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Effect of Inorganic Nanocomposite Based Liners on Deodorization of Kimchi

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Abstract This study aims to reduce the rancid odor generated during the fermentation process of kimchi by inserting zinc oxide (ZnO) into an inorganic porous material with a high surface area to decompose or adsorb the fermentation odor. ZnO activated by the presence of moisture exhibits decomposition of rancid odors. Mixed with Titanium dioxide (TiO_2), a photocatalyst. To manufacture the packaging liner used in this study, NaOH, $ZnCl_2$, and TiO_2 powder were placed in a tank with diatomite and water. The sludge obtained via a hydrothermal ultrasonication synthesis was sintered in an oven. After being pin-milled and melt-blended, the powders were mixed with linear low-density polyethylene (L-LDPE) to make a masterbatch (M/B), which was further used to manufacture liners. A gas detector (GasTiger 2000) was used to investigate the total amount of sulfur compounds during fermentation and determine the reduction rate of the odor-causing compounds. The packaging liner cross-section and surface were investigated using a scanning electron microscope-energy dispersive X-ray spectrometer (SEM-EDS) to observe the adsorption of sulfur compounds. A variety of sulfur compounds associated with the perceived unpleasant odor of kimchi were analyzed using gas chromatography-mass spectrometry (GC-MS). For the analyses, kimchi was homogenized at room temperature and divided into several sample dishes. The performance of the liner was evaluated by comparing the total area of the GC-MS signals of major off-flavor sulfur compounds during the five days of fermentation at 20°C. As a result, Nano-grade inorganic compound liners reduced the sulfur content by 67 % on average, compared to ordinary polyethylene (PE) foam liners. Afterwards SEM-EDS was used to analyze the sulfur content adsorbed by the liners. The findings of this study strongly suggest that decomposition and adsorption of the odor-generating compounds occur more effectively in the newly-developed inorganic nanocomposite liners.

Keywords Off-flavor from kimchi, Inorganic nanocomposite liner, Sulfur compounds, Active packaging

Introduction

Kimchi, an iconic fermented food in Korea, has recently received public attention as a Korean food overseas. According to the Korea Agriculture & Food Trade Information (KATI), the global export of kimchi has grown steadily over the last five years from $ 78,900,000 in 2016 to $ 144,500,000 today (2021). Long-term storage and distribution result in the emission of fermentation odor due to the accumulation of metabolic gases from microorganisms trapped within kimchi containers. Volatile sulfur containing compounds are perceived as being noxious by foreign consumers unfamiliar with the kimchi odor, thus affecting the display and sale of products.

Sulfur compounds play an important role in determining the quality of kimchi products because of their low tolerance threshold and strong odor characteristics[1]. These compounds originate from kimchi ingredients including, cabbage, radish, red pepper, garlic, ginger, and green onion[2,3]. Major volatile sulfur compounds, such as allyl methyl sulfide, allyl methyl disulfide, dimethyl disulfide, dimethyl trisulfide, diallyl sulfide, and diallyl trisulfide, are responsible for the unpleasant consumer experience abroad[4,5,6,7]. Therefore, in this study, these compounds were selected as the key targets for deodorization.

To alleviate this problem, current packaging uses gas absorbents. These sachet-type packaging materials have limitations owing to the high cost and the need for a new production line or personnel to attach the sachet manually. A consumer warning in the form of a “Do not eat” label would also have to be printed or attached to the container to ensure consumer safety. Although studies of a natural deodorant[8] exist, the performance of these materials lacks efficiency. Thus, the development of a functional packaging that can utilize the existing kimchi packaging facilities to solve the manufacturing process, cost, and safety issues of sachet-type packaging is necessary. Liner packaging was selected as it is

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likely to be in contact with the fermentation gases.

The purpose of this study was to identify the critical sulfur compounds responsible for the perceived unpleasant smell in the provided samples of kimchi and develop an integral packaging using nano metal oxide materials such as ZnO and TiO_2 capable of deodorization[9] which were later incorporated into diatomite and L-LDPE[10] to synthesize a M/B.

Materials and Methods

1. Materials

Mildly flavored kimchi (Nasoya, Pulmuone, Iksan-si, Korea) was provided by the Jeonbuk Pulmuone Kimchi factory. Zinc chloride (CAS 7646-85-7), sodium hydroxide (CAS 1310-73-2), and titanium dioxide (CAS 13463-67-7) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Celite 263LD was acquired from Imerys (Paris, France). Linear low-density polyethylene (LDPE) was obtained from Lotte Chemical (Seoul, Korea) and azodicarbonamide foaming agents (UNICELL-D330 & UNICELL-D900) were purchased from Dongjin Semichem (Seoul, Korea).

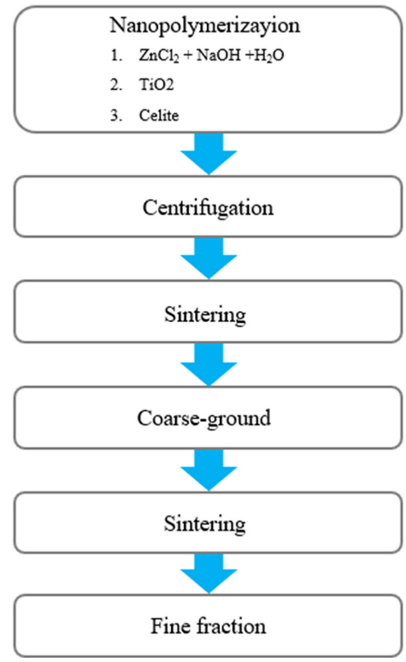

Fig. 1. Hydrothermal synthesis method.

2. Preparation of Inorganic Nanocomposite Liner

2.1. Material Processing

To synthesize the inorganic nanocomposites, sodium hydroxide solution was processed, as shown in Figure 1. Sodium hydroxide solution and distilled water (DW) were placed in the stirring tank along with 5 wt% of $ZnCl_2$ and 5 wt% TiO_2 powder. Thereafter, 15 wt% of Celite 263LD was added and the mixture was ultrasonicated at 500 W and 20 kHz. After centrifugation, the sludge was sintered (800°C, 1hr) and milled to approximately 10 μm.

2.2. Manufacture of Foam Sheet

Master batches were manufactured using a twin-screw extruder (Φ40, L/D = 36:1), which was mixed with 5 wt% ZnO, 5 wt% TiO_2, and 15 wt% Celite 263LD, (obtained by the hydrothermal synthesis method), and 75 wt% L-LDPE. The extruder temperature conditions were as follows: C1 = 120°C, C2 = 160°C, C3 = 160°C, C4 = 160°C, C5 = 160°C, C6 = 180°C, adaptor = 180°C, and dies = 200°C The extrusion was done at a screw speed of 90 rpm. A conveyor belt was used to cool the air-cooled extruded strands to form an M/B pellet.

To manufacture the PE foam sheets, foaming agent (7 wt%) and LDPE (93 wt%) were mixed in a tumbler mixer (20 L) and extracted from a single-screw T-die PE foam sheet extrusion line (HSS-PE105, Hansung Hanalon Co., Ltd. Φ110, L/D = 23:1). The extruder temperature was maintained at C1 = 120°C, C2 = 140°C, C3 = 160°C, C4 = 160°C, and dies = 180°C.

2.3. Inorganic Nanocomposite Liner Packaging

After cooling, the sheet was cut into a circle with a diameter of 7.25 mm and thickness of 0.98 mm. The final composition of the inorganic nanocomposite (IN) liner was 5 wt% ZnO, 5 wt% TiO_2, and 15 wt% Celite 263LD. Other samples with lower TiO_2 contents were discarded during the manufacturing process because of browning, which could be of concern to the consumer.

3. Equipment to Test Deodorization of Kimchi

The total sulfur content during the first, third, and fifth days of fermentation was measured and compared between the IN and PE foam liners using a gas detector (WANDI, GasTiger 2000, Korea). This equipment measures the colorless and odorless harmful gases in industrial sites and has a sulfur measurement range of 0-100 ppm, resolution of 0.01 ppm, and error ≤ ±3%. Kimchi samples were stored at 0~6°C, homogenized for 2 min in a blender, and then moved to a rubber bucket and mixed by hand for 3 min. A small quantity (50 g) of kimchi was distributed into containers with either PE foam or inorganic nanocomposite liners. The cap was locked and sealed with parafilm. Finally, the containers were sealed in an aluminum pouch with high gas barrier properties[11,12]. Each sample was measured by inserting a suction needle on days one, three, and five to measure the amount of total sulfur.

The profiles of the sulfur compounds, cited as the main components responsible for the unpleasant flavor of kimchi, were confirmed by GC-MS used at the Korea Food Research Institute. Homogenized samples (50 g) were placed in a glass desiccator sealed with parafilm, and the odor component was extracted by incubation at 70°C for 20 min. Then, 1 mL of the headspace gas was injected. The column used for GC-MS was a DB-WAX column (60 m × 0.25 mm film, thickness 0.25 μm), and the GC-MS carrier gas was high-purity helium at a flow rate of 1 mL/min. Volatile compounds were measured using an autosampler (Multipurpose Sampler with DHS option, MPS, Gerstel, Germany) equipped with 2.5 mL headspace, with GC-MS (7890B/5977A MSD, Hewlett-Packard Co., PA, USA). The analysis conditions are shown in Table 1. Component analysis of each peak was performed using a total ionization chromatogram (TIC). The mass spectra were analyzed based on the mass spectrum library (WILEY10N). The following formula was used to compare the reduction rates of the sulfur compounds:

$$\textit{Sulfur compound reduction rate (\%)} = \frac{\textit{PE area} - \textit{IN area}}{\textit{PE area}} \quad (1)$$

PE and IN area are the amount of sulfur compound within liner applied containers.

The cross-sectional structures of the IN and PE foam liners used for the GC-MS analyses were observed by scanning electron microscopy (SEM) (Hitachi, SU8220, Korea Basic Science Institute), and the sulfur density was scanned with EDS (HORIBA, E_{MAX} EVOLUTION EX-370 & X-MAX 50, Korea Basic Science Institute) to detect sulfur compound adsorption.

To verify the suitability of the porous materials, the specific surface area of diatomite was measured using a BET analyzer (BELSORP-mini II, SOLETECH). A sample of 5 g was prepared at an adsorption temperature of 77 K and adsorptive N_2.

4. Arrangement of Kimchi Packaging Units

It takes approximately four weeks to transport and store kimchi in local warehouses when kimchi is exported to the United States. Quality issues for kimchi, such as an unpleasant odor, have been reported and are thus expected. Hence, an evaluation test was conducted in the distribution field. Three boxes, A, B, and C were prepared with temperature loggers

Table 1. Analysis condition for GC-MS

Parameters	Condition
Column	Agilent J&W GC column DB-WAX (60 m × 0.25 mm × 0.25 μm)
Carrier gas	He
Flow rate	1 mL/min
Injector temperature	250°C
Detector temperature	230°C
Oven temperature	40°C(3 min) to 150°C(10 min) at a rate of 2°C/min, 200°C(10 min) at a rate of 4°C/min
Run time	90 min
Split ratio	Splitless
Mass range	40-550 m/z

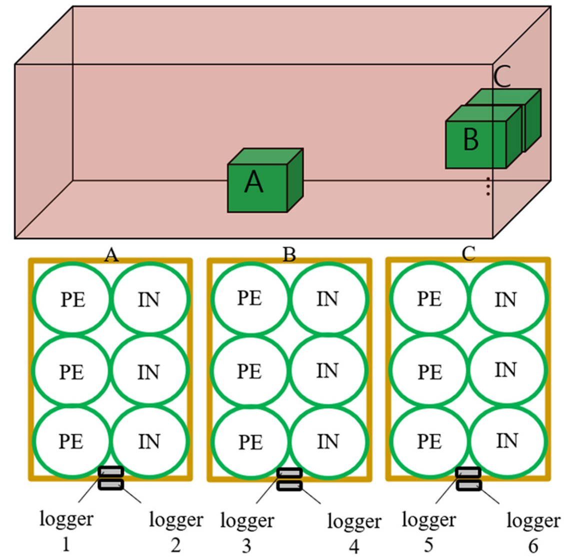

Fig. 2. Packaging samples and temperature logger layout for the transportation environment test.

(Testo 174T, Lenzkirch, Germany) attached inside and outside of the boxes and shipped from the production site to California, USA (Figure 2). All boxes were loaded with samples containing both PE foam and inorganic nanocomposite liners. After four weeks of shipping, the temperature loggers were retrieved to analyze the temperature profiles during shipping. The shipping container box can be loaded up to the 11th stage. Box B and box C were placed on the 10th stage in front of the entrance, where the temperature was expected to fluctuate significantly. Box A was placed at the center to secure the average temperature data.

5. Statistical Analysis

Duncan's test and one-way analysis of variance (ANOVA) were used to analyze the data using the Statistical Package for the Social Sciences (SPSS, Version 20.0, SPSS Inc., Chicago, IL, USA). The level of significance was set at $P < 0.05$ level for all analyses. Data were obtained from triplicate experiments.

Results and Discussion

1. Particle Size of ZnO

Nano-sized ZnO and TiO_2 particles were incorporated into diatomite, an inorganic porous material[13] via the hydrothermal method[14]. The particles initially had a maximum particle diameter (D99) of 400 μm, which was unsatisfactory for the master batch. After pin milling, the maximum particle diameter (D99) was reduced to 16 μm, and the average particle diameter (D50) was 3 μm (Figure 3). These powders were later used as the master batch ingredients.

Although inorganic nanocomposite powders were produced at particle diameters of up to 16 μm, bulky cell structures or

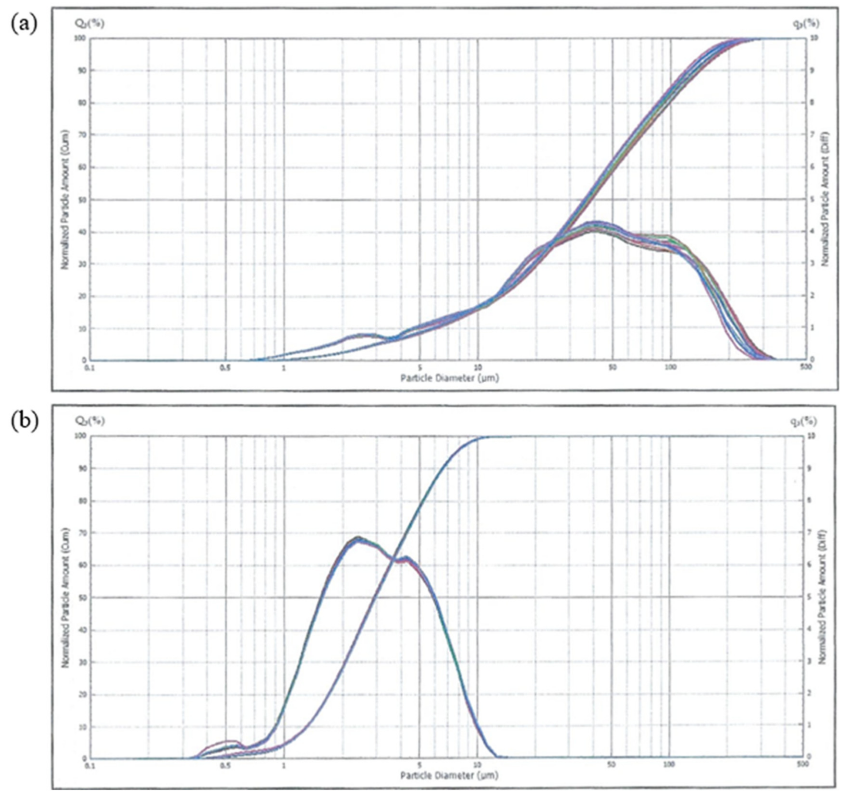

Fig. 3. Particle size distributions (a) before and (b) after pin-milling.

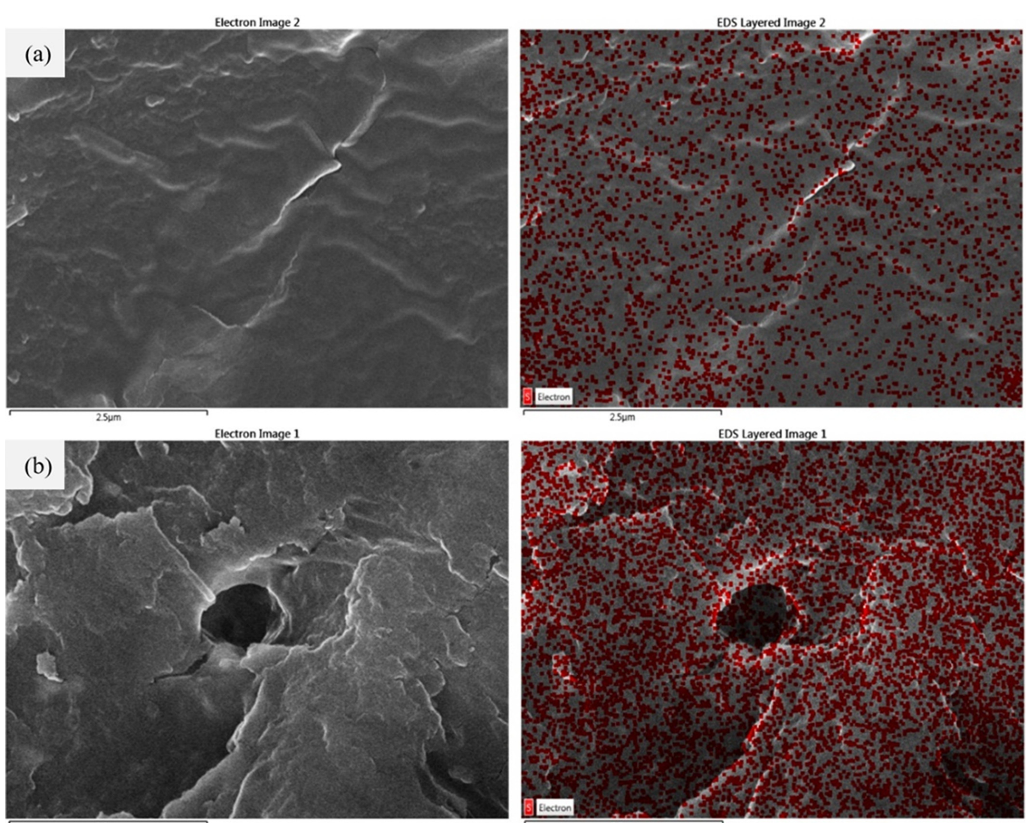

Fig. 4. Energy dispersive spectroscopy of (a) PE foam liner and (b) IN liner surface

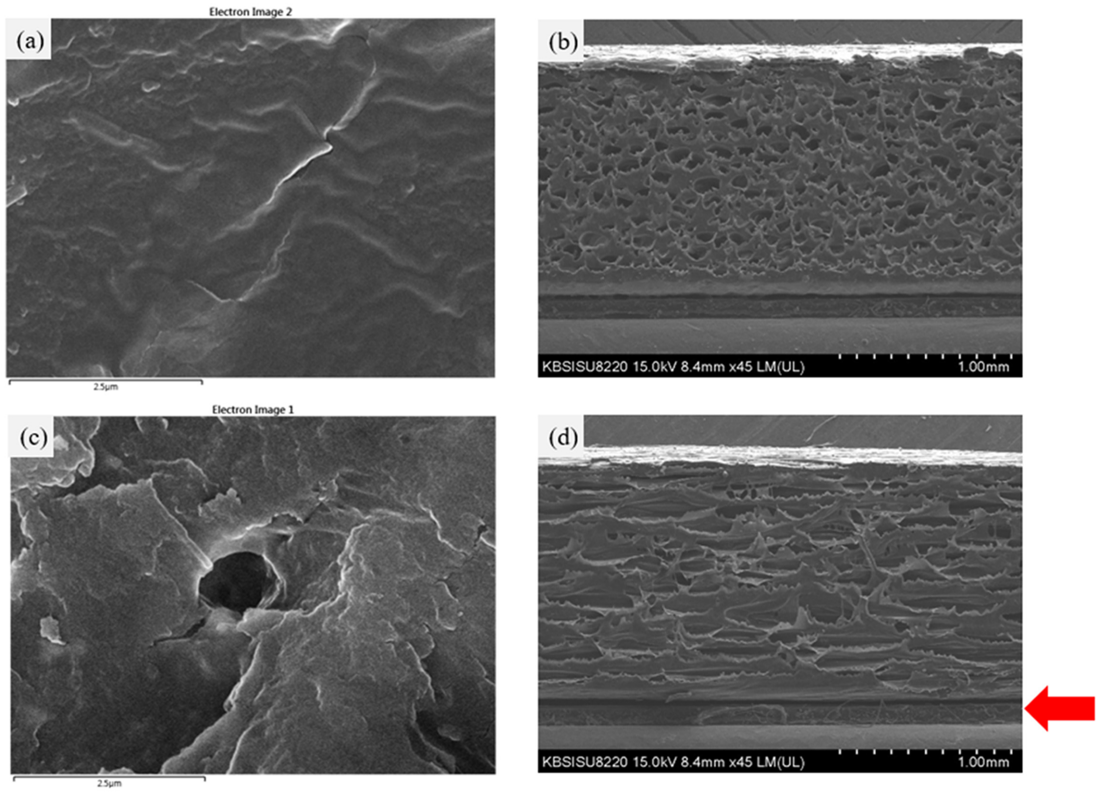

Fig. 5. SEM image of (a) closed cell structure surface of PE foam liner, (b) cross-section of PE foam liner (c) partially opened cell structure of IN liner (d) cross-section of IN liner.

open cells occurred in the liner, as shown in Figures 4 and 5. Therefore, it appears that it is necessary to pin-mill to a single digit micron diameter in the future.

2. Selecting the Foaming Agent

The structure of a cross-section of the PE foam sheet sample was observed using a microscope (Olympus BX50). The PE foam sheets treated with 7 wt% UNICELL D-330 and D-900 are shown in Figure 6. The average particle sizes of the foaming agents were 3 and 8 μm, respectively. UNICELL D-330-treated cells exhibited stable closed foam cell structures, whereas D-900-treated cells were inadequately dispersed/distributed.

As shown in Figure 6, the foaming agent using UNICELL D-900 was unevenly dispersed because of the large foam size; therefore, UNICELL D-330, which had a comparatively smaller foam size, was utilized. In addition, to reduce the foaming time, the melt extrusion temperature (T-die) of PE was lowered to 180 °C and foamed at a low temperature; however, bulky cells were formed overall (Figure 5). Therefore, it appears necessary to investigate a foaming agent with smaller cell particles than D-330.

3. Shipping Environment Test

The packaged kimchi was stored in the factory warehouse for four days, transported by a refrigerated container ship for four weeks, and then stored and shipped to the local logistics warehouse. To check the temperature profile, six temperature loggers were mounted at positions A, B, and C in a 40 ft container (Figure 2).

During the shipping process, all packages were maintained at a stable temperature of 0°C on board (Figure 8). However, after arriving in Los Angeles, the temperatures changed significantly during unloading and transportation by refrigerated trucks. There is a possibility of quality changes in products or contamination from microorganisms during transport[15,16,17,18]. Therefore, additional measures, such as reducing the unloading time and adding thermal packaging, are required to maintain stable conditions during the transportation phases.

4. Analysis of Sulfur Compounds

The total amount of sulfur compounds generated during the fermentation of kimchi and their reduction by inorganic nanocomposite liners was analyzed via a gas detector (Table 2). As the fermentation progressed, the total amount of sulfur compounds increased significantly in PE liners compared to IN liners. The difference in the total amount of sulfur compounds between three and five days was much larger than that between one and three days. The decrease in the inorganic nanocomposite liner samples is presumably due to deodori-

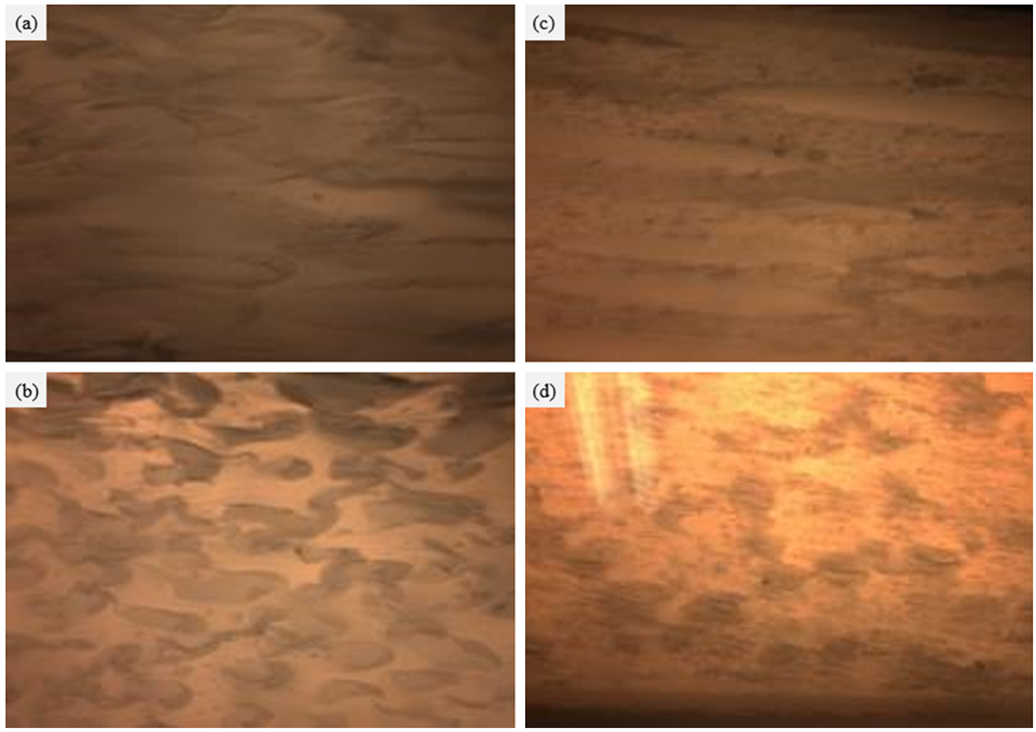

Fig. 6. Cross-section (×200) using UNICELL D-330; (a) Machine direction (MD), (b) Transverse direction (TD), and Cross-section using UNICELL D-900; (c) MD, (d) TD in PE foam liner

zation, with a decrease rate of 55.5% on day one, 56.0% on day three, and 62.3% on day five.

Based on the above results, GC-MS was used to determine the exact types of sulfur compounds and their extent of deodorization by area comparison. Table 3 shows the results for dimethyl disulfide, allyl methyl disulfide, dimethyl trisulfide, allyl methyl sulfide, diallyl disulfide, methyl propyl disulfide, diallyl sulfide, and allyl methyl trisulfide, based on the order of content. The detected sulfur compounds follow the trends observed in previous studies on the main compounds contributing to the rancid odors[4,5,6,7]. The average amount of deodorization in the inorganic nanocomposite liner compared

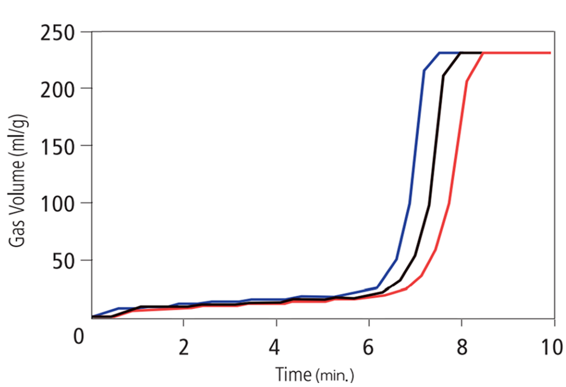

Fig. 7. Decomposition behavior of UNICELL-D series at a constant temperature of 200°C (Provided by Dongjin Semichem). ■: 2 μm ■: 6 μm ■: 15 μm

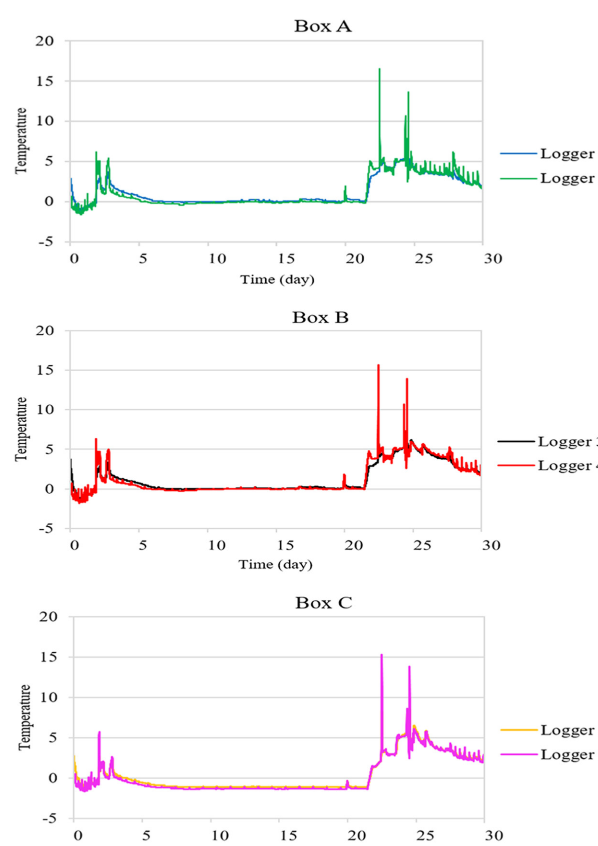

Fig. 8. Temperature profile of packaging samples.

Table 2. Total amount of sulfur compounds in PE and IN liners

Total sulfur compounds	Day 1	Day 3	Day 5
IN liners (ppm)	4.6^{b}	18.6^{b}	51.4^{b}
	4.9^{b}	21.9^{b}	56.0^{b}
	5.0^{b}	22.0^{b}	57.8^{b}
PE liners (ppm)	8.1^{a}	34.4^{a}	86.3^{a}
	8.3^{a}	37.5^{a}	88.7^{a}
	9.7^{a}	39.6^{a}	90.1^{a}

Note: Values in the same column followed by different letters are significantly different ($p < 0.05$) according to Ducan's multiple range tests.

to that in the PE foam liner was 67%. This result exhibits a trend similar to that of the total sulfur compound measured by the gas detector.

This experiment shows that the inorganic nanocomposite liners were more effective at adsorbing sulfur compounds than the PE foam liners over a five day period[19,20]. However, the adsorption efficiency decreased with time when the slope increased after three days (Table 2). Therefore, it is necessary to increase the adsorption capacity of the inorganic nanocomposites. In addition, a longer experiment duration with shorter measurement cycles is required to accurately identify the saturation point and the change in the adsorption rate over time. Therefore, a study of the generation and adsorption rate of sulfur compounds according to temperature and time is also needed to set the standard for the proper ripening time of kimchi packed in inorganic nanocomposite liners.

5. Visual Observation of Sulfur Adsorption

Cross-sections of the PE and inorganic nanocomposite liner samples used in GC-MS were used for further analyses (Figure 5). PE foam liners were observed to have a high concentration of micropore structures, and inorganic nanocomposite liners have large foam cells containing micron-scale particles. Subsequently, the surface of the liner was observed using SEM-EDS (Figure 4) for the detection of

Table 4. Diatomite specific surface area

Specific surface area	
BET	3.41 m^2/g
Total pore volume (p/p0 = 0.990)	0.03 cm^3/g
Mean pore diameter	30.71 nm

adsorbed sulfur particles after GC-MS. The PE foam liner surface was smooth, and no open cells were observed. Sulfur, indicated by red dots, was detected in small amounts. It appears that sulfur compounds were temporarily trapped in the micropores. On the other hand, the inorganic nanocomposite liner had a rough surface with open cells, and the sulfur density was higher even though the sulfur compounds had leaked from the open cells. That is, the adsorption of sulfur compounds is facilitated by inorganic compounds[19,20]. Table 4 indicates the total pore volume of the inorganic porous diatomite was approximately 0.03 cm^3/g, the average pore diameter was 30.72 nm, and the average specific surface area was 3.41 m^2/g.

Further studies on adjusting the addition ratio of the foaming agent to create a closed-cell structure are necessary to control the rate of gas leakage due to the partially opened-cell structure found in inorganic nanocomposite liners. Another refinement could be to address the solid surface of the liner (indicated by the red arrow in Figure 5), which was generated during the touch operation of the cooling roll. To effectively capture sulfur compounds and other gas generated from the packaged kimchi into the liner, this process requires further improvement by prolonging the cooling time. Finally, finding and applying a porous inorganic material with a larger specific surface area than diatomite can be expected to improve the adsorption performance[21].

Conclusions

In this study, the effect of a new packaging technology in which ZnO and TiO_2 were impregnated into diatomite, an

Table 3. GC-MS results of the sulfur compound of kimchi

Group	Hit name	Area			Reduction compared to PE (%)	Cas NO.
		0 day	5 day (PE)	5 day (IN)		
Sulfur compounds	Dimethyl disulfide (methyldithiomethane)	0	69,821,837	26,359,400	62.25	000624-92-0
	Allyl methyl disulfide	0	50,746,691	13,855,756	72.70	002179-58-0
	Dimethyl trisulfide (dimethyltrisulfane)	0	19,423,994	3,636,838	81.28	003658-80-8
	Allyl methyl sulfide (1-(methylthio)-2-propene)	0	16,772,959	10,456,728	37.66	010152-76-8
	Diallyl disulfide	0	4,732,592	652,307	86.22	002179-57-9
	Methyl propyl disulfide	0	2,109,689	896,487	57.51	002179-60-4
	Diallyl sulfide (thioallyl ether)	0	1,959,521	716,173	63.45	000592-88-1
	Allyl methyl trisulfide	0	1,394,622	341,261	75.53	034135-85-8
Average : 67.01%						

inorganic porous material, was analyzed to reduce the odor generated during kimchi fermentation. The GC-MS results showed that the inorganic nanocomposite liner containing nanomaterials reduced the content of sulfur compounds, which contribute to the odor of kimchi. The reduction rate was higher than 50% compared to the conventional PE foam liner. Additionally, it was found that these sulfur compounds were adsorbed within the inorganic nanocomposites in the liner.

To reduce higher sulfur concentrations in the future, it is necessary to select a foaming agent with a smaller cell structure, improve the cooling roll process, select inorganic porous materials with a larger specific surface area, and study fine powder technology.

Therefore, this study is expected to provide preliminary data for future studies on the application of various porous inorganic materials to improve the odor reduction of kimchi.

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