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Chemometric studies on mineral distribution and microstructure analysis of freeze-dried *Aloe vera* L. gel at different harvesting regimens

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ABSTRACT

This paper presents an analysis of elemental distribution, microstructure at pre- and post-rehydration, and swelling of freeze-dried *Aloe vera* L. gel, obtained from two, three and four-year-old plant in summer. To explore the seasonal influence, three-year-old plants from rainy and winter season are also considered for analysis. Mineral distribution is studied following the estimation of Ca, K, Mg, P, Na, Mn, Se, Al, Fe, Zn, Cu and Cd. A consolidated effort has also been made to study the concentration of total C and N content of gel at different growth periods. Re-hydration mediated continuous topography in poly-dispersed, unorganized and amorphic freeze-dried gel accounts for the post-rehydration cross-linking among the component polysaccharides. Chemometric analysis (principal component and cluster analysis) suggests that the mineral content of gel change as a function of growth periods of plants. High content of organic C and Se with maximum water absorbing potential (in the form of swelling activity) are mainly associated with three-year-old aloe of summer; the contents of N, Na, Ca, K, Mg, P and Cu are observed to be high in two-year plants. To the best of our knowledge and belief, this is the first time, Se (a bioactive antioxidant activity promoting element) is found to be present in aloe gel.

1. Introduction

In the present investigation *Aloe vera* L. (*A. vera*), native to Mediterranean region, is a plant from the Xanthorrhoeaceae family (Angiosperm Phylogeny Group III System, 2009; Ray and Aswatha, 2013), considered as the plant-system to study the mineral distribution at different age groups and harvesting seasons. Various therapeutic properties of *A. vera* (Choi and Chung, 2003; Hamman, 2008; Dutta Gupta, 2010; Ray et al., 2012, 2013) which is attributed by the diverse array of component compounds in gel including a combination of polysaccharides and their derived compounds, glycoproteins, phenolics, enzymes, minerals, amino acids, sterols, saponins, vitamins, *etc.* (Eshun and He, 2004; Rodriguez et al., 2010; Ray and Dutta Gupta, 2013), makes it popular from ancient times of human history. To explicate the diverse array of bioactivity of *A. vera* gel (AG), Davis (1997) proposed the "Conductor-Orchestra concept" that explains the functional reciprocity among more than 200 biologically active components present in aloe parenchyma. Presumably, the diverse array of biological activities of AG is expressed by the synergistic action of the component compounds rather than a single component. Different bioactive potentials, moisturizing and excipient activities of AG are exploited for the production of creams, lotions, soaps, shampoos, facial cleansers, ointments, tablets, capsules and so on, and it appears as a base material in a myriad of cosmeceutical products (Eshun and He, 2004; Dutta Gupta, 2010; Rodriguez et al., 2010; Ray and Dutta Gupta, 2013). It is worthy to mention that hypoglycemic potential attributes to AG, of several mineral elements are demonstrated by different authors (Rajasekaran et al., 2005; Rajasekaran et al., 2006; Rajendran et al., 2007; Hamman, 2008). It has been documented that the presence of potassium ions attributes the wound healing activity to AG (Femenia et al., 1999). Calcium, one of the important minerals responsible for bone and teeth formation (Ozcan and Haciseferogullari, 2007), is found to be abundant in AG (Yamaguchi et al., 1993). In nutraceutical industry, AG is being used as a mineral source of different functional foods, and as a supplement in other food products for the production of various health drinks and beverages (Rajendran et al., 2007; Hamman, 2008; Rodriguez et al., 2010).

Most of the characterizations of AG were framed within the physical and biochemical paradigms during various processing

Abbreviations: *A. vera*, *Aloe vera* L.; AG, *A. vera* gel; FD, reeze-dried.

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techniques (Femenia et al., 2003; Miranda et al., 2009; Gulia et al., 2010; Rodriguez-Gonzalez et al., 2011, 2012). The modification of the bioactive substances during processing leads to certain variations in the potential physico-chemical properties and modulates the inherent bio-active potential of AG (Gulia et al., 2010). Among the different dehydration processes, freeze-drying is considered as a potential dehydration method, which conserves the physico-chemical properties of AG to a greater extent (Femenia et al., 2003). Different dehydration methods and processing techniques can modify the micro-structure of gel which can be delineated by scanning electron microscopy (SEM). The micro-structure of the AG under different drying temperature and processing techniques have been studied by different authors (Miranda et al., 2010; Rodriguez-Gonzalez et al., 2011; Vega-Gálvez et al., 2011); however, the effect of growth periods remains uninvestigated. Apart from the polysaccharides and different processing techniques, mineral concentrations also influence the bioactive potential and nutritional attributes of *A. vera* (Femenia et al., 1999, 2003; Rajasekaran et al., 2005; Miranda et al., 2009).

Different minerals have distinct biological significance in the human diet, which are categorized as macro and micro nutrients, with reference to the functional foods. The desired level of minerals in the daily diet is necessary to maintain the physiological balance and the synchronized functionality of the living cells (Lozak et al., 2002). It has been documented that the presence of different functional proteins in *A. vera* also influences the significance of the bioactive potential of gel (Kostalova et al., 2004; Hamman, 2008). Protein content in AG has been estimated as the factor of total nitrogen content at different processing techniques (Miranda et al., 2009; Gulia et al., 2010) though the influence of growth periods is not yet explored. Since the agronomic management information on *A. vera* is scarce (Rodriguez-Garcia et al., 2007; Ray and Aswatha, 2013), and AG has been supplemented in various health drinks and used as a functional ingredient in food (Hamman, 2008; Rodriguez et al., 2010), the knowledge of elemental distribution in AG in relation to different harvesting regimens would be of help in selecting the potential growth period of *A. vera* to optimize the components of the value chain of *A. vera* processing. Therefore it is imperative to study the mineral distribution of AG at different harvesting regimens.

In this context, we present an analysis of the influence of growth periods of plants on mineral distribution of AG. The chemometric studies with reference to principal component analysis and cluster analysis have also been employed to delineate the mineral distribution in gel from different growth periods of *A. vera*. A consolidated effort has also been made to study the total carbon content, nitrogen content, micro-structure and swelling of freeze-dried (FD) gel at different harvesting regimens.

2. Materials and methods

2.1. Chemicals

Double deionized water obtained from Milli-Q water purification system (Millipore) was used in all of the dilutions. The standard element solutions were prepared by serial dilution of multi-element standards obtained from Merck (Darmstadt, Germany). HNO_3, HCl and $HClO_4$, and other reagents were also procured from Merck (Darmstadt, Germany). All reagents and solvents were of analytical reagent grade.

2.2. Plant materials

The *A. vera* L. plants were identified by Prof. G.G. Maity, Taxonomist, University of Kalyani, West Bengal, India, and was grown in the Agricultural and Food Engineering Departmental farm of Indian Institute of Technology Kharagpur, India (Ray et al., 2012). *A. vera* saplings were sown in July–August (2007–2008), under a randomized block design with forty replications (Ray and Aswatha, 2013). The overall topography of Kharagpur (22.33° N, 87.32° E) is flat to undulating terrain with lateritic sandy-loamy soil; seasonal changes are evident in Kharagpur throughout the year (Rautaray et al., 2003). On an average, 70–90% relative humidity, about 1800 mm annual rainfall and the velocity of wind ranged at 10–25 km/h creates a favorable condition for the growth of different medicinal and industrial crops in Kharagpur (Ray et al., 2013).

2.3. A. vera gel harvesting from different growth periods of plants

After 24 ± 1, 36 ± 1 and 48 ± 1 months of growth, the leaves were harvested during the summer (April–May) and used for extraction of parenchymatous gel. For clarity, the different age group of plants was hereafter referred to as two, three and four-year-plants. To study the influence of seasons apart from summer, three-year-plants were used to extract the gel during the rainy (July–August) and winter (December–January) season. It is worthy to mention that selection of different growth periods was made keeping in view of our previous investigations (Ray and Aswatha, 2013; Ray et al., 2013). Healthy and fresh leaves having a length of 35–45 cm were harvested from the first whorl position of plants between 8.30 a.m. and 9 a.m., and transferred to the laboratory on ice. Harvested leaves were washed with double distilled water to remove the dirt, and filleted. The translucent foliar parenchyma was blended into a single composite gel and allowed to freeze overnight at −20 °C for solidification, which was followed by freeze-drying for a consecutive 72 h. Pale yellowish FD parenchymatous gel obtained from two, three and four-year-old aloe in summer were assigned as S2, S3 and S4, respectively. FD-gel prepared from three-year-old-plants in rainy and winter season were designated as R3 and W3, respectively. Drying by sunlight or by any other convective drying method has not been opted to avoid the possible interference by high temperature on nutrient mineralization. The FD-AG was used in all of the analyses to study different parameters.

2.4. Swelling capacity

Swelling capacity was measured as increased bed volume after equilibration in excess buffer (Femenia et al., 2003). FD-gel (100 mg) was weighed into a graduated conical glass tube with an excess of phosphate buffer and allowed to stand for 12–16 h for optimum swelling. The suspensions were stirred and allowed to settle down to diminish the void volume between the adjacent swollen gel layers. After equilibration the swelled volume was recorded and expressed as ml/100 mg of FD-gel (ml/100 mg).

2.5. Microstructural analysis with SEM, and detection of elements by SEM-EDS

FD-Gel samples were positioned on stub prior to gold sputtering. The raster scan profiles of FD-AG were captured by JEOL JSMS 5800 Scanning Electron Microscope (Japan) at 300× magnification under a pressure of 40 Pa and accelerating voltage of 20 kV. The characteristic spectrum of energy dispersive X-ray spectroscopy (EDS) was captured with Oxford EDS detector coupled with SEM instrument for the preliminary detection of the predominant elements present in FD-AG prior to the quantitative estimation.

2.6. XRD and ATR-FTIR spectroscopy

FD-gel from the potential growth period was analyzed with PW 1710 X-ray defractometer (Philips, Holland) with Co target and Fe