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An investigation on dissolution kinetics of single sodium carbonate particle with image analysis method

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ABSTRACT

Dissolution kinetics of sodium carbonate is investigated with the image analysis method at the approach of single particle. The dissolution experiments are carried out in an aqueous solution under a series of controlled temperature and pH. The selected sodium carbonate particles are all spherical with the same mass and diameter. The dissolution process is quantified with the measurement of particle diameter from dissolution images. The concentration of dissolved sodium carbonate in solvent is calculated with the measured diameter of particle. Both surface reaction model and mass transport model are implemented to determine the dissolution mechanism and quantify the dissolution rate constant at each experimental condition. According to the fitting results with both two models, it is clarified that the dissolution process at the increasing temperature is controlled by the mass transport of dissolved sodium carbonate travelling from particle surface into solvent. The dissolution process at the increasing pH is controlled by the chemical reaction on particle surface. Furthermore, the dissolution rate constant for each single spherical sodium carbonate particle is quantified and the results show that the dissolution rate constant of single spherical sodium carbonate increases significantly with the rising of temperature, but decreases with the increasing of pH conversely.

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1. Introduction

Dissolution of solid particles has been studied over one century and it is widely applied into various chemical and physical applications such as food industry, pharmaceutical and household product. A typical dissolution is generally the process of solute forming a solution in solvent. Three main dissolution models are thus developed including mass transport model, surface reaction model and inter-facial barrier model [1]. Based on these dissolution models [2], a vast number of mathematical models were proposed, such as Noyes-Whitney equation [3] and Hixson-Crowell model [4], and used to quantify the dissolution kinetics at different dissolving conditions.

Traditional dissolution studies mainly rely on the dissolution experiments such as U.S. Pharmacopeia (USP) dissolution apparatus [5]. The dissolution process usually is measured with conductivity or refractometry measurements. Dissolution mechanisms can be interpreted with the existing dissolution models. Although conventional research is normally based on the dissolution tests of bulk of particles, many dissolution studies with the approach of single particle have been raised and implemented into several materials in the recent 20 years. For example Dorozhkin quantified the dissolution kinetics of single crystal

of natural fluorapatite with optical microscopy, SEM, Auger electron and IR reflection spectroscopy [6]. Raghavan *et al.* quantified the dissolution kinetics of single crystal of α -lactose monohydrate, and linear dissolution profiles were determined as a function of time [7]. Štěpánek simulated the effective dissolution according to the local fluxes as the solution of a convection–diffusion problem in the surrounding liquid phase [8]. However, it is rarely investigated for sodium carbonate which is one of the most important basic compositions of many products such as detergent and baking powders with the approach of single particle to study the dissolution kinetics. The dissolution properties of sodium carbonate show a large extend to affect the quality of final products. Therefore, this work is highly motivated to overcome this vacancy of dissolution studies for sodium carbonate with the approach of single particle to determine its dissolution mechanism at different conditions and quantify its dissolution kinetics. The dissolution conditions that affect dissolution kinetics of solid particles generally based on the physical and chemical properties of solution [9–10]. In this work, the dissolution experiments are carried out with increasing temperature and pH separately. Therefore, the dissolution mechanism of single sodium carbonate particle at increasing temperature and pH can be investigated and the dissolution kinetics are also quantified. In this way, the works in this paper will also provide vast information to extend the understanding of dissolution properties of sodium carbonate from the approach of single particle to bulk.

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The dissolution process of single particle is normally excessively fast and the entire dissolution environment changes within a very small scale, it therefore is very difficult to measure the dissolution process directly with the measurement techniques which are usually applied for a bulk of particles. To accomplish the dissolution experiments of single sodium carbonate particle in this work, the image analysis method *via* high speed camera is implemented. Different with image analysis with microscopy, high speed camera has its great advances to transfer the entire dissolution process into visualized videos and each frame of video can be saved into dissolution images for analysis. The time scale of saved images can be accurate to 1/10, 1/20 or 1/50 s. In this way, it is reliable to apply image analysis method accurately into quantification of dissolution process of single sodium carbonate particle.

2. Sample Selection

The original sodium carbonate particles used in this work are provided by Procter & Gamble Newcastle Innovation Center (UK). The purity of sodium carbonate particles is 99.9%. The samples of sodium carbonate particle used into dissolution experiments are selected with the following procedures: firstly, the original sodium carbonate particles provided by Procter & Gamble are weighed for one particle by another in order to select the particles containing the mass of (0.30 ± 0.02) mg. Then the selected particles from with same mass are observed with an optical microscopy to define their shape and only the particles having the shape of sphere are selected. The definition of particle shape in this work is based on the classification scheme by Riley [11]. According to Riley's definition, the particles having the sphericity higher than 0.775 can be considered as the high spherical particles where the sphericity is measured as the ratio of the diameter of a circle having the same area of measured particle to the diameter of the smallest circumscribed circle of measured particles. In this way, the optical microscopy of Nikon Eclipse Ti Microscopy is used in this step to measure the sphericity and the particles with the sphericity higher than 0.775 are selected as the spherical sodium carbonate particles. Finally, the diameter of spherical sodium carbonate particles is measured with this optical microscopy. Only the spherical particles having (1.50 ± 0.02) mm diameter which is measured from its smallest circumscribed circle are selected as the samples used into the dissolution experiments. An example of the sample of spherical sodium carbonate particle was shown in Fig. 1.

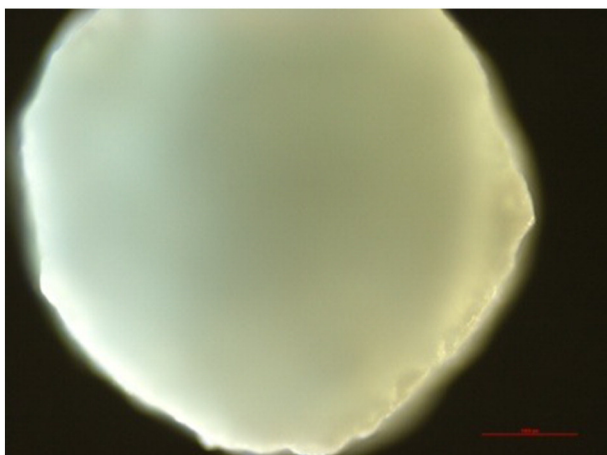


Fig. 1. Spherical sodium carbonate particle into dissolution experiment.

3. Experimental methodology

The scheme of a typical dissolution experiment in this work is shown in Fig. 2. For a typical dissolution experiment, one sample of spherical

sodium carbonate particle is placed at the bottom of a transparent 12 mm square glass cell. The transparent 12 mm square glass cell used in this work is 12 mm glass cell (PCS115) purchased from Malvern Tec. The high-speed camera of Photron Fastcam SA5 is set-up with the frame of 50 FPS and resolution of 640×640 to record the dissolution process of this sample. When the high-speed camera starts to record, the cell is immediately filled with 0.2 ml solution. In this way, the dissolution process of the sample is recorded as a video and the video also is transferred into 50 images per second automatically. These images are then used to measure the diameter of particle with dissolution time. Because the density of sodium carbonate particles used in this work is assumed to be a constant, the concentration of dissolved sodium carbonate is calculated based on the measured diameter values. In this way, the dissolution rate constant is thus quantified based on the calculated concentration of sodium carbonate by fitting with the mathematical model for each case.

The solvent used in dissolution experiments is deionized water and it is prepared in advance to obtain required temperature and pH. To obtain different temperature, the deionized water is pre-heated to 30 °C, 40 °C, 50 °C, 60 °C and 70 °C respectively. Because the dissolution process is very fast and the temperature drops less than 1.5 °C according to the test, it is unnecessary to heat the cell during the dissolution process and each temperature used for dissolution experiment can be considered as a constant. Besides the deionized water with different temperature, the deionized water is also prepared by mixing with standard pH buffer solution to obtain different pH. The standard pH buffer solutions used in this work are PHA-4 and PHA-10 buffer purchased from OMEGA. After adjusting the amount of pH buffer solution, the deionized water can show as different pH as 4, 7, 8, 9 and 10 respectively. These prepared deionized water with different pH are also used for the dissolution experiments in this work.

4. Mathematical Model

4.1. Surface reaction model

Surface reaction model assumes that the dissolution rate is proportional to the particle surface area and the dissolution process is not controlled by the mass transport of dissolved solute but rather depended on the chemical reaction on particle surface. In this way the chemical reaction on the particle surface can be considered as the limiting step of the dissolution process [12].

Normally the dissolution rate is expressed as Eq. (1).

$$\frac{dC}{dt}V = K_r A \quad (1)$$

where K_r is the dissolution rate constant of surface reaction model, A is the surface area of spherical particle, C is the concentration of dissolved solute.

Because the shape and density of sodium carbonate particle is assumed as the constant during the dissolution process, the real-time surface area of the particle can be calculated with Eq. (2).

$$A = A_0 \left(\frac{M}{M_0} \right)^{2/3} \quad (2)$$

where A_0 is the initial surface area of particle, M_0 is the initial mass of particle and M is the mass of undissolved particle.

As the density of sodium carbonate is a constant in this work, the mass of undissolved particle is thus converted with the real-time concentration and initial concentration of sodium carbonate in solvent which is shown as Eq. (3).

$$M = M_0 - (C - C_0)V \quad (3)$$

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