Journal of Food Engineering 153 (2015) 1–7

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Sugar alcohols – Multifunctional agents in the freeze casting process of foods

P.T.N. Nguyen *, J. Ulrich

Martin Luther University Halle-Wittenberg, Center for Engineering Science, Thermal Process Engineering, D-06099 Halle (Saale), Germany

ARTICLE INFO

Article history:
Received 9 October 2014
Received in revised form 29 November 2014
Accepted 3 December 2014
Available online 12 December 2014

Keywords:
Fast dispersal tablet
Freeze casting
Binder
Sugar alcohol

ABSTRACT

The successful production of a new convenient dosage form, namely, a fast and easily dissolving/dispersal tablet was presented in a previous study (Nguyen and Ulrich, 2014). The highlight and interesting point of that work is the introduction of sugar as a binder which dramatically improved both the tensile strength and dispersal behavior of produced tablets. In this work, the investigation on the effect of sugar alcohols (i.e., isomalt and xylitol) was carried out in order to find the mechanism on how the binders (sugar alcohols) change the properties of the tablets. The effects of sugar, isomalt and xylitol on the key properties of the tablets (i.e., the tensile strength, the dispersal/dissolution behavior), the morphology of pores and tablets, especial the thermodynamic properties of aqueous suspensions were investigated. It was found that sugar alcohols depress the freezing points of aqueous suspensions, reduce the ice growth rate and induce to form a new pore morphology, a sphere-like porous solid body. Isomalt as binder improves the properties of the produced tablets to a higher degree than sugar and xylitol. A fast dispersal cocoa tablet (16 mm ∅, 10 mm height) with a fast dispersal time of lower than 1 min and a better crushing force of 45 N (i.e. 0.18 N/mm^2 diametral tensile strength) was obtained.

1. Introduction

Freeze casting is a well known cold compression technology which has been widely applied to produce porous materials, e.g. porous green bodies in ceramic industry (Donchev, 2005; Deville, 2008; Li et al., 2012). By freezing an aqueous liquid suspension of the relevant substance, a cold solidification and pressing is carried out. Followed by a solvent removal step (an evaporation or sublimation), a porous solid body in the desired mold form is obtained without using any external pressure (Nguyen and Ulrich, 2014). Non external pressure and low processing temperature are advantages which make freeze casting a promising technique for compacting of heat and pressure sensitive materials, especial in pharmaceuticals (Pachuski and Ulrich, 2007a,b; Szepes et al., 2007a,b; Witte and Ulrich, 2010). In addition, a high porosity leads to a high contact surface area and enhance a dissolution/dispersal rate of a solid dosage form. Such properties are very useful for fast dispersal/dissolving products which are of high interests in food industry e.g. instant foods and in pharmaceutical industry due to drug delivery issues. In term of a fast dispersal/dissolving tablet, a high dissolution/dispersal rate is the first order of requirement. However, the mechanical strength of tablets also needs to be high enough to be handled and to be stable under processing. In the freeze casting process, the porosity of produced tablets is linked reciprocal to their tensile strength. A tablet owning a higher porosity may have a better dissolution rate, however, a high porous structure also induces a lower mechanical stability and vice versa. In order to get the balance of both the tensile strength and the porosity (or the dissolution/dispersal rate), a binder which is highly soluble in the dispersal medium is strongly recommended (Szepes et al., 2007a,b; Pachuski and Ulrich, 2007a,b; Witte and Ulrich, 2010).

Our previous study (Nguyen and Ulrich, 2014) proved that an addition of sugar (sucrose) in the aqueous cocoa suspension successfully improved the mechanical strength and the dissolution/dispersal behavior of produced cocoa tablets by the freeze casting process. A fourfold improvement (decreased) of the dispersal time and a twofold increased tensile strength were found when sugar was added into the aqueous phase with ratio of 20 g sugar/100 g water. As result, a fast dissolving cocoa tablet (16 mm ∅, 10 mm height) with a dispersal time of less than 1 min and a crushing force of 34.5 N (i.e. 0.14 N/mm^2 diametral tensile strength) was obtained.

This study is aimed to find how the binders (here are sugar alcohols) change the properties of tablets. The effects of sugar alcohols (including sugar, isomalt and xylitol) on the ice crystallization as

* Corresponding author.
E-mail addresses: thi.nguyen@iw.uni-halle.de (P.T.N. Nguyen), joachim.ulrich@iw.uni-halle.de (J. Ulrich).

http://dx.doi.org/10.1016/j.jfoodeng.2014.12.007
0260-8774/

well as the physical properties of produced cocoa tablets (pore morphologies, dissolution/dispersal behaviors and tensile strengths) in a freeze casting process were investigated.

2. Materials and methods

2.1. Materials

Sugar (sucrose), xylitol (⩾98.5%, Carl Roth GmbH + Co. KG) and isomalt (Cargill GmbH, Germany) were used as binders. Cocoa powder (Gepa The Fair Trade Co., Germany) with particle sizes smaller than 160 μm was chosen as a food candidate. The cocoa powder is the highly defatted cocoa with the cocoa butter content of 10–12 wt% and K_2CO_3 as impurities. Distilled water was used as a solution base (or dispersing medium).

2.2. Freeze casting procedure

To produce tablets an experimental setup of the freeze casting process was applied (see Fig. 1). This process includes four main steps: (1) suspension preparation using mixing; (2) casting in the preferred form mold; (3) solidification using freezing at low temperature at ambient pressure; (4) solvent removing by sublimation under reduced pressure (vacuum). The detail descriptions of the freeze casting process were presented in the previous paper (Nguyen and Ulrich, 2014).

Here, the effects of different binders, e.g. sugar alcohols including sugar, isomalt and xylitol, in the defined concentrations were investigated. The suspension was prepared by mixing cocoa powder in an aqueous alcohol sugar solution. The solid loading of suspensions was kept constant of 28 g cocoa powder per 100 g distilled water. Sugar alcohols (sugar, isomalt and xylitol) contents in aqueous solution was raised from 0 g to 20 g solute per 100 g distilled water. The suspensions were frozen in 4 h under temperature of −24 °C, then ice crystals were removed by sublimating in 20 h under temperature of −8.1 °C and reduced vacuum (Nguyen and Ulrich, 2014).

The quality of produced tablets was evaluated though their dispersal behavior, mechanical strength as well as morphology of pores.

2.3. Characterization of aqueous suspensions

Rheological characterization of the aqueous suspensions was performed by using a rotational viscosimeter ViscoTester 550 (VT550 DIN/ISO Cylinder Package, HAAKE). All measurements were carried out with a stainless steel coaxial cylinder measuring system at 20.0 ± 0.1 °C with a shear rate of 200 s^{-1}. Three parallel measurements were carried out.

Differential Scanning Calorimetry (DSC) measurements were carried out by a DSC 12E, Mettler Toledo instrument in order to determine the freezing and melting points of the suspensions. Approximately 17 mg of suspension sample were weighed out in a 25 μL aluminum crucible. One cooling and one heating period in temperature range from 20 °C to −30 °C was carried out with a cooling/heating rate of 1 K/min at a nitrogen flow of 30 mL/min.

Krüss K10T was used to determine the surface tension of the suspension at the room temperature of 25 ± 1 °C.

2.4. Characterization of freeze-casted tablets

A digital microscopic system (VH-Z100, Keyence) was used to record the overview of morphology of the pores and tablets. The structures of freeze-casted bodies were studied by using a scanning electron microscope (Philips XL 30 ESEM). To get better SEM images, samples for scanning microscopy examination were mounted and coated with platinum in a sputter coater before SEM measurement.

The crushing strengths of produced tablets (∅ ∼ 16 mm, $h \sim 10$ mm) were measured with crushing force tester (TBH 30, Erweka), then the tensile strengths were calculated from these data and the geometrical parameters of tablets (Mitutoyo, Japan) via the equation of Fell and Newton (Fell and Newton, 1970).

Porosities of tablets were determined by mercury porosimeter Pascal 140 + 440 Series, Porotec. Dissolution profiles of tablets were determined by a simple visible method. These methods were presented in detail by Nguyen and Ulrich, 2014.

The measurements of crushing force are reproduced six times and all the other measurements were carried out in triplicate.

3. Results and discussion

3.1. Characterization of freeze-casted tablets

For the fast dissolving tablets, in order to get an improvement in both the tensile strength and dispersal behavior, binders which are highly and fast soluble in the dispersal medium are strongly recommend (Szepes et al., 2007a,b; Pachuski and Ulrich, 2007a,b; Witte and Ulrich, 2010; Nguyen and Ulrich, 2014). In addition, the binders also need to interact well with the main active ingredients in both solid and liquid state to get a stable suspension during the sample preparation step as well as to form a binding to reinforce the tablet body during the freeze casting process. In order to classify the potential binder groups, a simple screening of binders (i.e., sugar alcohols, acidic food additives) based on their solubility and dissolution rate in water, and the stability of the suspensions were investigated. As results three promising substances: sugar (sucrose), isomalt and xylitol were chosen as binders for the present study.

The tablets produced by the freeze casting process with a one side and a two-side freezing mode of the aqueous suspensions resulting by mixing of cocoa powder (at a cocoa content ratio of

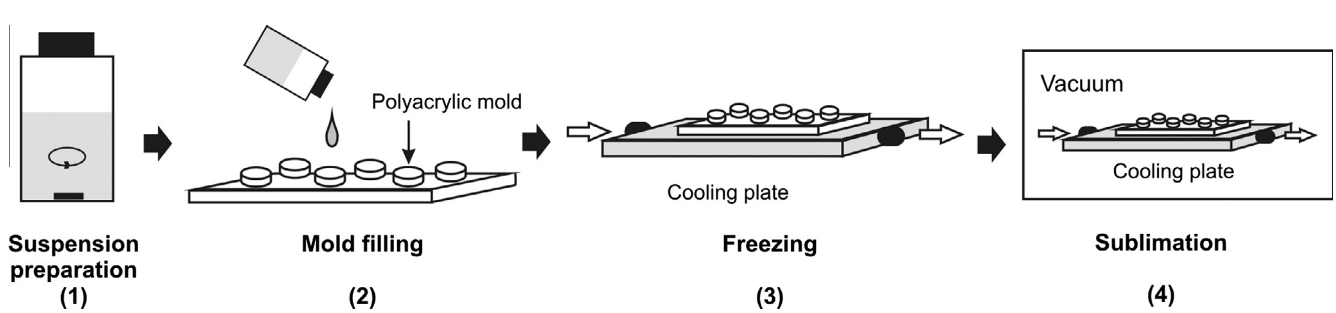

Fig. 1. Experimental setup of the freeze casting process.