Food Sci. Biotechnol. Vol. 14, No. 1, pp. 82 ~ 88 (2005)

Food Science and Biotechnology

Permeabilities of Korean Earthenware Containers and Their Potential for Packaging Fresh Produce

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Abstract Ethnic Korean *onggi* earthenwares were fabricated using different clay formulations and glazing treatments. Their permeability properties against oxygen, carbon dioxide, and moisture were measured at 20°C and examined from the aspect of food preservation. *Onggi* walls consisted of micropores that offered higher gas and moisture permeation rates when compared with other food packaging materials. Earthenware walls were unique in having CO_2/O_2 permeability ratios of 0.60-1.00. Gas and moisture permeabilities were lower, with wall structure having lower porosity and surface glazing. Results revealed *onggi* jar packages of grape fruits could attain wide range of O_2 and CO_2 concentrations. *Onggi* containers present good opportunity to obtain optimum packaging conditions for respiring or ripening products, depending on commodity type.

Keywords: moisture, oxygen, carbon dioxide, permeability, modified atmosphere

Introduction

Earthenware pottery containers, with volumes ranging from 1 L for small food vessels to 400 L for larger-sized containers, have traditionally been widely used in Korea for storing cereals, vegetables, meat, and fish (1, 2). A variety of salted fermented foods have been developed for ripening and storing in these earthenware pots, which are believed to play an essential role in producing the desired microbial flora and characteristic flavors. Korean pottery has microscopic pores and a red color (3). In ancient literature and cultural stories, the porous physical structure of the potteries, which differs according to the manufacturing process used, is said to contribute to producing the desired quality of the respective food. The type of oxidized earthenware used in Korea is called *onggi*, and is usually manufactured by forming and drying fine clay paste, and subsequent firing at 950-1,110°C in a tube kiln. The earthenware may be coated on the surface with a natural ash-glaze to give the desired barrier properties.

The wide usage of *onggi* in Korean food culture is different from that of normal porcelains, which are appreciated for their artistic beauty in food presentation. The Korean people believe that *onggi* helps to maintain the quality of food contained inside it (2). Yoo *et al.* (4) showed that Korean *doenjang* (soybean paste) ripened in *onggi* vessel contained a higher amino-type nitrogen content, resulting in the improved sensory quality compared to *doenjang* ripened in plastic containers. Even though *onggi* is said to have some food preservation function in packaging and containing food, so far, there is a paucity of scientific investigation on its effectiveness and principles. To evaluate the effectiveness of *onggi* in food preservation and ripening, the degree of its barrier against moisture and gas needs to be determined and related to the food quality preservation, because quality changes in packaged foodstuffs depend on interactions between the containment and the external environment (5, 6). Oxygen permeation of the package is closely related to various chemical and biological changes of the food, such as rancidity development, respiration, microbial growth, enzymatic browning, and nutrient loss. The permeation of carbon dioxide is often helpful in maintaining aerobic respiration processes in fresh produce, and avoids excessive package expansion or product leakage from fermented foods evolving CO_2 gas (7-9). The water vapor permeability of packaging is often tailored to control the moisture or salt content of the contained products. The combination of O_2 and CO_2 permeabilities plays a role of creating modified atmosphere inside the container of fresh produce. Even though *onggi* containers are currently used as open-mouth, those with controlled permeability characteristics may be fabricated as a novel food package or storage vessel to build up a desired atmospheric condition internally with an elaborate design of the hermetic closure system.

Therefore, evaluation of the permeability properties as functions of the manufacturing variables of this type of earthenware would aid in the understanding of the true functional mechanism of the Korean *onggi*, and in developing container conditions for different types of foods. The objectives of this study were thus to evaluate the moisture and gas permeability properties of *onggi* manufactured using different clay formulations and glazing treatments. The observed properties were correlated with the microstructural properties of the materials. The potential of *onggi* in food packaging and preservation application was tested for maintaining a modified atmosphere of fresh produce package.

Materials and Methods

Earthenware containers Three types of *onggi*, differing in their clay formulation, were manufactured by simulating a traditional process in the Laboratory of Porcelain Manu-

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Received September 7, 2004; accepted October 14, 2004

facturing, Yangsan College, Yangsan, Korea. The clay formulation was designed to produce different porosities and physical properties in the pottery; two types of clay, which are commonly used in *onggi* manufacturing industry in Ulsan, Korea, were formulated in different ratios to manufacture three types of *onggi* (Table 1 and 2). Each clay type was produced by mixing soils from different locations in equal proportions. The particle diameter of the clay material measured by light scattering after sieving (10, 11) and the specific surface area determined using a gas adsorption analyzer (TriStar3000, Micromeritics, Norcross, GA, USA) (12) are presented in Table 1. Clay "a" was of the finer particles, and had a slightly larger surface area than Clay "b". The *onggi* containers were shaped into small cylinders having dimensions of 8.0 cm (diameter) x 12.5 cm (height), with an internal volume of 628 mL. A glaze coating was used as another control variable for the surface treatment to modify the barrier properties. This coating was applied on the external surface only, or on both the inside and outside surfaces, while an uncoated container was subjected directly to drying and firing.

The *onggi* manufacturing procedure consisted of shaping the container, followed by drying for 24 hr under ambient conditions. The *onggi* potteries were then fired at 750°C for 7 hr, followed by optional glaze coating, and firing was carried out at 1,100-1,190°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, measured using a specific gravity hydrometer. The entire drying and firing processes required careful control of the heating and cooling cycles. Thickness of the pottery was about 4 mm, deduced from an average of five measurements taken at five different positions. Flat slab-form samples ($4\times4\times0.72\ \mathrm{cm}^3$) were also made following the same procedure to measure the density of the *onggi* potteries based on their weights and dimensions.

Micromorphological analysis Pore size and porosity of three types of unglazed *onggi*, assumed to be important factors controlling the barrier properties (13), were measured using a mercury porosimeter (AutoPore IV9510, Micromeritics, Norcross, GA, USA). Averages of at least three measurements were used. Morphologies of the sample surface and a cross-section was observed using a scanning electron microscope (Philips XL30-FEG, FEI Co., Hillsboro, OR, USA) after Pt-coating. The cross-sections of the walls were obtained by breaking the containers.

Gas permeability measurement Permeabilities of the samples to oxygen and carbon dioxide were measured at 20±1°C. The jars were covered with a 5-mm thick acrylic lid, and the top sealed using a silicon adhesive (Fig. 1). Three silicon sampling ports were made on the acrylic lid for gas flushing and headspace gas sampling. The jar samples were prepared at least 7 days before the experiment for the silicon adhesive to dry. In the gas permeability test, the jars were flushed with CO_2 gas injected through silicon sampling ports to replace the air inside the earthenware. A vacuum pump linked to another sampling port was used to assist the gas flushing operation. The flushed jars were placed under atmospheric air conditions with controlled temperature to allow CO_2, O_2, and N_2 gases to permeate through the jar wall. One milliliter of headspace gas in the pottery was sampled periodically using a gas-tight syringe, and the concentrations of O_2 and CO_2 were determined using a Varian Model 3800 Gas Chromatograph (Varian Inc., Palo Alto, CA, USA) equipped with an Alltech CTR I Column (Alltech Associates Inc., Deerfield, IL, USA) and a thermal conductivity detector. The temperature of the column was maintained at 40°C, with injection and detection temperatures at 70 and 90°C, respectively. Helium was used as the carrier gas at a flow rate of 30 mL/min.

The gas permeability through the pottery wall was determined based on the changes in the O_2 and CO_2 concentrations using the method described below. When the pottery jar is flushed with CO_2 gas and exposed to ambient air, the mass balance of Gas i (O_2 or CO_2) in the

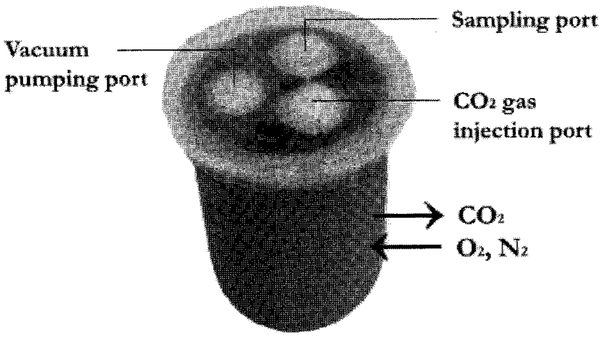


Fig. 1. Photographic view of *onggi* containers submitted to gas permeability measurement.

Table 1. Clays used for manufacturing the earthenware used in this study

Clay	Particle diameter, weight averaged (μm)	Specific surface area ($m^2\cdot g^{-1}$)	Source
a	53.7	27.5±2.1	Seonghwan and Muan, Korea
b	71.1	26.0±1.9	Yangsan and Sanchung, Korea

Table 2. Microstructural properties of the earthenware samples

Pottery type	Clay mix ratio (a:b)	Pore diameter (nm)	Porosity (%)	Density ($kg\cdot m^{-3}$)
A	100:0	829±21	14.6±0.2	2,213±17
B	60:40	1,152±11	19.6±0.1	2,172±33
C	40:60	892±46	21.9±0.1	2,122±37

jar is determined as the sum of the gas permeations through the silicon sealing area (including the acrylic lid) and the jar wall as follows:

$$\frac{dn_{i,in}}{dt}=\frac{\bar{P}_{i,s}A_s(p_{i,out}-p_{i,in})}{L_s}+\frac{\bar{P}_{i,p}A_p(p_{i,out}-p_{i,in})}{L_p} \qquad \text{Eq. (1)}$$

where $n_{i,in}$ is the mole number (mmol) of Gas i inside the jar at time t, $p_{i,in}$ is the partial pressure (atm) of Gas i inside the jar at time t, $p_{i,out}$ is the partial pressure (atm) of Gas i outside the jar, $\bar{P}_{i,s}$ is the permeability coefficient of the silicon sealing ($mmol{\cdot}mm{\cdot}m^{-2}{\cdot}hr^{-1}{\cdot}atm^{-1}$), $\bar{P}_{i,p}$ is the permeability coefficient of the pottery wall ($mmol{\cdot}mm{\cdot}m^{2}\ hr^{-1}{\cdot}atm^{-1}$), A_s and A_p are the areas of the silicon sealing and jar wall, respectively (m^2), and L_s and L_p are the thicknesses of the silicon sealing and the jar wall, respectively (mm).

If the ideal gas state equation is applied to express the mass change on the left side ($dn_{i,in}$) in terms of the changes in internal partial pressure, Eq. (2) and (3) result.

$$\frac{V\cdot 101325dp_{i,in}}{RTdt}=\frac{\bar{P}_{i,s}A_s(p_{i,out}-p_{i,in})}{L_s}+\frac{\bar{P}_{i,p}A_p(p_{i,out}-p_{i,in})}{L_p} \qquad \text{Eq.(2)}$$

$$\frac{dp_{i,in}}{dt}=\frac{RT}{101325V}\left[\frac{\bar{P}_{i,s}A_s(p_{i,out}-p_{i,in})}{L_s}+\frac{\bar{P}_{i,p}A_p(p_{i,out}-p_{i,in})}{L_p}\right] \qquad \text{Eq.(3)}$$

where V is the internal jar volume (mL), and R is gas constant (8.314 J K^{-1} mol^{-1}). The value of 101325 was added in the equations to have consistency of atm in units of partial pressure.

When the outside conditions are constant with fixed $p_{i,out}$, then Eq. 3 can be integrated to produce Eq. 4

$$\ln\left(\frac{p_{i,out}-p_{i,in,o}}{p_{i,out}-p_{i,in}}\right)=\frac{RT}{101325V}\left[\frac{\bar{P}_{i,s}A_s}{L_s}t+\frac{\bar{P}_{i,p}A_p}{L_p}t\right] \qquad \text{Eq.(4)}$$

where $p_{i,in,o}$ is the initial partial pressure of Gas i inside the pottery (atm).

If non-permeable control containers, such as glass or metal jars, are fastened using the same silicon sealing method, flushed with the gas, and exposed to the same environment to measure the gas concentrations, then the second term of the right side bracket in Eq. 3 cancels due to $\bar{P}_{i,p}$ being zero. Therefore, $\frac{\bar{P}_{i,s}A_s}{L_s}$ can be obtained easily from the gradient of a plot of $\ln\left(\frac{p_{i,out}-p_{i,in,o}}{p_{i,out}-p_{i,in}}\right)$ versus t for a given gas concentration data set. Once determined, the value of $\frac{\bar{P}_{i,s}A_s}{L_s}$ can be substituted into Eq. 4, determining the relationship of $\ln\left(\frac{p_{i,out}-p_{i,in,o}}{p_{i,out}-p_{i,in}}\right)$ versus t in the normal permeable pottery jar experiment, and the value of either $\bar{P}_{i,p}$ or $\bar{P}_{i,p}/L_p$ of the pottery wall can be determined. Upon multiplication of $\bar{P}_{i,p}$ by, $\frac{RT}{101325}$ the permeability coefficient can be found in unit of mL mm m^{-2} hr^{-1} atm^{-1} based on the atmospheric pressure and experimental temperature.

The gradients of the lines in the plots of $\ln\left(\frac{p_{i,out}-p_{i,in,o}}{p_{i,out}-p_{i,in}}\right)$ versus t were calculated using linear regression for $R^2>0.98$, with three replicates for each type of *onggi*.

Moisture permeability measurement To determine the moisture permeation of the pottery jars, jars with hermetically closed acrylic lid 5 mm thick, similar to Fig. 1 but without silicon sample port, were prepared after filling with 375 mL of distilled water. The pottery jars were then placed inside a desiccator filled with dry silica gel and kept at 20±1°C, with the resulting weight change recorded over a period of 20 days. The moisture permeance, $\bar{P}_w/L_p$, was determined from the rate of the weight loss using Eq. 5

$$-\frac{dW}{dt}=\frac{\bar{P}_wA_p(p_{ws}-p_{w,out})}{L_p} \qquad \text{Eq. (5)}$$

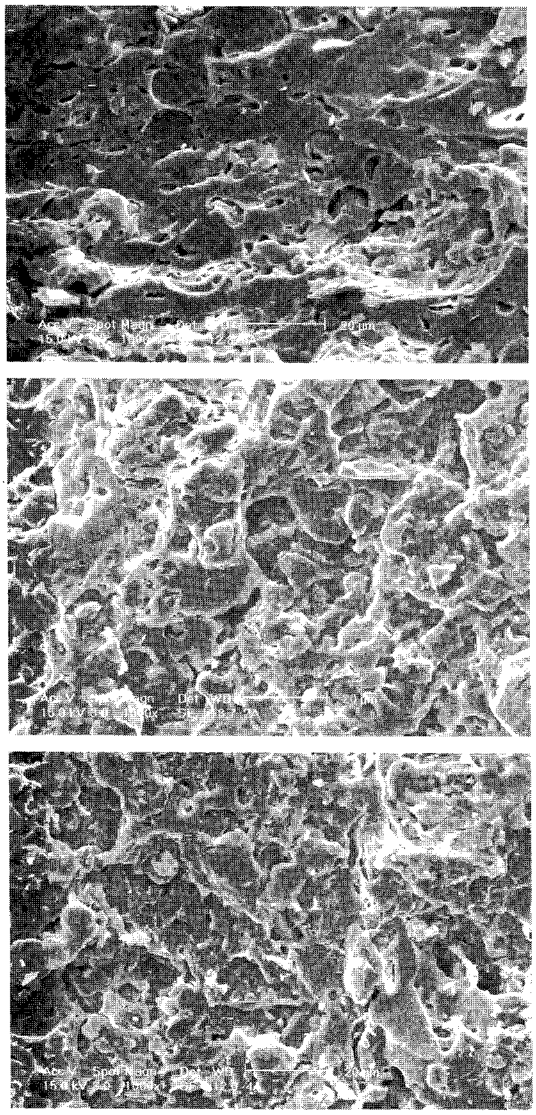
where W is weight of the water (g) at time t (d), A_p is the surface area of the pottery wall (m^2), p_{ws} is the water vapor pressure inside the pottery jar (Pa) (assumed to be the saturation vapor pressure of water at 20°C), $p_{w,out}$ is the water vapor pressure outside the pottery jar (assumed to be zero), and $\bar{P}_w$ is the moisture permeability coefficient ($g{\cdot}mm{\cdot}m^{-2}{\cdot}d^{-1}{\cdot}Pa^{-1}$). In Eq. 5, any moisture transmission through the acrylic lid and silicon seal was regarded as being negligible, which was confirmed by the control experiment using non-permeable glass containers sealed the same way. It needs to be mentioned that the mass transfer resistance of stagnant air considered in water vapor permeability test (14) was not incorporated in Eq. 5, which apparently describes the rate of moisture loss. Moisture permeation through the pottery wall comes in combination with liquid water layer and water vapor contacting the wall. The gradient of the weight loss, -dW/dt, was calculated using a linear repression of the data with $R^2>0.99$. Three replicates were used to measure each pottery type.

Modified atmosphere package experiment One hundred and fifty grams of Campbell Early variety grape fruits were put into the cylindrical *onggi* jar and stored for 6 days at 20°C to see potential of attaining modified atmosphere. The jar containing fruits was covered with a 5-mm thick acrylic lid and sealed with hot-melt glue. Gases inside the container were sampled periodically through a silicon sampling port to measure O_2 and CO_2 concentrations by the gas chromatography. Triplicate jars were prepared for each treatment. Respiration rate of grape fruits was measured by a simple closed system of glass jar (980 mL volume) containing 150 g of grapes (15, 16).

Results and Discussion

Microstructural properties Even though the earthenware materials may have visually similar outward appearances, they could have different microstructures and, therefore, different barrier properties (17). The micromorphology of the pottery is thought to determine the barrier properties, and thus was measured first to obtain a basic understanding of the samples. Type A *onggi* had the lowest porosity, while Type C was the most porous (Table 2). Inclusion of large particle clays in higher proportions produced a product with a high porosity (Table 1). The slight difference in the wall porosity of the three earthenwares is also reflected in the different microstructures observed using the scanning electron microscope (Fig. 2), which revealed that Type A *onggi* contained small pores, Type B *onggi* had a relatively small number of large pores, and Type C *onggi* contained numerous pores with various diameters. Higher porosity reduced the density of the *onggi* (Table 2), and glaze coating was observed to clog the micropores on the earthenware surface (Fig. 3). Glazing on the inside and outside of the container provided the highest physical barrier by clogging the pores on both surfaces. The internal porosity and the degree of surface imperviousness from these coatings are expected to influence the mass transfer properties of the pottery wall.

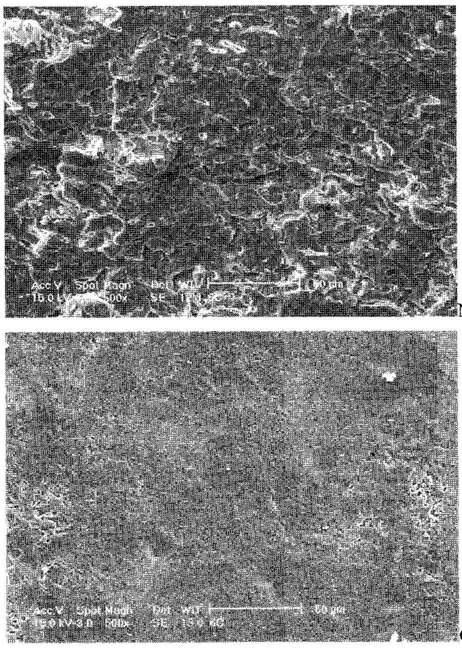
Gas permeation properties Figure 4 shows the changes in O_2 and CO_2 concentrations of *onggi* jars flushed with CO_2 gas. Silicon sealing was shown to have some degree of permeation, and while the earthenware wall provided a


Type A

Type B

Type C

Fig. 2. Scanning electron micrographs of the internal microstructure of the unglazed *onggi* containers.


Non-glazed surface

Glazed surface

Fig. 3. Scanning electron micrographs of the surface microstructure of Type A *onggi* containers.

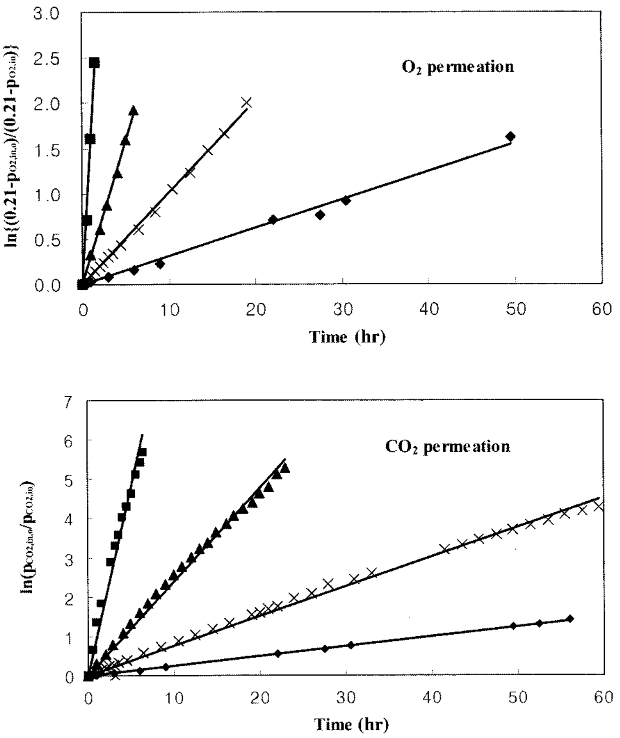


Fig. 4. O_2 and CO_2 permeation from Type A *onggi* containers that were initially flushed with 100% CO_2. ◆ = control glass jar with silicon sealed on top; × = glazed on both surfaces; ▲ = glazed on outer surface; ■ = unglazed.

Table 3. Gas permeance ($\bar{P}_i/L_p$) of earthenware to O_2 and CO_2 at 20°C

Type of earthenware	Glazing treatment	Gas permeance (mmol·atm^{-1}·m^{-2}·hr^{-1})		Permeability ratio ($\bar{P}_{CO2}/\bar{P}_{O2}$)
		O_2	CO_2	
A	Both surfaces	57.4±19.1	40.3±12.5	0.70
	Outside surface	166.7±2.9	166.7±30.8	1.00
	None	1,061.7±204.5	909.6±345.0	0.86
B	Both surfaces	715.0±173.4	531.3±121.4	0.74
	Outside surface	2,187.0±604.8	1,317.4±239.0	0.60
	None	5,390.8±192.9	3,334.7±376.2	0.62
C	Both surfaces	903.7±353.4	805.2±252.3	0.89
	Outside surface	2,108.0±168.8	1,921.8±137.6	0.91
	None	6,364.4±543.4	5,002.5±269.0	0.79

high degree of gas permeation, a glazing treatment slowed down the gas permeation significantly. The gas permeance values of the various *onggi* walls are shown in Table 3, calculated from the gradients of plots of the changes in the gas concentration. The earthenware with a higher porosity was more permeable to O_2 and CO_2, with the gas permeability of the pottery increasing in the order of Type A<Type B<Type C. The effect of glazing treatment on reducing the gas permeability was greatest in Type A *onggi* with the lowest porosity.

The gas permeation rate of the pottery wall is equal to that of a microperforated or microporous film, being much higher than that of the most polymeric films and sheets (18, 19), and thus would be expected to confer unique properties on the earthenware containers from the aspect of food storage and preservation. Many fresh produces and some fermented food products require highly permeable packaging materials to preserve their quality and package integrity (7, 8, 20-22). It is noteworthy that perforation in produce packaging has been attempted as a tool to control the atmosphere and relative humidity of the packaging by elevating the exchange rate of gas and moisture across the film (23, 24). Different clay mixing ratios and glazing treatments can be used to obtain the desired gas permeation rates for different food products (Table 3).

The permeability ratios of CO_2 to O_2 of the earthenware containers was in the range of $\bar{P}_{CO2}/\bar{P}_{O2}$=0.60-1.00, which is very different from those of most plastic films (typically, in the range of $\bar{P}_{CO2}/\bar{P}_{O2}$=4-7) (18, 25). The *onggi* permeability ratios are similar to those of air or perforations (15, 19), and thus suggest that gas permeation through the pottery wall is achieved mainly through the micropores. The uniqueness of the permeability ratio and high gas transmission rate of the *onggi* containers can provide an opportunity to obtain optimum packaging conditions for some foodstuffs such as fresh produce, which would be difficult to obtain using plastic packaging.

Moisture permeation property Figure 5 shows the weight loss with time in an *onggi* container containing water exposed to 0% humidity. The silicon sealing on the acrylic lid was found to have negligible moisture permeation, as observed by the imperceptible weight loss of the control glass jar. The *onggi* container walls were highly permeable to water, particularly when the surface was not coated with a glaze. Even when glazed, the container walls were shown to be permeable. The moisture

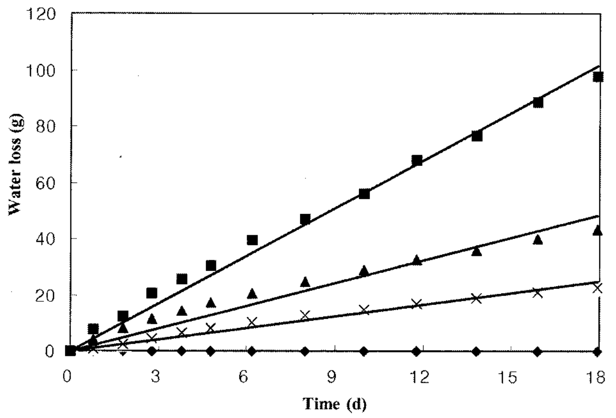


Fig. 5. Moisture loss from Type A *onggi* containers containing 375 mL of water and exposed to 0% humidity. ◆= control glass jar with silicon sealed on top; × = glazed on both surfaces; ▲ = glazed on outer surface; ■ = unglazed.

permeances determined from the gradient of plots of moisture loss versus time from the data are shown in Table 4. The glazing treatment on one surface reduced the moisture permeance by at least 50% for all earthenware types; the degree of reduction was greater for Types B and C, which is different from the effect of glazing on the gas permeation (Table 3). As expected from the differences in porosity and pore size (Table 2 and Fig. 2), the more porous *onggi* had a higher moisture permeance, with Type C *onggi* having the highest moisture permeance, and Type A *onggi* having the lowest one.

The water permeability coefficients calculated from the data of moisture permeances of unglazed earthenware walls were 0.256, 1.045, and 1.666 g mm m^{-2} d^{-1} Pa^{-1} for Types A, B, and C *onggi*, respectively (Table 4), which are much higher than those of highly water-permeable plastics, such as cellulose acetate and polystyrene, whose permeability coefficients are 3.9×10^{-3} and 6.3×10^{-4} g·mm·m^{-2}d^{-1}·Pa^{-1} at 25°C and relative humidity of 90%, respectively (25). Even the glazed pottery walls were much more permeable to water than the plastic films. A high water transmission rate may be desirable to avoid saturation and to maintain a required relative humidity for storing wet foods such as fresh produce. Plastic film usually causes moisture condensation inside the package when used for packaging fresh produce; thus, some means for promoting moisture transfer across the film or for

Table 4. Moisture permeance ($\bar{P}_w/L_p$) of earthenware at 20°C

Type of earthenware	Glazing treatment	Moisture permeance ($g{\cdot}Pa^{-1}{\cdot}m^{-2}{\cdot}d^{-1}$)
A	Both surfaces	0.0148±0.0077
	Outside surface	0.0282±0.0188
	None	0.0639±0.0047
B	Both surfaces	0.0290±0.0070
	Outside surface	0.0514±0.0104
	None	0.2612±0.0952
C	Both surfaces	0.0529±0.0154
	Outside surface	0.1475±0.0096
	None	0.4165±0.0616

Table 5. Modified atmosphere attained inside the *onggi* container containing 150 g of grape fruits at 20°C

Type of earthenware	Glazing treatment	Gas concentration (%)	
		O_2	CO_2
A	Both surfaces	8.9±2.0	14.8±2.8
	Outside surface	19.7±0.1	0.8±0.1
	None	20.1±0.1	0.3±0.1
B	Both surfaces	19.9±0.4	0.7±0.5
	Outside surface	20.2±0.3	0.1±0.1
	None	20.5±0.6	0.3±0.3
C	Both surfaces	20.0±0.2	0.4±0.1
	Outside surface	20.1±0.0	0.1±0.1
	None	20.2±0.1	0.0±0.0

absorbing transpired moisture are used (18, 23, 26). Korean *onggi* pottery may be adopted to provide a high moisture transmission rate to avoid moisture condensation inside the packaging. The pottery thickness and glazing treatment can also be varied to control the moisture transmission rate.

Gas concentration in onggi containers of grape fruits Concentrations of O_2 and CO_2 reached almost equilibrium stage in all *onggi* container packs at 20°C after 3 day storage, with additional 3-day storage bringing forth little changes. The equilibrated gas concentrations based on 6-day storage are presented in Table 5. Most packs except that of both side-glazed Type A *onggi* had internal atmospheres close to air due to high gas permeability (Table 3). Gas permeability of most *onggi* containers appeared to be too high to attain a significant modification of internal atmosphere, compared to the grape respiration, which was measured as 8.5 and 8.9 $mL{\cdot}kg^{-1}{\cdot}hr^{-1}$, respectively, for O_2 consumption and CO_2 evolution for the concentration range of 5-21% O_2 and 0-20% CO_2. However, the Type A *onggi* jar glazed both internally and externally attained modified atmosphere of 8.9% O_2 and 14.8% CO_2 concentrations, which is close to the optimal atmosphere (5-10% O_2 and 15-20% CO_2) for limited-period storage of grapes (27). However, it should be mentioned that the objective of this study was not to attain optimal package condition for a produce. This study shows that *onggi* jars with controlled permeability have a potential of application in modified atmosphere packaging for respiring or ripening products. A variety of modified atmosphere can be created by selecting proper type of *onggi* and tailoring package conditions of product weight and dimension. The attainable window of package atmosphere can be expanded using *onggi* with wide spectrum of permeability characteristics. Further study specific for the product or commodity is needed to achieve optimal *onggi* conditions, and works are in progress in the author's laboratory. Some means of closure system also needs to be developed for practical applications.

In conclusion, Korean *onggi* earthenware has very high gas and moisture permeation rates compared with other food packaging materials, which arise from its highly porous microstructure, and this is desirable for the preservation of certain food products. It is also unique in having CO_2/O_2 permeability ratios in the range of 0.60-1.00. The gas and moisture barrier properties of *onggi* can be varied and controlled using different clay formulations or glazing treatments. These *onggi* containers present a good opportunity to provide the optimum conditions for modified atmosphere package, depending on the products used.

Acknowledgments

This work was supported by a research grant from the Ministry of Agriculture and Forestry, Korea. Gyeong Hee Seo received scholarship from BK21 program of Korean Ministry of Education.

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