



## Research Paper

## Effect of a new shell material—Jackfruit seed starch on novel flavor microcapsules containing vanilla oil

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## ABSTRACT

Vanilla essential oil was microencapsulated by different shell materials including jackfruit seed starch (JM), chitosan (CM), and  $\beta$ -cyclodextrin ( $\beta$ M) based on the ultrasonic method. The effects were compared among the different shell materials when the yield, encapsulation efficiency, storage stability and slow-releasing potential were used as evaluation indexes. The results showed that the yield of  $\beta$ M and CM were 87.99% and 86.35%, on the other hand, the encapsulation efficiency (EE) of these two microcapsules were 77.92% and 76.64%, respectively. While the yield and EE of JM was 84.82% and 79.33%, respectively. The microcapsule surface formed smooth membrane structure. At the same time, the results of peroxide value and the aroma intensity also demonstrated that JM had good storage stability and slow-releasing potential so that its shelf life could reach 250 days. By contrast, the shelf life of  $\beta$ M and CM only reached to 160 days. The EE, storage stability and slow-releasing potential of JM were more excellent than those of  $\beta$ M and CM, although the yield of JM was not outstanding. All these results indicated that the jackfruit seed starch showed its tremendous potential to be a good shell material of microcapsule and be well used in the food industrial.

## 1. Introduction

Vanilla (*Vanilla planifolia* Andrews) is a valuable natural flavor plant grown in tropical and subtropical areas. As known for 'Flavor Queen', vanilla is one of the most commonly used flavorings in confectionery, food products, beverages, ice cream, perfume, cigarettes, and pharmaceutical products in the world (Dong et al., 2014). Vanilla oil was extracted from the vanilla pods. However, its applications were limited when the effective components of vanilla oil were easily influenced by factors of air, light, moisture and high temperature due to volatility and instability. Taking many factors into considerations, the technology of microcapsule was good at protecting core materials from environmental factors. Therefore, vanilla oil could be extensively used in food processing industry by the method of microcapsule, which could restrict its aroma from degradation or loss from processing and storage (Yang et al., 2014).

Selection of shell materials should meet the national standards of food additives which required they should not react with core materials, and non-toxic, good film-forming, liquidity and low moisture absorption (Anjani et al., 2007). As the most commonly used materials, natural materials is one of the most important microcapsule shell materials which have been applied successfully for the process of

microencapsulation. The most important and primary advantages of the products were its low immunogenicity, good biocompatibility, biodegradable and the non-toxic side effects (Li et al., 2015). Natural materials were composed of two categories in which one was proteins and the other was carbohydrates. The diverse hydrophilic and lipophilic groups or mission area of the proteins made them a key role as emulsifier in the emulsion system to reduce the interfacial tension of oil phase and water phase and formed a stable water-in-oil or oil-in-water system. (Avramenko et al., 2016; Akhtar and Dickinson, 2003). Materials such as starch, maltodextrin, small molecule carbohydrate, chitosan, dry powder of starch syrup are generally used for the production of powder grease as carbohydrates shell materials for its good solubility, and the maintaining of a low viscosity when the solid content is high. Being different from other carbohydrates, starches have interface features with high efficiency of microcapsule and could be microencapsulated with no use of other colloid and protein (Błaszczak et al., 2013).

Jackfruit (*Artocarpus heterophyllus* Lam.) belonged to the family of Moraceae and was widely grown in tropical countries (Madruga et al., 2014). According to the documents, it was introduced to China thousand years ago, and now, they are planted in Hainan, Guangdong, Guangxi and Yunnan provinces of the tropical and subtropical districts

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(Zhang et al., 2016; Xu et al., 2015). At present, the area of its plantation is nearly 15 thousand hectares (Wu et al., 2013). Jackfruit seed is rich in starch which accounts for approximately 60%–80% of its dry weight. That means the Jackfruit seed has tremendous potential to be developed and utilized as a resource of starch. However, it is usually discarded as waste except sometimes they are boiled or roasted as a snack (Zhang et al., 2016; Mahanta and Kalita, 2015). As the previous results showed that there were some advantages such as small particle size, high solubility, strong water and oil absorption, low viscosity, thermal stability of paste and other properties on seed starch (Saviane and Silver 2011; Ocloo et al., 2014). These properties will contribute to load core materials and retain physicochemical properties of these materials. Therefore, one of the best ways to utilize the seeds is to extract the starch due to their high starch concentration and use in different industries based on its suitability. However, so far, there were no studies that jackfruit seed starch was employed in microcapsule as shell material. In addition, it has not been reported on the preparation of vanilla oil microcapsule by jackfruit seed starch.

Therefore, the aim of this study was to evaluate the potential of the jackfruit seed starch as a novel shell material. The ability of different shell materials (jackfruit seed starch, chitosan and  $\beta$ -cyclodextrin) were evaluated by detecting the yield, encapsulation efficiency, storage stability and slow-releasing potential of the microcapsule. The investigation was to provide a new shell material for the microcapsule of vanilla essential oil and find a new way for the reasonable utilize of the jackfruit seed starch.

## 2. Materials and methods

### 2.1. Materials

Jackfruit cultivars were harvested at July 2015 from the garden of Spice and Beverage Research Institute (Hainan, China) and seeds were obtained from bulbs correspondingly; The jackfruit seed starch was extracted by the method of Zhang et al. (Zhang et al., 2016); Vanilla pods were harvested at November 2015 from the garden of Spice and Beverage Research Institute (Hainan, China), and vanilla essential oil were extracted by the supercritical CO<sub>2</sub> technology (SFE-2, ASI Corporation, America);  $\beta$ -cyclodextrin and chitosan were purchased from AK Biotech Ltd. (Jinan, China). All other chemicals and reagents arose out of analytical grade.

### 2.2. Preparation of microcapsules

The microcapsule was prepared according to the method of Chatterjee and Judeh with some slight modifications (Chatterjee and Judeh, 2015). Vanilla essential oil was dissolved in a supersaturated solution prepared by the shell materials. Then the mixture was managed for 20 min by ultrasonic cell crushing (JY92-2D, Xingzhi of Ningbo Inc, China). The mixed solution was put into the water bath (HHS-8S, Yichang Instrument Factory of Shanghai, China) for 15 min and then was refrigerated for 24 h under 4 °C in the refrigerator (MeiLing science and Technology Co., Ltd, Hefei, China) when the solution was cooled to the room temperature. After filtration, samples were put into the vacuum freeze dryer (18ND, Xingzhi of Ningbo Inc, China) and were dried to constant weight. The seed starch and  $\beta$ -cyclodextrin shell solutions were dissolved with distilled water, while chitosan solution was dissolved with 1% acetic acid solution, and the ratio of shell weight to the essential oils was 6:1. The liquid samples were transferred to a standard plastic cup and frozen at –18 °C, and then were performed by freezing for 4 h at –20 °C, thawing for 24 h at 30 °C until the weight kept constant.

#### 2.2.1. The yield and the encapsulation efficiency of microcapsule

The yield of microcapsule was estimated by the mass of microcapsule powder between the shell material and the core material. And

the EE of microcapsule was estimated by the ratio of encased essential oil to total oil (Xiao et al., 2011) as follows:

$$EE\% = \frac{X-X_1}{X} \times 100\%$$

Where X (g) and X<sub>1</sub> (g) were the weight of total oil and surface oil.

Surface oil was extracted with petroleum ether as follows. 10 g microcapsules were put into a 250 mL conical flask and added 50 mL petroleum ether. It was managed for 20 min by ultrasonic oscillation machine. The solution was then filtered into a constant weight flask and vaporized to keep constant weight again. The surface oil (X<sub>1</sub>) was calculated in accordance with the following formula:

$$X_1\% = \frac{m}{M \times A} \times 100\%$$

Where m (g) was the constant weight of oil, M (g) was the weight of microcapsule powder, and A (g) was the weight of essential oil microcapsule powder.

Total oil content was measured by using alkaline ether extraction method. Briefly, 1.25 mL ammonia solution was added into 5 g test portion and the mixture was thoroughly incorporated. The mixture was then added with 10 mL of ethanol and then vibrated for 2 min to be fully blended followed by being kept in a water bath at 60 °C for 5 min. The resulting solution was cooled in cold water and 25 mL ether was added into the solution followed by being vibrated for 0.5 min. Subsequently, 25 mL of petroleum ether was then added into the final mixture and kept in static state for 30 min after shaken for 0.5 min. The organic layer was collected after repeat the extraction 2–3 times. Finally, the content of total oil was calculated as follows:

$$X\% = \frac{(m_1 - m_0) \times 100}{m_2 \times \frac{v_1}{v_0}}$$

where X (g/100 g) was the weight of fat in the test samples, m<sub>1</sub> (g) was the weight of bottle plus fat, m<sub>0</sub> (g) was the weight of bottle, m<sub>2</sub> (g) was weight of sample, v<sub>0</sub> (ml) was the total volume of ether layer, v<sub>1</sub> (ml) was the volume of pipetted ether layer, and calculation results keep two significant figures.

#### 2.2.2. Microscope observation

The morphology of microcapsules was examined by optical microscope (Olympus BX51, Olympus Corporation, Tokyo, Japan). Microcapsules were dissolved with distilled water (1:10, microcapsule to distil water), then the mixture was placed on a glass slide and observed by microscope (100×).

#### 2.2.3. Analysis of microcapsule storage stability

Essential oil microcapsules and samples were placed in a 60 °C oven and the peroxide value which was chosen as the index to reflect the storage stability was measured every 3 days. The peroxide value was detected by the method described by Zhang et al. (Zhang et al., 2015) and AOAC Official Method 965.33 (2015) with some modifications. 4 g microcapsules fully grounded with 50 mL chloroform solution. The filtered solution conducted by chloroform and ice acetic acid, and then added with 1.00 mL saturated potassium iodide solution followed by lightly shaking. Afterwards, 100 mL water was added. Next, the samples were titrated with sodium thiosulfate standard titration solution until the mixture turned light yellow. Finally, 1 mL starch indicator liquid was added into the solution, keep shaking and titrating until the blue just disappeared. At the same time, the blank experiment was carried out by the same method. To calculate peroxide value through the following formula, the results were the average value of three repeated tests.

$$X = \frac{(V_1 - V_2) \times c \times 0.1269}{M} \times 100 \times 78.8 \times 2$$

where X (mmol/kg) was peroxide value, V<sub>1</sub> (mL) and V<sub>2</sub> (mL) were the

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