 2014).

2.2. *Puta* system

Puta is the heating arrangement used in the preparation of ashes mainly from metallic / mineral origins. It means the application of specific quantum of heat needed for individual substances in which heat is produced by fuels like cow dung, goat dung or wheat husk in pits of different dimensions and more or less quantum of heat leads to spoiling of medicines (Chaudhary A. , 2010). Further, it enables the product to spread quickly in the micro circulation of the body. *Puta* causes the reduction of impurities and toxic effects (*doshas*) and induction of therapeutic quality and finally it causes the ash of the raw material processed in this heating

arrangement. Therefore, an understanding of the *puta* is very important in preparation of different ashes and greatest care has to be taken for providing particular quantum of heat for a specific time duration for formation of a particular compound which would be therapeutically effective with less or no adverse effect to the human (Devanathan, 2011).

According to the type of metal or mineral to be incinerated, dimensions of the pit, the number of cow dung cakes to be burnt and number of *putas* (heating cycles) to be given are described in Ayurvedic texts (Savrikar & Ravishankar, 2011).

Any kind of *puta* provides three phase of temperature pattern; pattern of ascending temperature, pattern of maximum temperature range and pattern of self-cooling. All these three combiningly produce the desired product (Mohapatra, Gupta, & Jha, 2013). Maximum temperature range (maximum range of temperature plotted for maximum duration of time) and self-cooling play more important role for conversion and compounding of materials.

2.2.1. Classification of *puta*

Different types of *putas* are explained and depending upon the source of energy, *puta* can be categorized as shown in Figure 2.1.

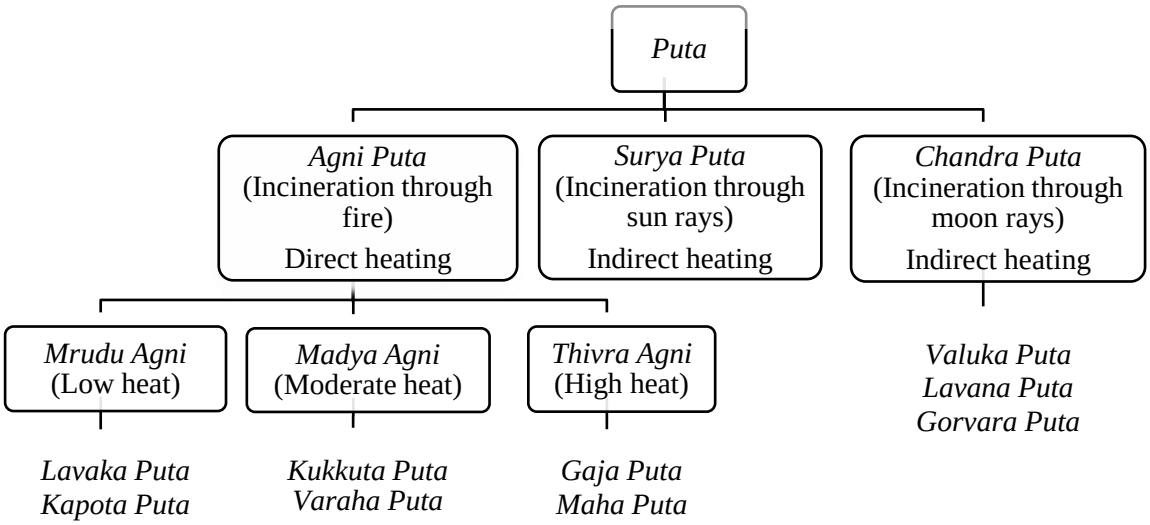

Figure 2.1 : Different types of *puta* (Devanathan, 2011)

According to dimensions of the pit and number of cow dung cakes (*Goma Verati*) used, *Puta* can be classified as *Maha Puta*, *Gaja Puta*, *Varaha Puta*, *Kukkuta Puta* etc. (Vagbhatacharya, 2003) as mentioned in Table 2.1.

Table 2.1 : Classification of *Puta*

Name of *Puta* system	**Dimensions of the pit**		**No. of cow dung cakes to be burnt**	**Metal / mineral incinerated**
	Ayurvedic units	**SI units**		
Maha Puta	*Dohasta pramana (48 Angula*)*	90cm×90cm×90cm (36"×36"×36")	1500	*Hirak* (Diamond) *Abhra* (Mica)
Gaja Puta	*Rajahastha pramana (30 Angula*)*	57cm×57cm×57cm (22.5"×22.5"×22.5")	1000	*Abhra* (Mica) *Paparad* (Kavadi) *Godanthi* (Selenite) Gem (Red,Blue) *Mandura* (metal – Sn) *Hakbeli Katu*
Varaha Puta	1 *aratne*** (22 Angula*)	42cm×42cm×42cm (16.5"×16.5"×16.5") (Jhalakikar, 1890)	150	*Tamra* (Copper) *Swarna Makshika* (Chalcopyrite)
Kukkuta Puta		45 cm diameter (Dole & Prakash, 2010)	50-60 (Dole & Prakash, 2010) 100(Vagbhat acharya, 2003) 10 (Sharanga Dara Samhitha)	*Swarna Bhasma* (Gold)

* 1 *Angula*=0.75 inches (Jhalakikar, 1890)

**1 *aratne*= distance between tip of the little finger and elbow (Jhalakikar, 1890)

1. ***Maha Puta***

   An underground cavity measuring 3'×3'×3' in length, breadth and depth is prepared. About 1000 cow dung cakes are arranged at the bottom of pit, then properly sealed crucible with material to be incinerated is placed and again 500 cow dung cakes are arranged over it covering the crucible from all sides. Then cow dung cakes are ignited and heat is given to the crucible till all the cow dung cakes are burnt and gets cooled on itself. At the beginning the intensity of heat is not intense but as all the cow dung cakes burnt, it reaches to the maximum

temperature and remains steady for a long time (Vagbhatacharya, 2003). *Maha Puta* is used to prepare ashes like Diamond, Mica.

2. ***Gaja Puta***

The structure of *Gaja Puta* is similar to that of *Maha Puta*. The only difference is in its dimensions and the number of cow dung cakes used. *Gaja Puta* is an underground cavity measuring 2'×2'×2' in length, breadth and depth. About 700 cow dung cakes are arranged at the base on which the crucible is kept for heating. Moreover, 300 cow dung cakes are placed on the upper surface of the crucible, covering it on all sides. The cow dung cakes are ignited and the crucible receives heat from all sides (Vagbhatacharya, 2003).

Gaja puta is used to prepare ashes like Mica (*Abhra bhasma*), Selanite etc.

3. Varaha Puta

Varaha Puta is an underground cavity measuring 16.5" ×16.5" ×16.5" in length, breadth and depth (Jhalakikar, 1890). About 100 cow dung cakes are arranged at the bottom then properly sealed crucible is placed and again 50 cow dung cakes are arranged over it covering the crucible from all sides. It offers less intense heat than *Maha Puta* and *Gaja Puta*. *Varaha Puta* is used to prepare chalcopyrite ash (*Swarna Makshika bhasma*), copper ash (*Tamra bhasma*) etc (Vagbhatacharya, 2003).

4. Kukkuta Puta

The cow dung cakes are placed on a circular area of about 18" in diameter. The crucible containing drug substance is properly sealed and kept on the surface of cow dung cakes and they are ignited. It requires only 50-60 cow dung cakes (Dole & Paranjpe, 2006) and reaches the temperature between 200 ^{0}C to 250 ^{0}C (Vagbhatacharya, 2003).

Kukkuta Puta is used to incinerate soft drug substances.

5. ***Kapota puta***

The fuel used for this type is only 8 cow dung cakes. They are placed either underground or on the surface of the earth. It creates heat of very low intensity. It is used for the drug substances which are very soft. This is used to prepare *bhasma* of Mercury (Vagbhatacharya, 2003).

6. Gobara Puta

Whenever the fuel used is of the bits and fragments of cow dung collected from a cow-pen, the *puta* is known as *Gobara puta*. Sometimes, the husks of the grains are also used for the fuel purpose.

The crucible containing a drug substance is kept in the middle of an iron pot and the bits and fragments of cow dung are placed surrounding it and pressed around the crucible. The fuel ignited burns slowly and creates less intense heat (Vagbhatacharya, 2003).

2.3. Ash of Chalcopyrite (*Swarna Makshika Bhasma*)

2.3.1. Chalcopyrite

Chalcopyrite ash *(Swarna Makshika Bhasma)* is one of the most famous mineral in therapeutic practices in Ayurveda. It is widely used in the treatment of anemia, obesity, diabetes, skin disorders and jaundice (Lagad et al., 2011).

Chalcopyrite is an important source of pure copper and also it is known as Copper Pyrite. Chemical formula of Chalcopyrite is $CuFeS_2$ and it contains Copper 34.5 %, Iron 30.5 % and Sulphur 35 % (Minerals-n-more.com). Chalcopyrite is brittle and can be easily powdered (webmineral.com). It is brassy yellow in colour and the cut surface has golden yellow dots or lines. In many cases, it develops a deep blue, green or black tarnish (Minerals-n-more.com). Chalcopyrite looks like gold but is slightly harder, more brittle and less resistant to chemicals. When Chalcopyrite is rubbed, hands get blackish stains. It burns on fire with white fumes and emits smell like Sulphur (Devanathan, 2011).

Structure and Physical Properties of Chalcopyrite (Minerals-n-more.com)

- Colour–Brassy yellow
- Luster – metallic
- Transparency – crystals are opaque
- Crystal Structure – Tetragonal bar 42m
- Hardness – 3.5-4
- Specific gravity – 4.2
- Bonding – covalent
- Melting point – 880^0C

2.3.2. Preparation processes of ash of chalcopyrite

1. Purification of Chalcopyrite

Various methods are described in literature for purification of Chalcopyrite and four different purification methods proposed by different researchers are:

(i) Purification by roasting it in castor oil i.e. Eranda (Ricinuscommunis) taila or juice of Matulung (Citrus medica) or juice of rhizome of Kadali (Musa sapiens) for 48 minutes. Lemon juice can also be used instead of Matulung (Vagbhatacharya, 2003).

(ii) Purification by heating on intense fire and then dipping it into decoction of *Thripala*. It can also be purified by steaming it in the juice of rhizome of *Kadali* with the help of *Dola Yantra* (Dole & Paranjpe, 2006).

(iii) Chalcopyrite with more copper, is purified by rubbing chalcopyrite powder 3 parts with 1 part of Rock salt (saindhava) and then mixed with Citrus medica juice and Citrus limonum juice. This mixture is then heated till the container becomes red hot. Then cooling on its own, the mixture is washed with water number of times and dried in the sun. The substances like sour juices and rock salt produce hydrochloric acid, which acts on the copper contents of chalcopyrite and forms copper chloride, which is soluble in water and gets washed out during washing process. (Dole & Paranjpe, 2006).

(iv) Chalcopyrite powder is mixed with equal amount of castor oil and heated in an iron vessel. Sulphur fumes that escape catches fire and burn out. Intention

of this is to reduce the copper element and eliminate the Sulphur from Chalcopyrite during the purification (Dole & Paranjpe, 2006).

2. Purification of Sulphur

Sulphur is a crystalline, nonmetal used in ayurvedic preparations as a major ingredient. Traditionally, Sulphur is known with other names like *Gandagam, Kaarizhai Natham, Parainatham, Parai Veerayam* etc. Different methods are described in literature for purification of Sulphur.

(i) A wide- mouthed earthen vessel is partly filled with ghee mixed with cow milk and leaving some empty space in the pot above the milk. The mouth of the vessel is covered with a piece of cloth, tighten and then Sulphur is sprinkled over the cloth. The vessel is closed with another deep earthen vessel and the joints are sealed with clay and mud. The lower pot is kept in a ground pit. Then heat (*mrudu agni puta*) is supplied by burning cow dung cakes kept on the covered earthen lid. After it cools, the vessel is opened and it is observed that melted Sulphur drips into the milk. Then the product is collected, washed with hot water and the process is repeated three times using the resultant Sulphur. Purified Sulphur thus prepared is ready to be used in medicinal preparations (Vagbhatacharya, 2003)

(ii)Any one of *Jyotishmati* (Celastrus PaniculatusWilld.) oil, *Sarshapa* (mustard) (Brassicanigra) oil, Eranda / Castor oil (Ricinus Cummunis L.), *Kusumbha* (Schleicheaoleosa Lour) oil, milk of ewe, cow ghee, cow milk or *Aranalam* (sour gruel made of fermentation of rice water) is poured into an earthen pot up to the $3/4^{th}$ level. The mouth is covered tightly with a cloth and then Sulphur is sprinkled on the cloth and it is closed with an iron lid. The joints are sealed with clay and mud. The pot is kept in a ground pit and it is covered with an earthen lid. Then it is heated with *mruduagni puta* by placing cow dung cakes on it. Then the pot is opened once it is cooled. It can be observed that the product has melted and mixed / resolved into the liquid poured in to the lower pot. The product is collected and it is subjected to *Bhavana* with

juice of *Dhattura* and the whole process is repeated to get purified *Gandhaka* (Sharma, 1979).

(iii) The juice of *Brihati* (Solanumindicum Linn.), juice of *Ajamoda*, juice of *Bhringaraja*, juice of *Dhattura* or juice of *Tilaparni* is taken and an equal quantity of Sulphur is added and it is subjected to *Mardana* separately with each juice for three hours. The product is colleded with equal quantity of ghee into an iron vessel and it is melted. Next the collected product is dripped with goat milk. Then the whole process is repeated for seven times to get purified Sulphur (Sharma, 1979).

3. Incineration of Chalcopyrite

Purified Chalcopyrite is rubbed with sesame oil or shegoat's urine or decoction of *Kulattha* (Doichosbiflorus) or buttermilk and heated, thus incineration of Chalcopyrite gets ready (Shridatta, 1983).

Vagbhatacharya (2003) mixed the powdered purified Chalcopyrite with equal amount of purified sulphur and processed with the juice of *Jambira* (Citrus limonum). Then sealed in a crucible and subjected to *Varaha Putas* for five times. Vagbhatacharya (2003) also mentioned that Chalcopyrite can be incinerated with castor oil or cow's ghee or the juice of Citrus medica.

The incinerated chalcopyrite ash prepared by above three procedures shows variations in its colour like brick red, haematite coloured and blackish. Apart from the procedures, the colour of ashes depends upon the copper content of initial chalcopyrite (Dole & Prakash, 2010).

2.4. Previous Studies

Parmar et al (2010) obtained temperature profiles of *gaja puta* and *ardhagaja puta* by burning cow dung cakes in preparation of *Vanga bhasma.* Number of cow dung cakes used for *gaja puta* and *ardhagaja puta* are 94-110 and 45-65 respectively (Parmar et al., 2010). Average temperature profiles obtained for *gaja puta* and *ardhagaja puta* are shown in Figure 2.2 and Figure 2.3 respectively.

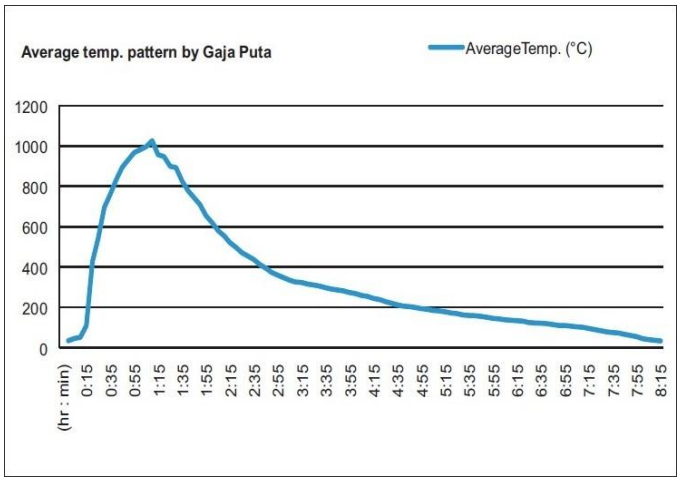

Figure 2.2 : Average temperature profile of *gaja puta* (Parmer et al, 2010)

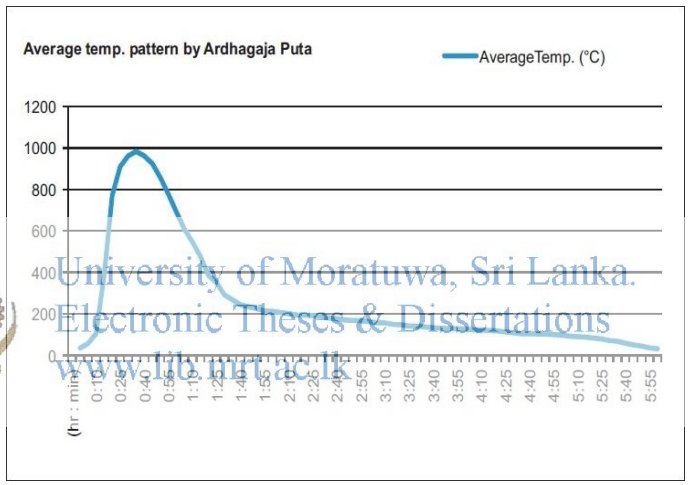

Figure 2.3 : Average temperature profile of *ardhagaja puta* (Parmer et al, 2010)

Singh and Chaudhary (2013) obtained temperature profile for incineration of Chalcopyrite ash by burning 6 kg of cow dung cake for each *puta*. However, the type of *puta* and number of cow dung cakes used for the incineration is not mentioned. The maximum temperature attained was 850 – 880 ^{0}C and total duration of *puta* was approximately 5 ½ hours. It required 16 *putas* (6 kg cow dung cakes for each *puta*) for complete conversion of 100 g raw Chalcopyrite to ash form (Singh & Chaudhary, 2013). Temperature profile obtained by burning 6 kg of cow dung cakes is shown in Figure 2.4.

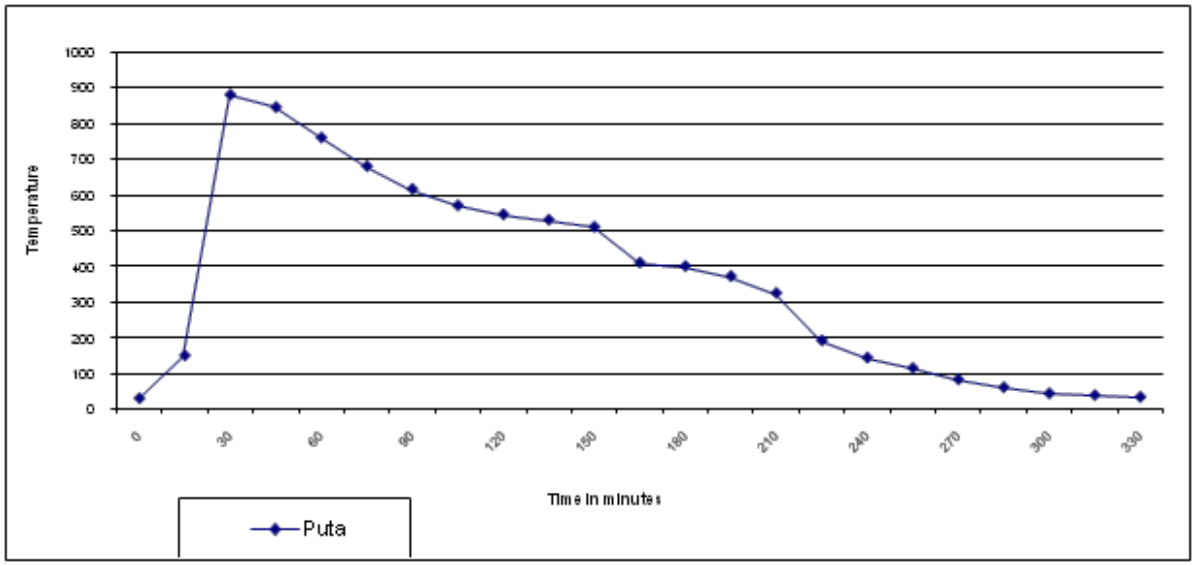

Figure 2.4 : Temperature profile obtained by burning 6 kg of cow dung cakes (Singh & Chaudhary, 2013)

Temperature pattern was obtained by Devanathan (2013) using 35 kg of cow dung cakes for incineration in each *puta* and the procedure followed by him is mentioned. In their work, Chalcopyrite powder has been ground with Aloevera juice in an end runner, till it attained semisolid paste like consistency. Then small round pellets have been made and dried. After drying, these pellets have been kept in earthen plate and sealed with the help of cloth smeared with clay. This has been subjected to *Varaha puta*. The same process has been repeated for 29 times to obtained Chalcopyrite ash with desired quality (Devanathan, 2013). Temperature pattern obtained in the above process by burning 35kg of cow dung cakes is shown in Figure 2.5.

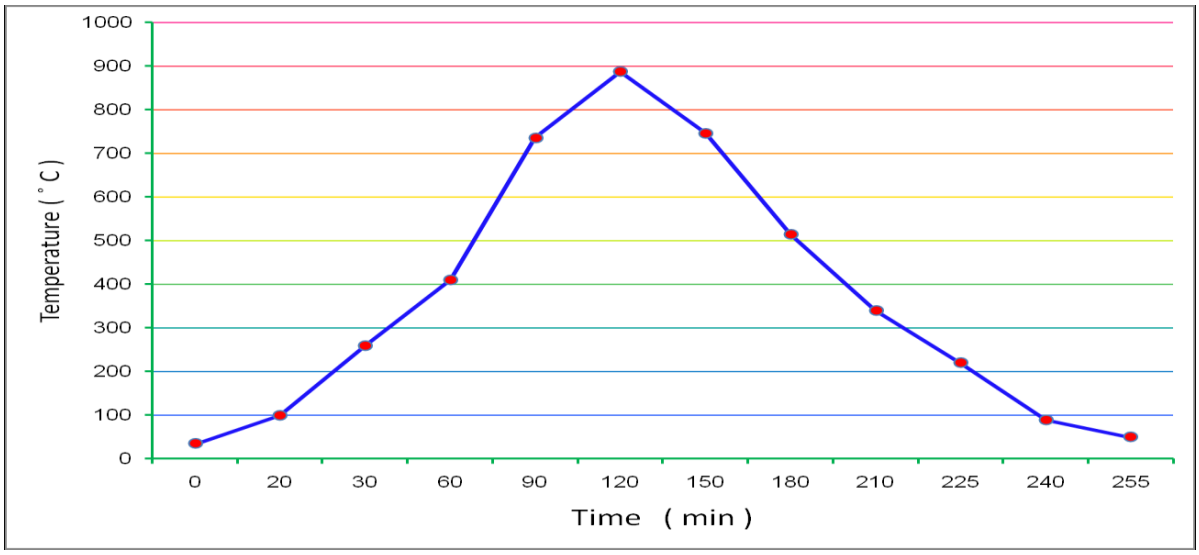

Figure 2.5 : Temperature pattern obtained by burning 35 kg of cow dung cakes (Devanathan, 2013)

2.5. Characterization of ash (*bhasma*)

The characterization of ash is done by traditional methods of evaluation (*Bhasma pariksha*) and modern analytical techniques.

2.5.1. Traditional methods

Characterization can be done by physical, chemical and physico-chemical tests.

1. Physical tests

   There are certain physical characteristic that properly processed *Bhasma* should possess (Chaudhary, 2010).

   (i) Colour (*Varna*)

   A specific colour is mentioned for each ash (*bhasma*). Alteration in specific colour suggests that *bhasma* is not prepared properly. Because a particular metallic compound is formed during *Bhasma* preparation and every chemical compound possesses specific colour.

(ii) Lusterless (*Nishchandratvam*)

Ash must be lusterless before therapeutic application. Luster is a character of metal. After proper incineration, luster of metal should not remain. For this test, ash is observed under bright sun light, whether luster is present or not; if luster is still present, it indicates further incineration.

(iii) Floatability (*Varitara*)

Floatability test applied to study lightness and fineness of final ash is floating character of ash on stagnant water surface. This test is based on the law of surface tension. Little amount of ash is taken and sprinkled slowly on stagnant water surface from a short distance. Properly incinerated ash will float on water surface.

(iv) Fineness (*Rekhapurnata*)

This test is applied to study fineness of ash. Ash particles should be of minimum size for easy absorption and assimilation in the body. Ash should be so fine that it can fill furrows of finger tips. A little amount of ash is rubbed in between index finger and thumb to observe whether particle can fill furrows of finger tips.

2. Chemical Tests

(i) *Apunarbhava*

Apunarbhava means incapability to regain original metallic form. For this ash is mixed with equal quantities of seeds of Abrusprecatorius, honey, ghee, borax and jiggery and it is sealed in earthen pots and similar grade of heat used for preparation of particular ash is applied and on self cooling product is observed. Lustrous particles in it show presence of free metal, which is indicative of improper incineration (Sharma, 2004).

(ii) *Niruttha*

Nirutta is to test inability to regain metallic form of metallic ash. In this test, ash is mixed with a fixed weight of silver leaf, kept in earthen pot and similar grade of heat is applied and after self cooling, weight of silver is taken. Increase in weight of silver leaf indicates improperly prepared ash (Sharma, 2004).

3. Physico-Chemical Analysis

(i) Loss on drying

One gram of ash is taken in a crucible and dried in an oven at 105 ^{0}C for about 5 hours. The sample is allowed to cool and the dry mass is determined. The difference in mass is used to determine the loss on drying and express as percentage (Anonymous, 2001).

$$\text{Moisture content} = \left(\frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}}\right) 100$$

(ii) Estimation of Total Ash

A suitable quantity of the sample is weighed accurately in a silica crucible. The sample is spread uniformly on the bottom of the crucible, incinerated, cooled and weighed. Difference between the empty crucible weight and crucible with incinerated ash gives the total ash value (Anonymous, 2001).

(iii) Estimation of Acid Insoluble Ash

The residue from total ash estimation is boiled with hydrochloric acid. The insoluble matter is washed with hot water, transferred to a crucible, dried and weighed. Difference between the empty crucible weight and crucible with incinerated ash gives the acid insoluble ash value (Anonymous, 2001).

(iv) Estimation of Water Soluble Ash

The residue from total ash estimation is boiled with distilled water. The insoluble matter is washed with hot water, transferred to a crucible, dried and weighed. This weight was subtracted from the total ash taken which gives the water soluble ash content (Anonymous, 2001).

2.5.2. Modern analytical techniques

Use of modern analytical techniques is the highest need of time for the development of quality control parameters of Ayurvedic ashes. Further, traditional methods of evaluation of ashes can be proved by using modern analytical techniques.

1. XRD analysis

   The prepared ash can be analysed for their crystallinity or amorphous behaviour using X-ray diffraction (XRD). Generally metal oxide are crystalline in nature (Wadekar MP, 2005).

2. Chemical composition of ash

   The chemical composition of bhasma is analysed using energy dispersive x-ray analysis (EDX) attached to SEM or using Atomic Absorption Spectroscopy (Wadekar MP, 2005).

3. Partical size distribution

   During the process of incineration, particle size of the material reduces (Sarkar & Chaudhary, 2010) and therefore it increases solubility and hence bioavailability of final ash. Particle size distributions of ashes can be analyzed using Particle size analyzer.

3 METHODOLOGY

3.1. Cow dung cake Analysis

Cow dung cakes were prepared using different compositions of cow dung, paddy husk and saw dust as shown in Table 3.1 to identify suitable composition. Diameter and height of the mould used to prepare cow dung cakes were 6" and 1" (Nishteswar & Vidyanath, 2010) respectively. After sun drying, calorific value of each sample using Bomb calorimeter and moisture content were measured.

Table 3.1 : Compositions for preparation of cow dung cakes

Sample No.	Composition (%)		
	Cow dung	Paddy husk	Saw dust
1	60	40	-
2	82	18	-
3	76	24	-
4	63	20	17

Figure 3.1 : Cow dung cakes

3.2. Establishment of temperature profiles

In order to measure *Puta*, the pits were built using soil bricks and clay and the dimensions of the 3 types of *Puta* and number of cow dung cakes burnt are shown in Table 2. For *Varaha Puta* and *Gaja Puta*, number of cow dung cakes to be burnt is 150 and 1000 as reported in literature (Batta, 1997). However, the maximum

number of cow dung cakes that could be arranged in above 2 pits were 120 and 600 respectively even though the cow dung cakes were prepared according to the dimensions given in Ayurvedic texts. As mentioned in literature (Vagbhatacharya, 2003), 1500 cow dung cakes could be arranged in the pit of *Maha puta.*

Table 3.2 : Details of *Puta* systems

Puta	**Dimensions (cm)**	**No. of Cow dung cakes**	**W't of Cow dung cakes (kg)**
Varaha Puta	42 × 42 × 42	120	12.7
Gaja Puta	57 × 57 × 57	600	81.8
Maha Puta	90 × 90 × 90	1500	186.7

Arrangement of cow dung cakes for *Varaha Puta*:

80 cow dung cakes were filled to the *Varaha Puta* pit, placed the sealed earthen crucibles containing mineral and then they were covered with remaining 40 cow dung cakes. Partly filled cow dung cakes in *Varaha Puta* are shown in Figure 3.2.

Figure 3.2 : Partly filled cow dung cakes in *Varaha Puta*

Arrangement of cow dung cakes for *Gaja Puta*:

400 cow dung cakes were filled to the *Gaja Puta* pit, sealed earthen crucible containing materials was placed and then they were covered with remaining 200 cow dung cakes. Figure 3.3 shows the arranging of cow dung cakes in *Gaja Puta*.

Figure 3.3 : Arranging of cow dung cakes in *Gaja Puta*

Arrangement of cow dung cakes for *Maha Puta*:

1000 cow dung cakes were filled to the *Maha Puta* pit, sealed earthen crucibles containing material was placed and then they were covered with remaining 500 cow dung cakes. Burning of cow dung cakes in *Maha Puta* is shown in Figure 3.4.

Figure 3.4 : Burning cow dung cakes in *MahaPuta*

Thermocouple wires were inserted into the crucibles containing material for all the three *Putas* to record temperatures inside the crucibles (approximately 250 ml volume) using Temperature Data Logger (Model: TM-947SD, Type: 4 Channel Thermometer).

Then cow dung cakes were ignited on all four sides and in the middle of the pit at once and when burning was over, the contents in the crucible were allowed to self cool completely.

3.3. Verification of temperature profile of *Varaha Puta*

Chalcopyrite ash was prepared in the traditional method and using the muffle furnace for the verification of the temperature profile obtained from the traditional *Varaha Puta*.

Among the various methods described in the literature review, method described by Vagbhatacharya was selected for the preparation of Chalcopyrite ash.

1. Analysis of Chalcopyrite

   Chalcopyrite mineral samples purchased from the local market from three different suppliers and also imported chalcopyrite ash available in the market were analyzed for its composition of Cu, Fe and Si using Atomic absorption Spectrophotometer (Model: GBC933AA, Type: Atomic Absorption Spectrophotometer).

   When samples do not contain the required Cu content of Chalcopyrite mineral, Cu was externally supplemented in the form of Copper Sulphide to supplement the required Cu content. Therefore, 90 g of powdered mineral was mixed with 95 g of Copper Sulphide to adjust the Cu content and the sample was again analyzed to identify the composition.

2. Purification of Chalcopyrite

   Then the prepared Chalcopyrite mixture (195 g) was roasted in an open pan with 200 ml of lime juice for 48 minutes as proposed by Vagbhatacharya.

Figure 3.5 : (a) - Raw Chalcopyrite (b) - purified Chalcopyrite samples

3. Purification of Sulphur

   Sulphur was purified using the method proposed by Vagbhatachrya. Approximately 2 L volume clay pot was filled with 1 L of cow's milk and the

mouth of the clay pot was tied with a piece of cloth. Then cow's ghee was applied on the cloth. Then 245g of powdered Sulphur was placed on the cloth (Figure 3.6-a) and that was covered and sealed with another similar size clay pot using clay-smeared cloth (Figure 3.6-b). After that the pot containing milk was buried and heated the outside of the upper clay pot using 2.5kg of cow dung cakes (approximately 20 cow dung cakes) (Figure 3.6-c). Then the melted Sulphur dropped gradually into milk. Finally Sulphur in the milk was washed with warm water and the process was repeated three times using the resultant Sulphur. The resultant Sulphur obtained after three processes is shown in Figure 3.6-d.

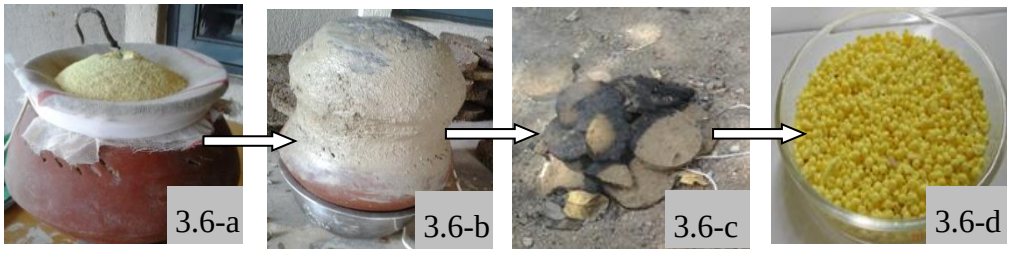

Figure 3.6 : Sulphur purification procedure

4. Preparation of pellets

Equal amounts of purified Chalcopyrite and purified Sulphur (200 g of each) were put into the mortar and lime juice was added until all the solids were covered. Then mixture was ground until all the lime juice evaporated, pellets were made (approximately 2cm diameter and 3mm thick) using resultant dough and sun dried. Grinding of mixture and prepared pellets are shown in Figure 3.7-a and 3.7-b respectively.

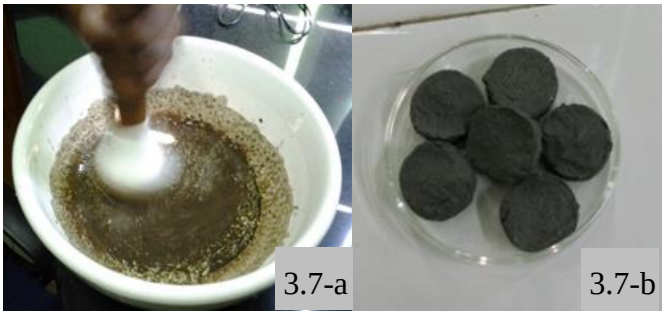

Figure 3.7 : Preparation of pellets

5. Incineration of pellets in *Varaha Puta*

38 numbers of sun dried pellets (149.6420 g) were placed in the earthen crucible and it was sealed with another crucible using clay smeared cloth. Then it was incinerated in *Varaha Puta* (*Puta* 1) using 120 cow dung cakes and allowed to self cool. The resultant *bhasma* from Puta1 was ground again with lime juice, pellets were prepared and incinerated in *Varaha Puta*. This process was repeated 5 times up to Puta 5 and material used in each *puta* is shown in Table 3.3.

Table 3.3 : Materials used for incineration of Chalcopyrite pellets in *Varaha Puta*

	Puta 1	***Puta 2***	***Puta 3***	***Puta 4***	***Puta 5***
Wt of cow dung cakes (kg)	12.5	12.9	12.4	12.6	12.4
No. of pellets burnt	38	15	10	8	7
Wt of pellets incinerated (g)	149.6420	55.4030	35.9800	29.245	24.2341

6. Incineration of pellets in muffle furnace

37 numbers of sun dried pellets (148.214g) were placed in the earthen crucible and sealed with another crucible using clay smeared cloth. Then it was incinerated in muffle furnace following the temperature profile obtained for traditional *Varaha puta*. Temperatures were set manually and set temperatures with time are shown in Table 3.4.

Table 3.4 : Set temperatures with time duration for muffle furnace to prepare Chalcopyrite ash

Time (min)	Set Temperature
0	25
40	60
50	120
60	275
70	500
80	650
90	800
100	850
110	850
115	off

The resultant ash (*bhasma*) from Cycle 1 was again ground with lime juice, pellets were prepared and incinerated in muffle furnace. This process was repeated 5 times up to Cycle 5 and materials used in each cycle is shown in Table 3.5.

Table 3.5 : Materials used for incineration of Chalcopyrite pellets in muffle furnace

	Cycle 1	**Cycle 2**	**Cycle 3**	**Cycle 4**	**Cycle 5**
No. of pellets burnt	37	15	11	8	6
Wt of pellets before incineration(g)	148.214	54.45	35.0649	28.1246	23.5782

7. Analysis of final ash

Final ashes prepared in both traditional *Varaha Puta* and in the muffle furnace were analyzed using traditional tests mentioned in ayurvedic texts such as colour (*varna*), lusterless (*Nishchandratvam*), floatability (*varitara*), fineness (*Rekhapurnata*), *apunarbhava*, *nirutta.* Further, loss on drying, total ash, acid insoluble ash, chemical composition (using Atomic Absorption Specroscopy), particle size distribution (using Laser Particle size analyzer) of the resultant products were estimated and X-Ray Diffraction (XRD) analysis was also done.

4 RESULTS AND DISCUSSION

4.1. Analysis of cow dung cakes

Table 4.1 shows the average calorific value and moisture content of each cow dung cake sample prepared.

Table 4.1 : Properties of cow dung cakes

Sample No.	Avg. Calorific Value (MJ/kg)	Moisture Content (%) (dry basis)
1	15.18	15.84
2	15.30	14.97
3	15.44	15.56
4	14.65	14.28

Maximum Calorific Value was obtained for cow dung cakes prepared using 76 % cow dung and 24 % paddy husk mixture (dry basis) and it was the most stable mixture among the prepared samples. Therefore, that composition was selected to prepare cow dung cakes as the fuel for traditional *puta* furnace.

4.2. Establishment of temperature profiles

Temperature profile of traditional *Varaha Puta* (temperature inside the crucible) is shown in Figure 4.1.

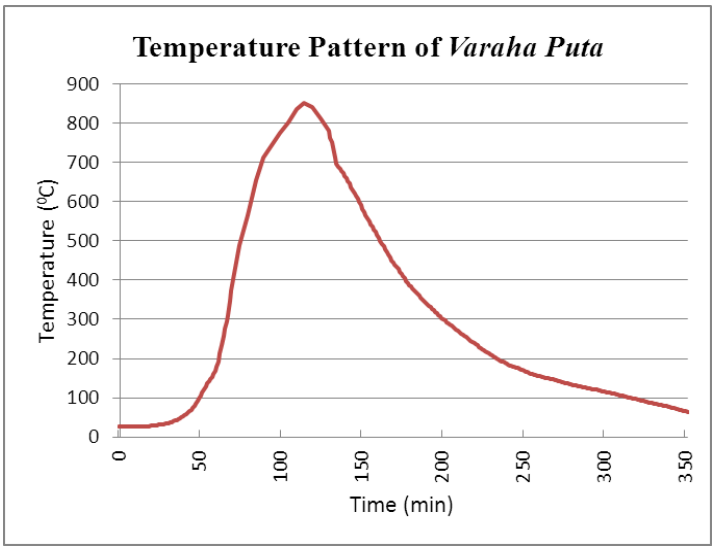

Figure 4.1 : Temperature profile of traditional *Varaha Puta*

In *Varaha Puta* furnace, the temperature gradually increased 35 min after ignition. Peak temperature observed was 850 ^{0}C at 115 min after ignition and that temperature remained for 5 min. Temperatures above 750 ^{0}C and above 600 ^{0}C were maintained for 35 min and 70±5 min respectively. *Varaha Puta* took about 5 hrs for self cooling (to 40 ^{0}C).

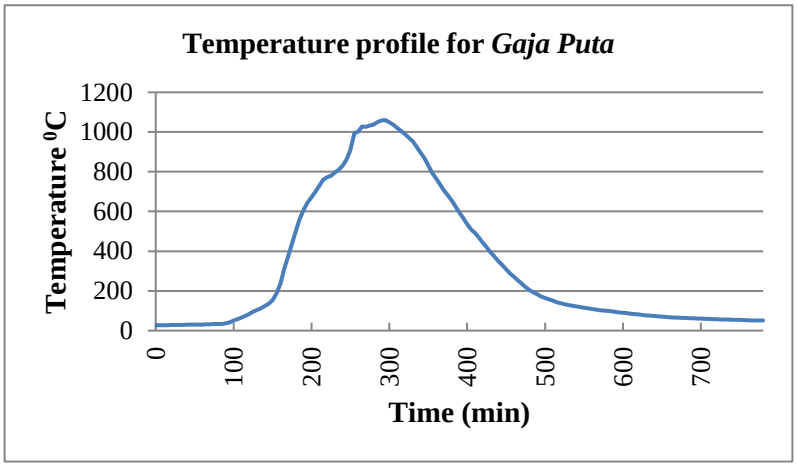

Figure 4.2 : Temperature profile of traditional *Gaja Puta*

As shown in the temperature profile of *Gaja Puta* (temperature inside the crucible in Figure 4.2) 90 ± 5 min after ignition, temperature gradually increased and the peak temperature was observed as 1060 ^{0}C at 295 min after ignition. Temperatures above 1000 ^{0}C, 800 ^{0}C and 600 ^{0}C were observed for 55 min, 125 min and 195 min respectively and about 11 hrs was taken for self-cooling (to 40 ^{0}C) of the *Gaja Puta*.

Temperature profile for *Maha Puta* (temperature inside the crucible) is shown in Figure 4.3 and temperature started to increase after 100 min of ignition. Peak temperature observed was 1360 ^{0}C at 305 min after ignition and it was started to decrease within another 7-8 min. Temperatures above 1300 ^{0}C and above 1100 ^{0}C were maintained for 40 min and 90 min respectively. Temperatures above 1000 ^{0}C, 800 ^{0}C and 600 ^{0}C were maintained for 115 min, 160 min and 210 min respectively. 13 hrs required for the self-cooling (to 40 ^{0}C) of the *Maha puta.*

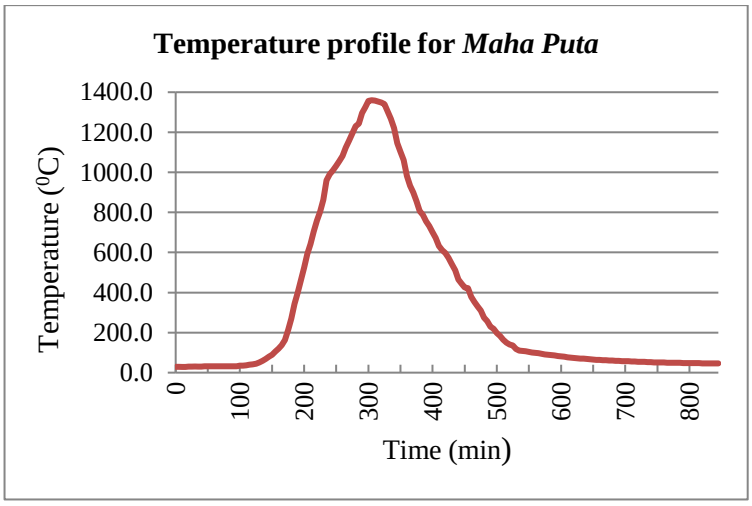

Figure 4.3 : Temperature profile of traditional *Maha Puta*

4.3. Verification of Temperature profile of *Varaha Puta*

1. Composition analysis of Chalcopyrite

   Even though Cu is one of the major elements in Chalcopyrite, three mineral samples purchased from different suppliers contained <0.02 % Cu by weight. Further, two of the imported Chalcopyrite ash samples purchased from the local market also did not contain considerable amount of Cu. Fe, Cu and Si contents of initial mineral samples is shown in Table 42. Therefore, CuS was added externally to adjust the required Cu percentage in the initial minerals.

Table 4.2 : Characterization of Chalcopyrite Samples

Constituent	Mineral samples			Ash samples	
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2
Fe (wt %)	37.3	44.10	40.11	60.44	69.83
Cu (wt %)	<0.01	<0.01	0.02	<0.01	<0.01
Si (wt %)	3.95	2.28	2.8	-	-

2. Incineration of pellets in *Varaha Puta* and in muffle furnace

   Weight loss after incineration of pellets in the traditional *Varaha Puta* and in the muffle furnace are shown in Tables 4.3.

Table 4.3 : Weight loss of ashes after incineration in traditional *Varaha Puta* and in muffle furnace

	***Puta* 1/ Cycle 1**	***Puta* 2/ Cycle 2**	***Puta* 3/ Cycle 3**	***Puta* 4/ Cycle 4**	***Puta* 5/ Cycle 5**
wt loss (%) in traditional *Varaha Puta*	59.38	25.02	13.74	9.84	8.72
wt loss (%) in muffle furnace	60.44	26.20	13.20	9.52	8.34

The weight loss during the each cycle in *Varaha Puta* and muffle furnace incinerated ashes are approximate to each other. This is an indication of the similarity of heat supplied in each cycle in both methods.

Colour change of resultant ashes from both methods during each stage and floatability of final ashes on surface of water are shown in Figure 4.4 and 4.5 respectively. It was observed that in both methods, colour has gradually changed from dark brown to reddish brown during 5 incineration stages indicating the similarity of heat supplied.

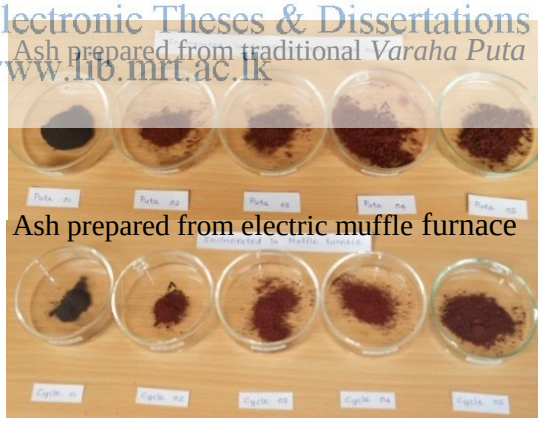

Figure 4.4 : Colour of resultant Chalcopyrite ash

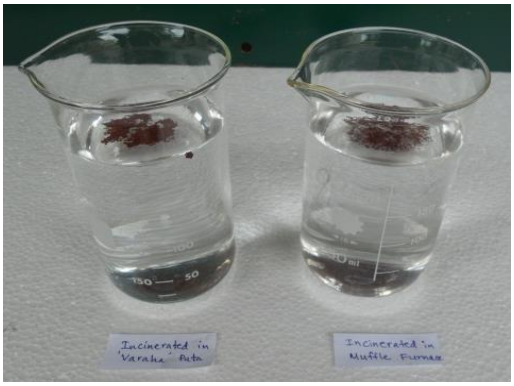

Figure 4.5 : Floatability test for Chalcopyrite ash

Results obtained for physical tests are summarized in Table 4.4.

Table 4.4 : Physical, chemical & physico-chemical properties of resultant ash

Property	**Traditional *Varaha Puta***	**Muffle Furnace**	**Recommended values**
Colour	Reddish Brown	Reddish Brown	Reddish Brown (Anonymous, 2010)
Rekapurnata	Fills the space between finger lines	Fills the space between finger lines	Fills the space between finger lines
Varitara	Floats on surface of water	Floats on surface of water	Floats on surface of water
Nirutta	Silver wt remain unchanged	Silver wt remain unchanged	Silver wt remain unchanged
Loss on drying	0.16 % (w/w)	0.25 % (w/w)	Not more than 0.5 % (w/w) (Anonymous, 2010)
Total ash	99.84 % (w/w)	97.29 % (w/w)	
Acid insoluble ash	19.39 % (w/w)	16.95 % (w/w)	Not more than 21 % (w/w) (Anonymous, 2010)

Colour of the final ashes (*bhasma)* obtained from both methods were reddish brown and it is similar to the colour of Chalcopyrite ash mentioned in Ayurvedic text (Anonymous, 2001). Both ashes (*bhasma*) filled the space between finger lines and floated on the surface of water confirming that they achieved the required fineness of the final *bhasma*. Weight of silver has not increased in both ashes and it indicates that there is no free metallic portion in the *bhasma* samples. According to Anonymous (2001), loss on drying of final *bhasma* should not more than 0.5 % w/w and *bhasma* prepared from both traditional *Puta* and from electric muffle furnace shown 0.16 % and 0.25 % of loss on drying respectively. Therefore results obtained were within the required level of loss on drying. The total ash contents of *Varaha Puta* and muffle furnace incinerated ashes are 99.84 % and 97.29 % respectively. Recommended acid insoluble ash content in Chalcopyrite ash is not more than 21 % (w/w) and the obtained are within the recommended range.

Chemical composition of raw mineral and resultant ashes is shown in Table 4.5.

Table 4.5 : Chemical Composition

Chemical constituent	**Wt (%)**			
	Raw mineral	**Pellets**	**Final *bhasma***	
			Puta	**Muffle**
Fe_2O_3	15	7.5	47	38
Cu	38	19.0	44	35
SiO_2	1.8	0.9	2	traces

Initial compositions of formulated Chalcopyrite by externally adding CuS were 15 % Fe_2O_3, 38 % Cu and 1.8 % SiO_2. In this preparation, more attention was paid to maintain correct Cu % in the initial sample as Cu is the main element in Chalcopyrite. The composition of Fe, Cu, SiO_2 in pellets becomes approximately half of the initial raw mineral composition since Chalcopyrite was mixed with equal amount of Sulphur in the preparation of pellets. After incineration, Fe, Cu & SiO_2 contents in ashes prepared in *Varaha Puta* were 47 %, 44 % & 2 % respectively while they were 38 %, 35 % & 2.75 % respectively in the ash prepared in the muffle furnace. The required Copper content in final *bhasma* should be in the range of 15-18

%. However, the results obtained in this work have deviated from the recommended range. The reason for this variation may be due to formulation of Chalcopyrite mineral and therefore the chemical structure / bonding of artificially prepared mineral can be different from the original mineral.

It is well known that size reduction of particles increases solubility and hence bioavailability. The particle size distributions of ashes prepared in *Varaha Puta* & muffle furnace are shown in Figures 4.6 and 4.7 respectively.

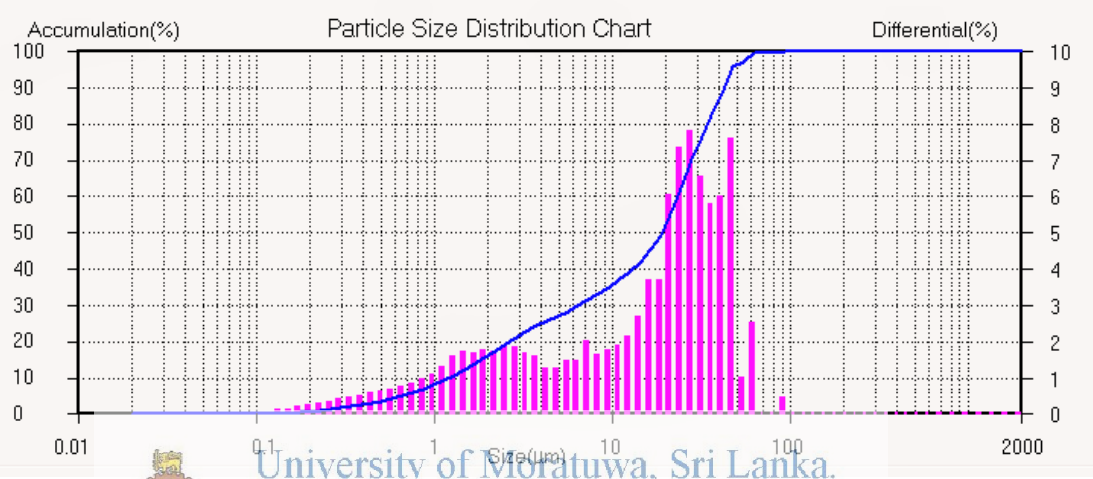

Figure 4.6 : Particle size distribution of Chalcopyrite ash prepared in *Varaha Puta*

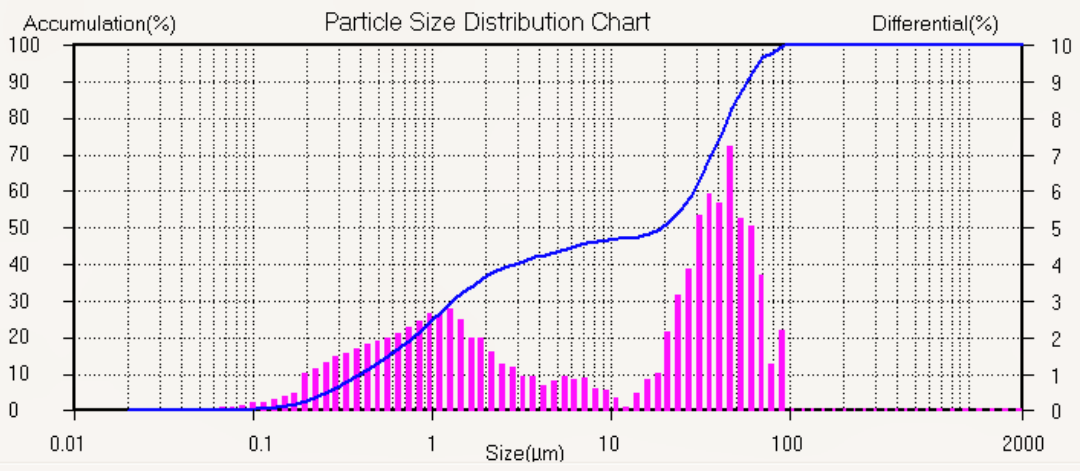

Figure 4.7 : Particle size distribution of Chalcopyrite ash prepared in Muffle furnace

Considering the particle size distributions of Chalcopyrite ashes, particle size of both ashes were varied in the range 0.05 – 92.57 µm (in diameter). As a percentage, 35.71 % of total ash prepared using traditional *Varaha Puta* is below 10 µm in diameter and that of in muffle furnace is 47.03 %. Further it showed that 50 % of particles of both ashes were below the 20 µm. Previous studies also, have shown that particle size of the Chalcopyrite ash varied between 3-100 µm (Devanathan, 2013). Therefore, obtained particle size distributions of both ashes are comparable to each other and similar with previous studies.

The XRD spectrums of initial pellets and ashes prepared in *Varaha Puta* and in muffle furnace are shown in Figures 4.8, 4.9 & 4.10 respectively.

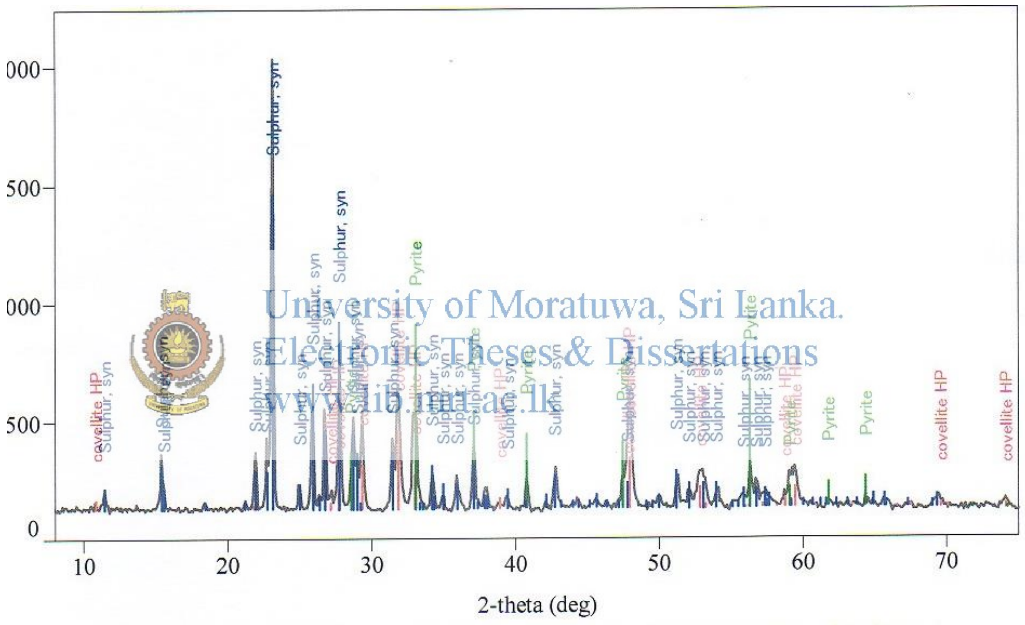

Figure 4.8 : XRD spectrum of initial pellets

According to the XRD spectrum of initial pellets, the sample contains Covellite (CuS), Pyrite (FeS_2) and Sulphur as major components. For preparation of initial pellets, CuS was artificially added to the pyrite mineral to adjust the Cu composition in the initial mineral. Further, Sulphur was added to it for preparation of pellets and therefore, CuS, FeS_2 and Sulphur phases are present in the initial sample.

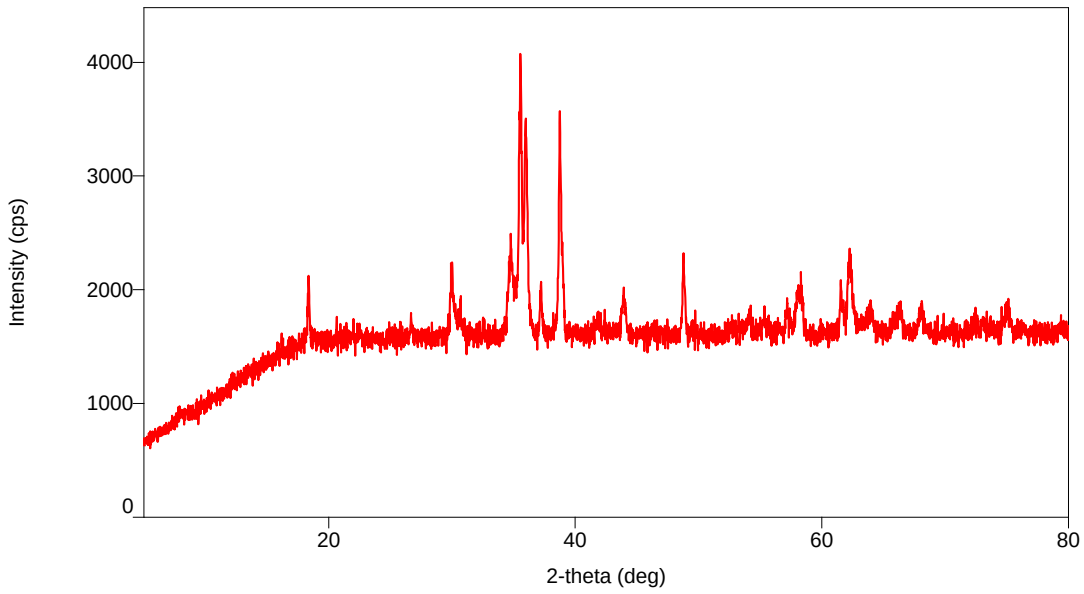

Figure 4.9: XRD spectrum of Chalcopyrite ash prepared in *Varaha Puta*

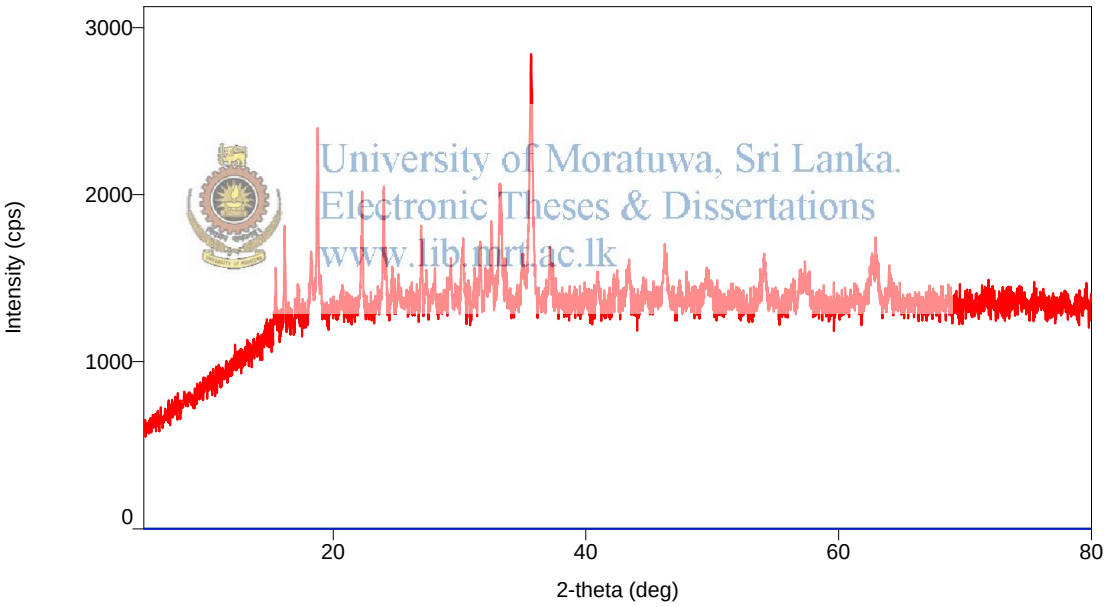

Figure 4.10 : XRD spectrum of Chalcopyrite ash prepared in muffle furnace

According to XRD spectrums of final ashes, possible phases present in ash prepared from *Varaha puta* are Chalcopyrite ($CuFeS_2$) and Cuprospinel ($CuFe_2O_4$) and that of from muffle furnace are Chalcanthite ($CuSO_4 \cdot 5H_2O$) and Iron Tin Oxide. The formation of some different compounds in the final products may be due to oxidation and reduction reactions of Cu, Fe with Sulphur in the presence of Oxygen.

Further, both Cu and Fe forms various complexes even under slightly different thermal conditions and therefore phases present in the final ashes may be varied.

5 CONCLUSION & RECOMMENDATIONS

Maximum Calorific Value was obtained as 15.44 MJ/kg for cow dung cakes prepared using 76 % cow dung and 24 % paddy husk mixture (dry basis) and also that was the most stable mixture among the prepared samples.

Temperature profiles for *Maha Puta*, *Gaja Puta* and *Varaha Puta* were determined using dried cow dung cakes as the fuel and the maximum temperatures achieved for *Maha Puta*, *Gaja Puta* and *Varaha Puta* were 1380 ^{0}C, 1060 ^{0}C and 850 ^{0}C respectively. Further, the temperature pattern of traditional *Varaha Puta* was matched with a muffle furnace and Chalcopyrite ash (*Swarna Makshika bhasma*) was prepared using both traditional method and electric muffle furnace and compared properties of the resultant ash (*bhasma*) from the two methods. The ash produced using both methods have shown comparable properties according to classical tests of Ayurveda and modern analytical techniques. Therefore, temperature profile established for traditional *Varaha Puta* by burning cow dung cakes was verified using the electric muffle furnace and then muffle furnace can be used to prepare ashes those used *Varaha Puta* for their preparation process.

Special attention should be paid when selecting initial raw material for ash preparation because some materials specially minerals purchased from local market did not contain required elements of the mineral.

Therefore, when preparing various ashes in muffle furnace, temperature data which were obtained from traditional *Varaha Puta*, *Gaja Puta* and *Maha Puta* methods can be utilized to control the temperature in muffle furnace.

Further research studies can be carried out in following areas to identify the effectiveness of the Chalcopyrite ashes prepared using traditional method and muffle furnace and to verify the temperature profiles of traditional *Gaja Puta* and *Maha Puta*.

1. To analyze the effectiveness of Chalcopyrite ashes prepared using traditional *Varaha Puta* and muffle furnace, experimental studies are necessary to carry out with selected animals by administering them both ashes.

2. To verify the temperature profiles of traditional *Gaja Puta* and *Maha Puta*, ashes can be prepared using traditional methods and muffle furnace and then properties of ashes should be compared.

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