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Particle Size Analysis of *Palakarai Parpam*-A *Siddha* Drug

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Abstract

Parpam (*Bhasma*) is one form of internal medicine widely and effectively used in *Siddha* system of medicine. Shells of *Cypraea moneta* Linn. is commonly known as *cowrie* or *cowry*. In *Siddha*, it is known as *Palakarai* and in *Ayurveda* it is called as *Varatika bhasma*. The ingredients that are being used for the preparation of a parpam have to be subjected to several stages of "*pudam*", known as calcination process. The selected *parpam* is subjected to analysis of particle size distribution through Scanning Electron Microscopy (SEM), XRD and Particle Size Analyser. The atomic and weight percentage of elements using SEM/EDS are also studied. The results of SEM/EDS, XRD and Particle Size Analyser are discussed in this paper.

Keywords: *Cypraea moneta, pudam* , particle size, SEM/EDS analysis, *Siddha* drug

INTRODUCTION

Generally, minerals directly or indirectly are being inducted as medicine in Indian system of medicine as well as Allopathic system (Eleza, *et al.*, 2002). With the development of specialized processing techniques, minerals and metals are used frequently in therapeutics, especially in the form of *parpams* (*bhasmas*). (Jha. *et al., 2007).* Shells of *Cypraea moneta* Linn. are commonly known as *cowries* and called as *chozhi* (or) *palakarai* in Tamil. It belongs to the phylum Mollusca and the class Gastropoda. Similar to the shells of oyster, *palakarai* also is the shell for protecting the sea mollusc C*ypraea moneta*. The shell, *palakarai,* is obtained abundantly along the Indian coast. In ancient days palakarai were valued as currency equal to gold, and used as ornamental materials. In traditional system of medicine the shells of *Cypraea moneta*, have been used as medicine to cure various ailments mainly related with stomach (Vedhagiri *et al.,2007).* Palakarai parpam was found to be effective in anti-pyretic and anti-inflammatory in experimentally induced albino rats (Devanathan, *et al., 2002).* Also it is used in treating Stomach poisoning and all toxaemic states, colic, retention of urine, gonorrhoea and inflammation of urino-genital tract. (Anonymous, *1993).* The particle size of the drug is an important factor for deciding its efficacy. The smaller the particle size the greater is the potency.(Panse,*2002).*This paper documents the particle characteristics of *palakarai parpam* by SEM/EDS, XRD and particle size analyze.

MATERIALS AND METHODS

The prepared sample of palakarai parpam as per the procedure specified (Kaviraj,*2001)* has been procured from Kaviraj pharmaceuticals, Moolapalayam of Erode District, Tamil Nadu and labeled as JVP. The electron microscopy has become an indispensable tool for research and development. Morphological and compositional information have been obtained from SEM and EDS analysis. (Yousuf *et al.,* 1992). It has a key role to play in the field of environmental and forensic science, industrial development, medical and biological fields pertaining to the examination of cells, tissues and micro organisms. The particle sizes are usually viewed in terms of geometrical shapes such as spherical surface, volume etc., or behavioral equivalence, like sedimentation (Burgers *et al., 2004).* The particles are randomly selected and measured from the micrographs. The SEM micrograph of 400 magnification has a multiplying factor width of 50 µm. Scanning electron microscope (SEM-Model: JEOL JSM 5610 Series) was used to examine the morphology and the particle size of the drug and SEM image of four hundred magnifications are considered. Probable elements present in atomic and weight percentage are analyzed using EDS.

The XRD technique is employed to assess the phase purity and crystallographic changes during calcinations process of *parpam*. The XRD pattern of *parpam* is recorded with high-resolution powder X-ray diffractometer, PANalytical –Philip–Netherlands / Model XPert Pro, with Cu-Kα radiation (l = 1.54056 A°) at 40 kev and 30 mA at the scanning rate of 2° per minute and 2θ was varied from 5 to 60°.

RESULTS AND DISCUSSIONS

SEM *Particle Size Analysis*

Figure-1 is representing SEM image of 400 magnifications of JVP. It is observed that the morphology of the particles is of granular amorphous nature. Most significant changes of particle size have been observed due to calcinations process. The range of particle size is varied from 12.82 to 8.34 microns at the

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final stage which clearly indicates the particle size reduction due to the repeated calcinations.

SEM-EDS Analysis

The Electron Density Spectrum of 400 magnifications of JVP is given in Figure 2. The elemental compositions of *palakarai parpam* are given in Table 1. Finally the weight percentage of calcium is maintained to 80.02% at the last stage. Magnesium, Zinc, Strontium and Barium were the other elements present as listed in Table 1. It is concluded that *palakarai parpam* is consisting mainly of calcium and Magnesium, Barium and a trace amount of Strontium as reported.

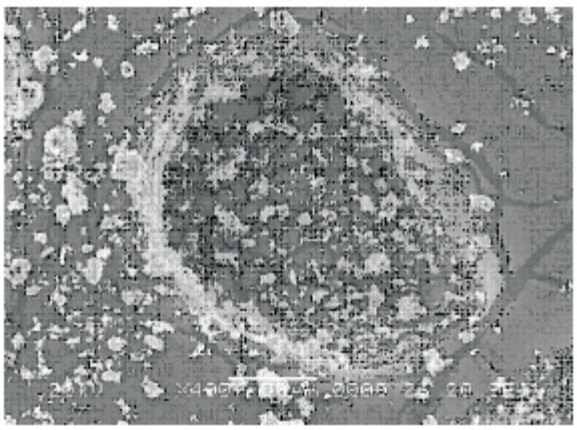

Figure 1. SEM image of *Palakarai Parpam* (400 Magnification)

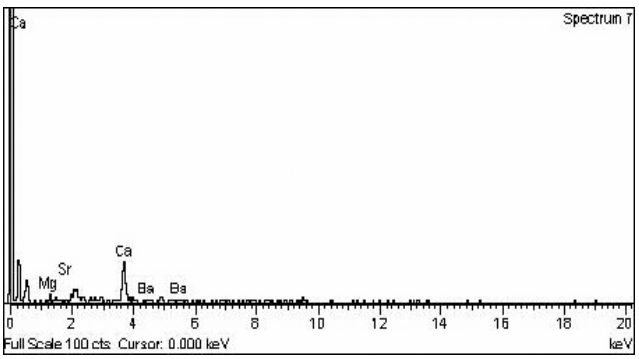

Figure 2. SEM –EDS of *Palakarai Parpam* (400 Magnification)

Table 1. Elemental Composition of Palakarai Parpam

S. No.	Element	Weight %	Atomic %
01	Calcium	80.02	78.38
02	Magnesium	11.70	18.90
05	Strontium	2.19	0.98
06	Barium	6.08	1.74

XRD Analyses

The XRD pattern (Fig 3) is analyzed and the peaks are indexed. Powdered XRD patterns of initial stage showed the characteristic peaks of poor crystalline calcium carbonate. All the peaks are indexed from their observed d-values. The structure is found to be orthorhombic with lattice parameters a =3.8502 A°, b = 10.8675 A°, c = 23.3284 A°, and $\alpha = \beta = \gamma = 90°$ with unit cell volume 976.19 $(A°)^3$.It is a known fact that calcite form of calcium carbonate is always of hexagonal structure. The peak around 2θ of 29° has been considered for particle size calculation. The increase in the intensity of the peaks is indicating the improvement of crystallinity due to calcinations process. The increase in unit cell volume is attributed to the increase of surface area which is a vital factor in drug interaction. The peaks around 2θ of 29.2° to 29.6° are attributed to the presence of calcium carbonate (Davis, *et al.*, 1997). The sharp peaks observed at JVP clearly implicates that the repeated calcinations promote crystallinity which is accountable for bioavailability and dissolution rate.

XRD Particle Size Analysis

X- Ray powder diffraction may be used to measure the average size in powdered sample. The lines in a powder diffraction pattern are of finite breadth but if the particles are very small the lines are broader than usual and the broadening increases with decreasing particle size. (Cullity, 1990).Using Debye-Scherrer's formula the particle size is determined

$$t = k \lambda / \beta \cos \theta$$

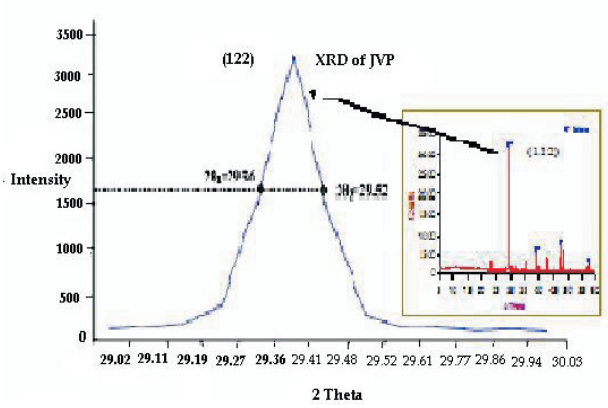

Figure 3. XRD of Palakarai Parpam

Where k is the size factor = 0.97, λ is the wave length of X-ray radiation used (1.540560×10^{-10} m) and 2θ is the angle at which the maximum intensity was observed. The full width at half maximum in radians $\beta = (2\theta_1 - 2\theta_2)/2$ = .0013963 radians and θ the angle corresponds to (122) Bragg reflection in radians (=29.44/2=14.72(Π/180) =0.256912 radians) and thus the particle size is determined as t = 10.70 microns.

Particle Size Analysis

The sample is also subjected to particle sizing systems. The summary of Number Weight Cumulative Distribution is presented in Table 2. It can be seen that 99% of the total particle number lies less than 12.25 microns and number of weight cumulative distribution varies between 12.25(99%) to less than 0.57(5%) microns. The volume weighted distribution is 5% of the total volume at less than 0.57 microns size with 99% of the total volume at less than 12.25 microns.

Table 2. Summary of Number Weight Cumulative Distribution

Particle Sizing Systems, Inc.
Santa Barbara, Calif., USA

Model 780 AccuSizer

CARISM - SASTRA
File Name = CSum_JVP080408 Time Date = 15:29: 6 4/ 8/2008
Sensor Model: LE400-05S EXT S/N: 0605903 Cal. File: 0605903S.sns
Date of Calibration: 05-16-06

Elapsed Time of Data Collection = 10 Sec.
Background File = BL080408.103
Combined -BL080408.103
Summary of Number Wt. Cumu. Distribution

5 % of total particle Number <	0.57 microns
10 % of total particle Number <	0.62 microns
15 % of total particle Number <	0.73 microns
20 % of total particle Number <	0.92 microns
25 % of total particle Number <	0.92 microns
30 % of total particle Number <	1.22 microns
35 % of total particle Number <	1.22 microns
40 % of total particle Number <	1.22 microns
45 % of total particle Number <	1.22 microns
50 % of total particle Number <	1.73 microns
55 % of total particle Number <	1.73 microns
60 % of total particle Number <	1.73 microns
65 % of total particle Number <	2.45 microns
70 % of total particle Number <	2.45 microns
75 % of total particle Number <	2.45 microns
80 % of total particle Number <	3.46 microns
85 % of total particle Number <	3.46 microns
90 % of total particle Number <	4.47 microns
95 % of total particle Number <	7.48 microns
99 % of total particle Number <	12.25 microns

User Defined Peaks:

Number Weight Mean Diameter = 2.44 um (Full Range; System defined)
Stnd Dev. = 2.92 um (119.8 %); Mode = 1.73 um; Median = 1.73 um
# of Particles in Range = 14906; Skewness = 4.88;

CONCLUSIONS

The particle size of *parpam* is reduced due to repeated heat treatment. Particle size from SEM and XRD particle size analysis agree well and the estimated size is around 10.7 microns. SEM-EDS analysis reveals that *palakarai parpam* is having calcium as prominent element and magnesium, barium and strontium as trace elements. The XRD study reveals that the crystallinty has been improved due to calcinations. The crystal structure is found as orthorhombic.

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