



Compressibility and dissolution characteristics of mixed fruit tablets made from guava and pitaya fruit powders



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ABSTRACT

This study reports the tableting of whole fruit powder from pitaya and guava and their dissolution in relation to use as drink's tablets. Pulps of both fruits with peels and seeds were freeze-dried into powders with addition of 10% maltodextrin. The fruit powders, individually as well as in a binary mixture (1:1), were analyzed for material properties and were found to be poor in flow. Among the three powders, guava powder attained the lowest density during compaction and exhibited as a poor compressible powder. Mixed fruit tablets containing 1% effervescent agent eroded quite fast in all three types of solvents studied. However, the acidic solvent (0.1 N HCl) was found to be not suitable for erosion of tablets containing polyvinylpyrrolidone (Kollidon CL). In terms of active ingredient release (antioxidant), Kollidon CL was found to be the best. In the case of color release (α^*), the faster the erosion, the better was the color intensity irrespective of dissolution media. As drink tablets, the mixture containing 10% sugar was highly preferred by majority of panelists (80%). A month long storage study with the mixed fruit tablet formulation at room temperature showed good microbial stability.

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1. Introduction

Production of tropical fruit powders is rapidly increasing in popularity among consumers as well as manufacturers due to their natural color, flavor, nutrition and taste. Fruit powders are taken either directly as drinks or used as secondary ingredients in baby foods, candies, fruit yogurt drinks and soups. There are various drying techniques used to prepare fruit powders and spray drying is the most convenient and most widely practiced in industry [1]. In many cases pulpy fruits need to be clarified. Technically it is quite impossible to convert pulpy fruits into powder by spray drying. This operation removes part of the pulp and coarse particles (which are rich sources of fiber and phenolic compounds) to enable proper functioning of the atomizer, [2]. Secondly, it is necessary to add quite high amounts of carriers to change the amorphous state by increasing glass transition temperature (T_g). Amorphous materials are frequently associated with the problem of stickiness or agglomeration due to the nature of the changing phase at the glass transition temperature (T_g) [3]. However, addition of additives eventually contributes to the increase in solid content as bulky materials, which adversely affects the sensory as well as nutritional properties. Due to the high sugar content of tropical fruits, high temperature drying by mechanical drier makes it sticky, and is thus difficult to convert into powder. Therefore, for producing whole fruit powder, drying fruits at low temperature and reduced pressure with low amounts of carrier (maltodextrin in our case) is apparently the best alternate [1]. For producing

whole fruit powder, freeze drying though costly, is the best option in relation to sensory and nutrition properties. Hence, in this study freeze drying was used to produce whole fruit powder.

In a typical pharmaceutical industry, compaction of powder is one of the important key operations in developing solid oral dosage forms. In the food industry applications of compaction are rather at the developing stages. However, candies and several types of food supplements produced by compaction are becoming popular in the retail market. Compaction of fruit powder into tablets is an excellent alternate for overcoming problems associated with post-processing handling, packaging and storage of fruit powder. Fruit powder in tablet form will be less hygroscopic due to reduction of surface area, which in turn facilitates in reducing packaging cost. Most importantly, transformation of fruit powder into compacted tablets will result in much reduced volumes and will be beneficial for increased shelf life, storage and transportation. The tablet form will also be convenient to use and affordable to consumers at all levels. Literature on the direct compression of whole fruit powder into tablets is scarce. Food tablets containing various food powders obtained from dates, spirulina and orange were investigated by Adiba et al. [4]. This report is mostly concerned with material properties and tablet dissolution behavior in different media. Satya Prakash et al. [5] investigated flowability, compressibility and in-vitro release of a laxative formulation of *Terminalia chebula* fruit powder tablets in relation to its use in herbal medicine. Compression behavior of a formulation containing about one third of the fruit powder along with other ingredients was reported. The major problem associated with tablet foods is the poor dissolution. Biopharmaceutical properties like dissolution and disintegration time are very important in the case of

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medicinal tablets or capsules. However, information on dissolution properties of formulated food tablets is lacking.

Pitaya and guava are two tropical fruits and are popular for their unique characteristics. Both the fruits are known to be high in nutritional properties. They are relatively high in antioxidant activity. Pitaya is a beautiful fruit containing water soluble betacyanins responsible for the intense red color and antioxidant activity [6]. Powder of this fruit has already been explored as a potential source of natural red colorant [7,8]. On the other hand, guava is well-known for its high vitamin C and fiber content [9]. However, the light greenish color of ripe fruits is lost during spray drying, which is quite usual [10]. Hence, a new tablet formulation blending the powders of both fruits to compensate for the color as well as the nutrition imbalance may result in a new product which is highly acceptable to consumers.

The present study was therefore designed to prepare (a) a composite formulation using whole fruits freeze-dried powder of pitaya and guava compressed into tablet form, (b) evaluate in-vitro release of its active ingredients, and (c) assess its suitability as a natural fruit drink tablet.

2. Materials and methods

2.1. Raw materials

Red pitaya and guava were purchased from the market in Sri Kembangan, Selangor, Malaysia. To minimize variation in fruit composition due to source and maturity, they were purchased from the same lot. Fruits are washed and mashed with a pulper without removing the skins and seeds in order to maintain the 'whole fruit'. Proximate analysis of pulp for moisture content, fat, crude protein, crude fiber, ash water activity and vitamin C were carried out, and samples were stored at $-20\text{ }^{\circ}\text{C}$ until further used.

2.2. Freeze drying of whole fruit pulp

Powder obtained by freeze drying of fruit pulp was found to be very hygroscopic and difficult to compact. To minimize this problem 10% maltodextrin was added to fruit mash, mixed by homogenization at 10,000 rpm for 10 min before freeze drying. Suspension of both fruits was then poured into a few rectangular plastic containers separately and covered with lids. The contents in the rectangle containers were frozen by storing in a freezer at $-20\text{ }^{\circ}\text{C}$ for two days. After freezing, the samples were transferred to a vacuum freeze dryer (Ben Hay, United Kingdom) and dried at $-35\text{ }^{\circ}\text{C}$ for 48 h at 0.25 MPa pressure. The pitaya and guava powders were obtained by crushing the dried material in the sealed plastic bags separately and stored in a refrigerator ($4\text{ }^{\circ}\text{C}$) until used in the experiments.

2.3. Powder properties

The material properties including moisture, water activity, densities and particle size of both powders were determined by the method described in Ng et al. [8]. Moisture was determined by oven drying in vacuum at $70\text{ }^{\circ}\text{C}$ for 24 h. Water activity of the powder was measured using the water activity meter (GDX Instrumentation Scientifique, France). Different types of densities including absolute, tap and bulk densities of powders were measured. The values of bulk density and tap density were used to calculate the Hausner ratio [11] and Carr index [12]. The Hausner ratio and Carr index were used to calculate flowability. A gas pycnometer (AccuPyc II 1340, Micromeritics, Norcross, USA) was used to determine the absolute density. The particle size of the guava and the pitaya fruit powder was measured using a particle size analyzer (Malvern Mastersizer 2000, Malvern Instrument Ltd, UK). A Scanning Electron Microscope (Philips XL 30 ESEM; MEMS and Nanotechnology Exchange, Virginia) was used to analyze the particle size and shape of the powders. Samples for the SEM were prepared first with Karnovsky's

fixative, taken through an alcohol dehydration series, placed in a critical point dryer, mounted and located in a gold coater and finally images were scanned on a digital imaging system.

2.4. Compression of fruit powder

Uniaxial compaction of pitaya, guava and the mixed (1:1) powder was carried out by using an Instron Universal Testing 5566 Machine (Canton MA, USA). For mixed tablets, equal amounts of both powders were mixed manually. Tablets were formed within a cylindrical die cast made of stainless steel having a diameter of 20 mm. Two gram (± 0.001) of powder weighed with a digital balance was compacted to various ultimate applied stresses within the die. The applied force and the cross-head displacement were recorded by computer software. All tablets were compacted by an ultimate force ranging from 1, 3, 5, 7, and 9 kN. The compaction of the powder was performed following the method described by Yusof et al. [7]. The procedure was followed by unloading and removing of the bottom punch and finally the tablet was ejected from the die. The thickness of tablets was measured in order to calculate density of the tablet. The tensile strength was determined by placing the tablet between two flatten plates and applying force using the Instron Universal Testing 5566 Machine at the speed of 10 mm/min with the force of 9.8 kN until a clear crack on the compacted tablet emerged. The tensile strength was calculated with the following formula [13].

$$\sigma_t = \frac{2F}{\pi DH} \quad (1)$$

where, F = the crushing force or tensile force (N), D = compact diameter (m), H = compact thickness (m).

2.5. Dissolution of fruit tablets

Dissolution of the mixed fruit tablets was carried out by observing kinetics of erosion of tablets in three different solvents as described in Adiba et al. [4]. Distilled water, 0.1 N hydrochloric acid solution and citrate buffer of pH 4 were used as solvents. Three mixed fruit tablet formulations, first having no disintegrant agent (NSD), second with 1% effervescent agent (EFA) (equal ratio of sodium bicarbonate and citric acid) and third containing 1% Kollindon CL (KCL; polyvinylpyrrolidone, E-1202) were used to observe the erosion kinetics. Erosion test was carried out under controlled temperature ($37\text{ }^{\circ}\text{C}$) till dissolution was completed. The percentage of erosion was calculated using the following equation:

$$\% \text{Erosion} = \frac{\text{Weight of tablet before immersion} - \text{Weight of tablet after immersion}}{\text{Weight of tablet before immersion}} \times 100\%. \quad (2)$$

In vitro dissolution of composite fruit tablets was carried out using a dissolution tester (PT-DT8, Germany) on six tablets from each tablet formulation in the above three solutions at $37\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$ and 50 rpm. About 500 mL liquid medium was poured into the dissolution beaker and 6 tablets were put simultaneously inside the medium. To observe the kinetic release of active ingredients (vitamin C & color), 50 mL liquid was withdrawn from each sample at 10, 20, 30, and 40 min intervals and replaced with an equal amount of fresh dissolution medium. The liquid was filtered through a filter paper (Whatman No. 1, $0.45\text{ }\mu\text{m}$; Toyo Roshi Kaisha Ltd, Japan) and kept in 50 mL centrifugal tubes for further analysis. The dissolution tester was stopped when all six tablets were completely dissolved in the medium. The time for complete dissolution was recorded using a stopwatch. Kinetic release of vitamin C and the changes in antioxidant and color as influenced by disintegrant agents and solvents during dissolution were assessed. Antioxidant activity was determined using the 2,2-diphenyl-1-picryl hydrazyl

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