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Chemical Engineering Research and Design

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# Assembled-magnetite membrane for recovery of starch granules in an open flow

Yushi Uno, Shintaro Morisada, Keisuke Ohto, Hidetaka Kawakita\*

Department of Applied Chemistry, Saga University, Saga 840-8502, Japan

## ABSTRACT

Magnetite was set in a channel as an open system and was placed there by application of a magnetic field to form an assembled-magnetite membrane. A starch-granules-dispersed solution flowed from the top of the channel to allow recovery of starch granules by the assembled-magnetite membrane by size effect. The size of starch granules dispersed was ranged from 1 to 60  $\mu\text{m}$ . The magnetite density in the membrane was strongly related to the recovery efficiency. To alter the density of the assembled-magnetite membrane, the amount of magnetite in the channel ranged from 0.05 to 0.5 g. With increasing membrane density, the recovery efficiency increased. After recovery by the membrane, the flown starch granules through the membrane were observed by optical microscopy, and were found to be less than 10  $\mu\text{m}$  in diameter. The proposed assembled-magnetite membrane has the potential for recovery of starch granules and other food particles used for cooking based on molecular gastronomy.

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**Keywords:** Magnetite; Assemble; Membrane; Starch granule; Gastronomy

## 1. Introduction

Membrane engineering has been utilized for the separation of biomacromolecules, proteins and DNAs, microorganisms, and colloidal particles. Control of the membrane pore size provides the means to separate various molecules and colloidal particles based on their size. In preparation of membranes, which may be composed of polymeric or inorganic compounds, the character and molecular weight of the membrane material are crucial factors in determining the separation and recovery efficiency. Once the membrane is prepared, it will have a pore-size range that allows the separation of particular analytes.

Magnetite is a ferromagnetic mineral that can be moved freely by application of a magnetic field. With chemical modification to the surface of magnetite, proteins and microorganisms have been adsorbed or separated (Eichholz et al., 2008; Honda et al., 1999; Kekkonen et al., 2009). Magnetite can also separate colloidal particles without the use of filtration. To increase the separation efficiency of magnetite, the surface area of the material is increased by lowering the particle size to the nanoscale level. The structure of the magnetite formed by use of a magnet is dependent on the magnetic flux,

the strength of the magnet, and the distance between the magnet and the magnetite. Such structural changes obtained in magnetite have been mainly studied from a physical perspective (Sano and Doi, 1983; Dubowik, 1996; Hanns and Andreas, 1999).

Starch granules, composed of amylopectin and amylose (Serge and Eric, 2010), are widely used in food engineering. The size of starch granules is generally in the range of 1–60  $\mu\text{m}$  (Ikeda et al., 2005), with the size and shape of the granules being dependent on the origin and the condition of the solution (Paul, 2001). The size separation and recovery of starch granules has been performed using membranes (Paul and James, 2012), centrifugation (Mahsa et al., 2003), and field-flow separation (Moon and Giddings, 1993). Each method is based on the size and density of the various starches. In each case, one kind of separation material is used for the separation of a certain size of starch granules. However, the use of one material that incorporates the various separation modes enables near full recovery of the starch types from different origins because of the diversity of its separation behavior. In food engineering, appropriate treatment of starch in cooking enhances the taste of food. In food preparation, separation and

\* Corresponding author. Tel.: +81 952 28 8670; fax: +81 952 28 8548.  
E-mail address: [kawakita@cc.saga-u.ac.jp](mailto:kawakita@cc.saga-u.ac.jp) (H. Kawakita).

Received 30 November 2012; Received in revised form 8 April 2013; Accepted 12 April 2013

mixing to promote cooking of ingredients has been achieved by centrifugation and mixing, while food processing during cooking, based on science and engineering, to enhance food taste has become known as molecular gastronomy (Hervé, 2006).

In this study, recovery of starch granules dispersed in solution was performed by size using an assembled-magnetite membrane formed by a magnet in an open channel. Magnetite was prepared at micrometer scale and placed in an open flow channel. The amount of magnetite set on the magnet in the channel was changed to alter the density of the set magnetite, as shown in Fig. 1. In assembled-magnetite membrane at high density, the interparticle distance is small. The starch-granules-dispersed solution is allowed to flow from the upper to the lower part of the channel, and the starch granules are recovered by the assembled magnetite, which acts like a membrane. The distance between magnetite particles would be about 50  $\mu\text{m}$ , which is suitable for starch-granules recovery by size effect because the size of the magnetite particles ranged from 10 to 100  $\mu\text{m}$ . The assembled structure of the magnetite depends on the species of magnetite, its amount, and the distance between the magnetite and the magnet.

To investigate construction of the assembled-magnetite membrane, magnetite of various densities were set in flow channels in an open system. A solution containing dispersed starch granules was allowed to flow through the assembled-magnetite membrane in the open system, and the concentration of recovered starch was determined for each fraction. During the recovery, a residue formed on the magnetite membrane, which changed the recovery behavior of the starch granules. The assembled magnetite has two advantages: (1) the gaps between the magnetite are controllable in changing the amount of magnetite set, species of the magnetite, and the distance between the magnetite and magnet in open flow, forming the several states of the gap in the membrane for the efficient recovery based on the size effect, and (2) filtered colloidal particle are easily obtained when the magnet to set the magnetite membrane is taken away. The relationship between the density of the assembled-magnetite membrane and the recovery efficiency of the starch granules was examined.

## 2. Experimental

### 2.1. Materials

Starch (origin: potato, KPH7012),  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ , glucose, and maltose were obtained from Wako Pure Chemical Industries (Japan). The magnet (0.23 T, length 5.0 cm) was obtained from BEST Corporation, Japan. Other chemicals used were of analytical grade or higher.

### 2.2. Preparation of magnetite

$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  (0.2 M) and  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  (0.1 M) were dissolved in 200 mL of water. An 8-mL aliquot of sodium hydroxide solution (10 M) was added to the solution at 500 rpm for 1 h at 303 K. After the reaction, sonication was performed for 10 min. The particles in the solution were recovered by centrifugation for 30 min at 12 000 rpm. The obtained black precipitate was washed with water, and dried in vacuum. Chemical characterization of the obtained magnetite was performed by Fourier transform-infrared (FT-IR) spectroscopy (JASCO FT/IR-410). The specific surface area was determined by nitrogen gas

adsorption and BET analysis (BELSORP-miniII-SP, BEL Japan). The black powder was observed by scanning microscopy (JSM-5200, JEOL, Japan) and optical microscopy (VH-S5, Keyence, Osaka, Japan). The yield of the powder was 1.9 g.

### 2.3. Recovery of starch granules by assembled-magnetite membrane in an open flow

The obtained magnetite was set in an open system, as shown in Fig. 1. The amount of magnetite set ranged from 0.05 to 0.5 g to control the various densities of the assembled-magnetite membrane. The flow channel was made from polyacrylic board, and measured 32 cm long and 0.4 cm wide. The slope of the channel was set at 3°. The distance between the magnetite and the magnet was 4 mm, while the distance of the set magnetite from the top of the channel was 10 cm. The magnetite volume was calculated by analysis of photographs taken with a digital camera (DMC-FH5, Panasonic, Japan), and the density of the assembled-magnetite membrane was calculated by dividing the amount of magnetite added by the volume of the magnetite.

Starch powder was dispersed to pure water at the concentration of 5.0 g/L. A starch-granules-dispersed solution was pumped by peristaltic pump (Perista Bio-Minipump AC-2120, Atto, Japan) from the top of the channel. The flow rate was set at 1.5 mL/min. The filtrate that flowed through the assembled-magnetite membrane was collected for each fraction. The concentration of the starch granules in the filtrate fractions was determined by measuring the turbidity (600 nm, UV-VIS 3100, Hitachi). The amount of starch granules recovered by the assembled-magnetite membrane was calculated from:

$$\text{Amount of starch recovered [g]} = \sum_0^v (C_0 - C_i)v_i \quad (1)$$

where  $C_0$  is concentration of starch granules dispersed in each fraction,  $C_i$  is the initial concentration of starch granules dispersed in solution, and  $v_i$  is the volume of each fraction. The flown starch granules in each fraction through the membrane were observed by optical microscopy. More than 100 samples were quantitatively analyzed from the observed images to determine the particle size distribution of the starch granules.

## 3. Results and discussion

### 3.1. Magnetite particle preparation

Magnetite prepared with aid of a magnet appeared as micrometer-scale particles. The magnetite was set up in an open channel to function as a membrane and was prepared according to the literature method (Chatterjee et al., 2003). Based on observation by optical microscopy, the diameter of the magnetite particles was approximately 150  $\mu\text{m}$ , as shown in Fig. 2a. The gaps between the assembled magnetites by the magnet would function as a microfiltration membrane based on the size effect.

The FT-IR spectrum of the obtained black magnetite is shown in Fig. 2b. The peaks at 3350  $\text{cm}^{-1}$  and 530  $\text{cm}^{-1}$  correspond to the hydroxyl group and Fe–O bonding, respectively. The specific surface area of the obtained particle was 40  $\text{m}^2/\text{g}$  based on nitrogen gas adsorption, indicating that the magnetite has a porous structure with multiple hydroxyl groups on the pore surface.

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