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Physical Properties of Korean Earthenware Containers Affected by Soy Sauce Fermentation Use

Gyeong Hee Seo, Jung Hyun Yun, Sun Kyung Chung[1], Woo-Po Park[2], and Dong Sun Lee*

Division of Food Science and Biotechnology, Kyungnam University, Masan, Gyeongnam 631-701, Korea
[1]Department of Baking & Confectionery, Changwon College, Changwon, Gyeongnam 641-771, Korea
[2]Division of Food Science, Masan College, Masan, Gyeongnam 630-729, Korea

Abstract Soy sauce was fermented at 20°C for 100 days in *onggi* containers (ethnic Korean earthenware) which had been fabricated using three different glazing treatments: unglazed, glazed only on the outside, and glazed on both surfaces. The changes in microstructure and permeability characteristics of *onggi* containers were examined after fermentation of soy sauce. The effect of repeated use of *onggi* containers on the fermentation was analyzed by the contact between an aqueous model solution and the *onggi* containers used once for soy sauce fermentation. The levels of reducing sugar and free amino acids produced from the dissolved starch and protein, respectively, in the solution were compared between the new and reused *onggi* containers. The moisture permeance and gas permeabilities of the *onggi* jars were progressively reduced with continuing use for soy sauce fermentation, probably due to clogging of micropores by solid materials. After having been used once for fermentation, the microbial cells and/or enzymes immobilized on the surface or in the micropores of the *onggi* containers seemed to contribute to accelerating the hydrolytic reactions of starch and protein.

Keywords: *onggi*, moisture transmission, gas permeability, microstructure, micropore

Introduction

Earthenware pots or containers called *onggi* have been widely used in Korea for ripening and storing a variety of salted fermented foods, including soy sauce, pickled fish, *kimchi,* and brewed wine (1). Compared to plastic and metal containers, the use of *onggi* containers as fermentation vessels for *doenjang* (soybean paste) and soy sauce improved the sensory quality due to the presence of desired microbial flora and characteristic flavors (2, 3). The *onggi* container structure consists of micropores that offer very high gas and moisture permeation rates (4). The clay type and surface glazing treatment are also reported to influence the gas and moisture permeability of the *onggi* containers. Unique permeability characteristics and wide range of controllable permeability present a good opportunity to obtain optimum packaging conditions for respiring or ripening products.

In most cases, *onggi* containers are repeatedly used for fermentation or containment of various foods. This repeated use affects the physical properties such as gas permeability and internal microstructure, which in turn influences the fermentation or ripening process and hence the final food quality. This study investigated the effect of repeated use in fermentation on the physical properties of *onggi* containers.

Materials and Methods

Earthenware container samples Three kinds of *onggi* containers, differing in glazing treatments, were manufactured from typical clay formulations of *onggi* (4). The *onggi* containers were shaped into small cylinders with a diameter of 8.0 cm, a height of 12.5 cm, a wall thickness of 4 mm, and an internal volume of 628 mL. A surface glaze coating was used as a control variable to modify the barrier properties: coated on the external surface alone, coated on both the inside and outside surfaces, and unglazed.

The *onggi* manufacturing procedure consisted of shaping the container, followed by drying for 24 hr under ambient conditions, initial firing at 750°C for 7 hr, applying the optional glaze coating, and a final firing in an industrial kiln at about 1,100°C for 11 hr. The glaze was prepared by dispersing humus and pine tree ash in water to obtain a soluble solid content of 35 Baumé. Twenty replicate containers were manufactured for each treatment.

Storage of fermenting soy sauce in the earthenware containers Seventy-seven grams of dried soybean *koji*, prepared from cooked whole soybean, were placed in an individual *onggi*, were covered with 350 mL of saline water (17-18% salt content) and then stored for 100 days of ripening at a temperature of 20°C (5). The containers used for ripening soy sauce were recovered after a predetermined period, washed and dried for 1 week at room temperature.

Internal morphology and permeabilities to gases and moisture The pore size and the porosity of the new and used *onggi* containers were measured using a mercury porosimeter (AutoPore IV9510; Micromeritics, Norcross, GA, USA). The morphologies of the sample surface and of a cross-section were observed using a scanning electron microscope (SEM, Philips XL30-FEG; FEI Co., Hillsboro, OR, USA) after coating the samples with platinum.

The gas permeation and moisture transmission of the *onggi* containers were measured according to the methods

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*Corresponding author: Tel: 82-55-249-2687; Fax: 82-55-249-2995
E-mail: dongsun@kyungnam.ac.kr
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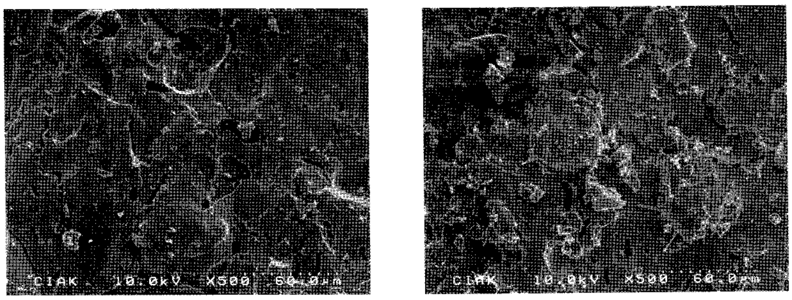
outlined by Seo *et al.* (4). The permeability of the samples to oxygen and carbon dioxide (mmol atm^{-1} m^{-2} hr^{-1}) was determined from the changes in the gas concentrations of sealed *onggi* jars flushed with CO_2 gas and exposed to air at 20°C. The moisture permeance (g Pa^{-1} m^{-2} day^{-1}) was determined from the weight loss rate of the sealed *onggi* jars filled with 375 mL of distilled water and placed inside a desiccator filled with dry silica gel at 20°C. All the results are the average of the measurements conducted in triplicate.

Effect of the reuse of container on the fermentation activity of model food A model food was prepared to see how repeated use of the container for fermentation affected the fermentative activity. An aqueous solution of soluble starch and protein solution, 0.1% each, was prepared. A 350 g solution of mixed protein and starch solution was placed in a container and stored at 20°C for 10 days. The reducing sugar and amino acids produced from respective hydrolysis of starch and protein were determined for triplicate container samples by the analytical methods described below.

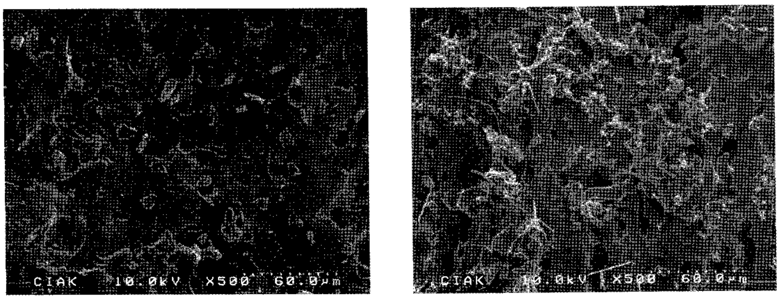
The total reducing sugar content of the sample was measured according to the method of Yu *et al.* (6). For the analysis of amino acid content, the samples were diluted (1:1) to precipitate the proteins, then stored at 4°C for 1 hr, and filtered to inject 20 μL of the sample into a Biochrom 20 Plus Amino Acid Analyzer (Biochrom Ltd., Cambridge, England) equipped with Li^+ anion-exchange Peek column (Biochrom Ltd.) and fitted with a fluorescence detector. Results were recorded using an Ezchrom Elite V2.8 Data Handling System.

Results and Discussion

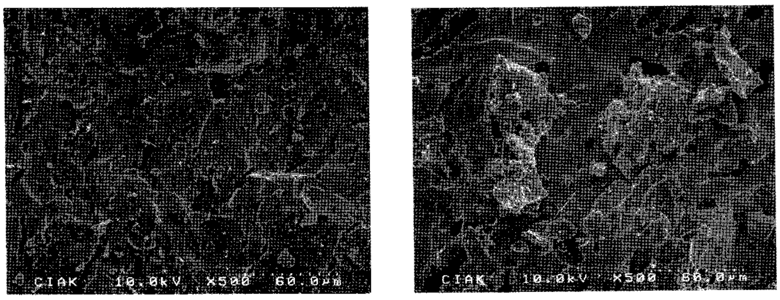
Change in microstructural properties of *onggi* containers used for soy sauce fermentation The internal microstructure of *onggi* containers in Fig. 1 showed very little difference with glazing treatment (Fig. 1). When compared to the reported internal morphology of the laboratory-fired *onggi* containers, the microstructure of industrial-kiln fired *onggi* containers was denser, probably



New *onggi* container glazed on both surfaces   Used *onggi* container glazed on both surfaces



New *onggi* container glazed only outside   Used *onggi* container glazed only outside



New *onggi* container unglazed   Used *onggi* container unglazed

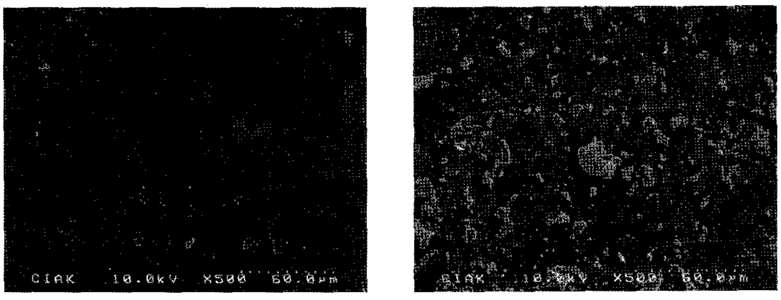
Fig. 1. Scanning electron micrographs of the internal microstructure of the *onggi* containers used for fermenting soy sauce. Used containers had been used for soy sauce fermentation at 20°C for 100 days.

Table 1. Pore size and porosity of new and reused *onggi* containers

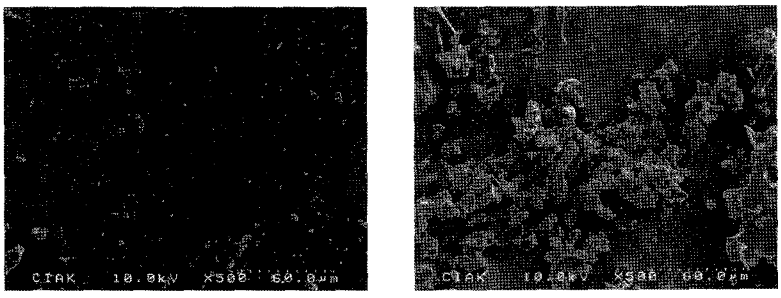
Glazing conditions	Ripening time (day)	Pore diameter (nm)	Porosity (%)
Both surfaces	Initial	53.60±2.83	8.39±0.30
	100	46.95±23.12	6.68±0.15
Outside surface only	Initial	147.90±0.00	7.28±0.33
	100	18.55±1.48	7.17±0.35
None	Initial	161.30±22.34	7.93±0.55
	100	23.50±1.84	5.79±1.75

due to harsh sintering conditions in industrial kilns. The pore size and porosity of these samples were smaller and lower, respectively, than those of Seo *et al.* (4). The morphology and color of earthenware can be altered by variations in sintering conditions (7, 8). The conditions of atmosphere and temperature in the firing and post firing phases can affect the body of the pottery by allowing the dominant gases to act on the surfaces of the pots and sometimes to penetrate into the body. Glazing on both surfaces seemed to reduce the pore size probably due to infusion of glaze material into the internal porous structure (Table 1). However, the effect of glazing on porosity was not clearly seen probably due to wide variability of the structure with the lower range of porosity.

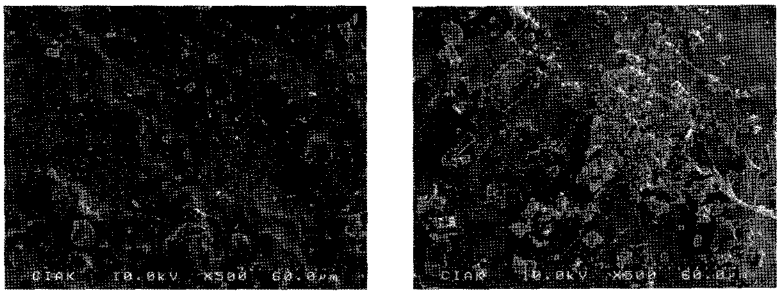
Surface glazing produced a smooth surface with few pores (Fig. 2). The *onggi* containers once used for ripening soy sauce were shown to retain solids inside the internal structure. The unglazed *onggi* container walls and those glazed only on the outside showed higher inclusion of solid particles. Solid materials were also observed to be adsorbed onto the non-glazed surfaces at higher amount than for the glazed ones. The solid materials entrapped inside the container wall structure and on the surface were presumed to consist of crystals of salt diffused through the wall, and the biological cells and enzymes adsorbed. These solid materials work to clog the pores on both the surface and inside of the wall. This clogging decreased the pore diameter and porosity after 100 days of fermentation use (Table 1). These changes in morphology were



New *onggi* container glazed on both surfaces Used *onggi* container glazed on both surfaces



New *onggi* container glazed only outside Used *onggi* container glazed only outside



New *onggi* container unglazed Used *onggi* container unglazed

Fig. 2. Scanning electron micrographs of the internal surface of the *onggi* containers used for fermenting soy sauce. Used containers had been used for soy sauce fermentation at 20°C for 100 days.

anticipated to change the mass transfer properties of the *onggi* container walls and were therefore examined in this study. Traces of organic materials may remain as surface residues or be adsorbed into the porous ceramic structure (9). Exact identification of the solid materials would improve our understanding of the effect of the entrapped solid particles on the physical property, and requires further study.

Change in gas permeabilities and moisture transmission of *onggi* containers used for soy sauce fermentation Table 2 shows the gas permeabilities to O_2 and CO_2, and the moisture permeance through the *onggi* container walls. Because of more concrete structure of the *onggi* containers fired in the industrial kiln of this study, their gas permeabilities and moisture transmission were lower than those of laboratory-fired containers reported by Seo *et al.* (4). This further again demonstrates the high dependence of mass transfer properties of *onggi* containers on the processing conditions.

It is interesting to note that there was little difference in initial moisture transmission and gas permeabilities between the earthenware containers glazed on both-surfaces and only on the outside surface (Table 2). Glazing even on a single surface only seemed to reduce the moisture and gas permeation significantly. Thirty-day storage of the fermenting soy sauce reduced the O_2 and CO_2 permeabilities of *onggi* containers significantly. The earthenware containers glazed on any surface did not undergo any further decrease in O_2 and CO_2 permeabilities after 30 days, whereas the unglazed container showed a consistent decrease in the gas permeabilities thereafter.

The container walls glazed on any surface did not show any noticeable change in moisture permeance, whereas the unglazed one showed some reduction.

As mentioned above, the moisture transmission and gas permeabilities of *onggi* jars used for soy sauce ripening should have decreased with time probably due to clogging of internal micropores by solid materials such as salt and adsorbed cells. This probably progressed through solute diffusion, moisture permeation and penetration of food particles, and microbial cells.

Effect of the reused container on the fermentative activity of model food Table 3 shows the production of reducing sugar and free amino acids from the aqueous starch and protein slurry which was contained in the *onggi* vessels that had been used once for soy sauce fermentation. After 10 days, the production of reducing sugar and amino acids tended to be higher with the reused *onggi* containers with an unglazed internal surface (the containers unglazed or glazed only on the outside). The magnitude of the difference between the new and reused vessels was more evident for amino acid production. However, there was no discernable difference between new and reused containers that were glazed inside (i.e. glazed on both sides) as the glazing prevented any direct contact between the soy sauce and porous internal structure. It was presumed that the microbial cells and/or enzymes immobilized on the surface or in the micropores of the reused container contributed to accelerating the hydrolytic reactions of the biopolymers, particularly protein. Because

Table 2. Effect of soy sauce ripening time on gas permeability and moisture permeance of earthenware at 20°C

Glazing conditions	Ripening time (day)	Gas permeance (mmol·atm^{-1} m^{-2} hr^{-1})		Moisture permeance (g Pa^{-1} m^{-2} day^{-1})
		O_2	CO_2	
Both surfaces	Initial	8.73±5.61	7.57±4.41	0.0004±0.0001
	30	3.62±0.79	3.45±2.49	0.0005±0.0002
	100	4.03±1.29	4.37±1.96	0.0009±0.0003
Outside surface only	Initial	9.73±4.70	8.86±4.91	0.0006±0.0001
	30	0.71±0.17	2.49±0.33	0.0005±0.0002
	100	0.58±0.08	3.28±0.37	0.0006±0.0001
None	Initial	44.15±5.57	36.71±7.52	0.0036±0.0010
	30	23.07±9.40	14.47±3.91	0.0024±0.0008
	100	3.20±1.95	3.82±0.33	0.0013±0.0001

Table 3. Effect of reusing *onggi* containers* on the production of reducing sugar and free amino acids from the model liquid food contained in them at 20°C for 10 days

Glazing conditions	Reducing sugar (mg/mL)		Free amino acid (mg/mL)	
	New	Reused	New	Reused
Both surfaces	0.284±0.060	0.305±0.056	0.038±0.020	0.044±0.011
Outside surface only	0.292±0.058	0.361±0.086	0.077±0.041	0.259±0.011
None	0.275±0.058	0.316±0.067	0.065±0.043	0.236±0.002

*The *onggi* containers were used for containing the model food, which was prepared by dissolving 0.1%(w/v) soluble starch and 0.1%(w/v) protein in water. Initial sample solutions were found not to contain reducing sugar and free amino acid. Reused containers were those which had contained soy sauce for 100 days at 20°C for the ripening.

the model solution was not prepared aseptically, the microbial load and enzymes initially present or contaminated during sample preparation would also have been responsible for the increase in reducing sugar and amino acids from the solution contained in fresh *onggi* containers.

There have been many applications employing porous ceramics as supports for cell immobilization in biochemical reactor systems to attain an enhanced fermentation productivity, improved cell stability, product quality, and continuous processing, etc. (10-12). The design of immobilization considers the absorbed molecule size relative to the pore size (13, 14). Even though the average pore sizes of the *onggi* container walls were very small (Table 1), some of the pores observed in Fig. 1 were larger than those of the cells and enzymes (2-10 μm), and thus may have been used as sites for their immobilization or trapping. Therefore, if the beneficial microorganisms and enzymes are immobilized (adsorbed) inside the wall structure of the used container, these *onggi* containers may offer a good control for creating the desired microbial flora and attaining consistent quality in the fermented foods.

Nevertheless, the continuing possibility of sticking by unwanted solid particles and microbial cells should be considered. Therefore, the possible immobilization of the microbial cells, enzymes and/or other solid materials should be carefully controlled in order to produce quality fermented products and avoid food safety concerns when the *onggi* containers are to be reused for storing or containing foods.

Acknowledgments

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