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LDPE composite films incorporating ceramic powder emitting far-infrared radiation for advanced food-packaging applications

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ABSTRACT: A ceramic powder that emits far-infrared radiation (FIR) was incorporated into low-density polyethylene (LDPE) via melt-compounding and subsequent melt-extrusion processes. To investigate the feasibility of as-prepared LDPE/FIR composite films for use in packaging applications, the composite films were characterized using Fourier transform infrared spectroscopy, X-ray diffraction, scanning electron microscopy, thermogravimetric analysis, differential scanning calorimetry, FIR emissivity and emissive power, antimicrobial activity assays, and storage tests. The physical properties and antimicrobial activities of the composite films were found to strongly correlate with the changes in the chemical and morphological structures that originate from different contents of FIR ceramic powder. A higher content of FIR ceramic powder in the LDPE/FIR composite film provided increased FIR emissivity and emission power of the composite and resulted in good antimicrobial activity. Storage tests also showed that incorporation of FIR ceramic powder into LDPE film was an effective method for maintaining the freshness of lettuce. Furthermore, the incorporation of FIR ceramic powder into LDPE films induced higher thermal stability and crystallinity and enhanced their barrier properties, which suggest these LDPE/FIR composite films are potential candidates for advanced packaging materials for the food and medical industries. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43102.

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INTRODUCTION

Packaging material is one of the most important factors in preventing deterioration in the quality of food products due to unfavorable environmental influences such as microbial contamination, oxygen, and moisture.[1,2] To protect food products from the external environment and to prolong their shelf life, there have been ever-increasing efforts to develop and modify plastic packaging materials with high thermal and mechanical properties, good gas and moisture barrier properties, and excellent antibacterial activity.[1–3] Specifically, common plastic films containing various inorganic fillers have been developed as new packaging materials with new features and functionalities or with improvements to existing characteristics.[3]

Among inorganic fillers, materials that emit far-infrared radiation (FIR) provide electromagnetic waves with wavelengths of 3 μm to 1 mm that can penetrate relatively deeply into most biological materials, exert strong rotational and vibrational effects at the molecular level, and transform light energy into heat energy without degrading or destroying the skin surface.[3–6] Food and other organic materials exhibit the highest absorption at wavelengths of 3–5 μm.[4] Because of these properties, FIR materials that include ceramics, metal oxides, and glasses have been applied to medical and health care uses, kitchen utensils, textiles, construction materials, and bath supplies.[3,7]

To use FIR materials in packaging materials, they must be applied by incorporation into common plastics such as polyethylene and polypropylene via melt-extrusion processes[3,4] or as a coating on substrates such as paper or plastic films.[8] However, the application of FIR materials to food packaging to maintain the freshness of foodstuffs has not yet been widely tested or comprehensively investigated.[3] In this study, four different LDPE and FIR (LDPE/FIR) composite films were prepared with a laboratory-scale twin-screw extruder. To investigate the feasibility of LDPE/FIR composite films as packaging materials, their physical properties, including thermal stability, mechanical strength, and moisture/gas barrier properties, were characterized as a function of FIR content. In addition to assaying the

antimicrobial activity of the films, the use of flexible film packaging was assessed for quality change based on the appearance of lettuce stored at 20°C.

EXPERIMENTAL

Materials

FIR ceramic powder composed of SiO_2 and Al_2O_3 as the main components and P_2O_5, TiO_2, K_2O, and Na_2O as minor components was purchased from Bio Ceramic Co. (Sungnam, Korea). Low-density polyethylene (LDPE), Lutene LB7500, with a melt-flow index (MFI) of 7.5 g/10 min (ASTM D1238) and a density of 0.918 g/cm^3 was purchased from LG Chemical Co. (Yeosu, Korea). Maleic anhydride grafted polyethylene (PE-g-MA), used as a compatibilizer, was purchased from Sigma-Aldrich (Yongin, Korea) All chemicals were used as received.

LDPE/FIR composite films were processed with a laboratory-scale twin-screw extruder (BA-19, BauTek Co., Uijeongbu, Korea) with a length/diameter (L/D) ratio of 40:19. Before melt-compounding and extruding, all of the ingredients were dried at 100°C for 24 h to remove water. LDPE/FIR composite films with FIR ceramic loadings of 0, 1, 3, and 5 wt % were prepared in the presence of 3 wt % PE-g-MA. The weighed materials were placed in a pouch and were mixed by shaking before the melt extrusion. The extruder was set to 170°C for the header, 170°C for zones 1 to 6, and 120°C for the feed zone. The barrel pressures were 5.2 kg_f/cm^2 for the melt-compounding process and 4.65 kg_f/cm^2 for the extrusion process. The prepared composite film samples were labeled LDPE/FIR-0, LDPE/FIR-1, LDPE/FIR-3, and LDPE/FIR-5 for 0, 1, 3, and 5 wt % FIR ceramic powder loadings, respectively. The composite films were maintained at a thickness of approximately 50 ± 5 μm to accommodate evaluation of their physical properties.

Characterization

Fourier Transform Infrared Spectroscopy. To investigate the interfacial interaction between LDPE and FIR ceramic powder in the composite films, Fourier transform infrared spectroscopy (FTIR) spectra were recorded on a Spectrum 65 FTIR spectrometer (Perkin Elmer, MA, Waltham) with a resolution of 16 accumulated scans over the range 4000–400 cm^{-1}.

Scanning Electron Microscopy. The dispersion state of FIR ceramic powder in the LDPE matrix was examined using a Quanta FEG250 scanning electron microscope (SEM; FEI Co., OR, Hillsboro). All samples were coated with a thin layer of platinum/palladium (Pt/Pd) for 60 s in a vacuum chamber before imaging.

Wide-Angle X-ray Diffractometry. To investigate the morphologies of the composite films, wide-angle X-ray diffractometry (WAXD) patterns were collected on a D/MAX-2500H X-ray diffractometer (Rigaku Co., Tokyo, Japan) with a nickel filter and a Cu$K\alpha$ ($\alpha = 1.5406$ Å) radiation source. The radiation source was operated at 40 kV and 40 mA, and all data were collected in the 2θ range from 2 to 60° at 0.02° intervals with a scan speed of 1.0°/min at room temperature.

Thermogravimetric Analysis and Differential Scanning Calorimetry. The thermal properties of LDPE/FIR composite films were assessed by thermogravimetric analysis (TGA 4000, Perkin Elmer Co.) and differential scanning calorimetry (DSC; Q10, TA Instrument Co., DE, New Castle). Both measurements were performed with a heating rate of 20°C/min under a nitrogen atmosphere.

Mechanical Properties. The mechanical properties of LDPE/FIR composite films were measured using a universal testing machine (OM100T, Qmesys Co., Kwangmyeong, Korea). All measurements were carried out for 10 samples at room temperature. The extension rate was maintained at 500 mm/min, and the load cell used was 20 kg_f with a gauge length of ~8 mm. The dimensions of the sheet used were 2.5 cm × 10 cm (width × length).

Barrier Properties. Related to the barrier properties of the composite films, the oxygen transmission rate (OTR) and water vapor transmission rate (WVTR) were measured with an OTR 8001 oxygen permeability tester and a WVTR 7001 water vapor permeation analyzer (Systech Instruments, IL, Johnsburg), respectively. OTR tests were carried out at 23°C and 0% relative humidity. WVTR tests were carried out at 90% relative humidity and 37.5°C, and the data were directly obtained from the phosphorous pentoxide (P_2O_5) moisture sensor. Both tests were performed in triplicate sets and averaged.

Far-Infrared Radiation Properties. The far-infrared emissivity and emissive power of the FIR ceramic powder and the composite films were examined in the wavelength range of 5 to 20 μm at 37°C using an FTIR (MIDAC 2400-C, Midac Co., CA, Westfield).

Antimicrobial Activities and Storage Tests. The antimicrobial activities of LDPE/FIR composite films were investigated according to the JIS Z 2801:2000 standard.[9] The tested microbes were strains DH5α *Escherichia coli* (*E. coli*) as a target Gram-negative organism and KCCM 11335 *Staphylococcus aureus* (*S. aureus*) as a target Gram-positive organism. The *E. coli* and *S. aureus* were individually grown on a MacConkey agar plate and a trypticase soy agar plate at 38°C for 24 h, respectively. A single colony was transferred into a 10 ml aliquot of nutrient broth or trypticase soy broth. Both sides of the LDPE/FIR composite films were sterilized using a UV-A light for 2 h and were inoculated with the cultured broth. Next, the microbial inoculum was covered with a thin sterile film, and the samples were incubated in a chamber at 38 ± 1°C and 90% humidity for 24 h. After incubation, the samples were washed with 30 mL of neutralizer, and the colony-forming units (CFUs) were determined. The antimicrobial rate (R) was calculated using the following equation:

$$R(\%) = (B-C)/B \times 100 \quad (1)$$

where B is the number of CFUs of viable microbial cells of the control LDPE/FIR-0 sample, and C is the number of CFUs for the LDPE/FIR composite film after 24 h. The test was performed in triplicate sets, and the results were averaged.

Plastic bags prepared using as-prepared LDPE/FIR composite films were used in assessing the change in freshness of lettuces simply by their appearance during storage at 20 ± 2°C and 65% relative humidity for 7 days.

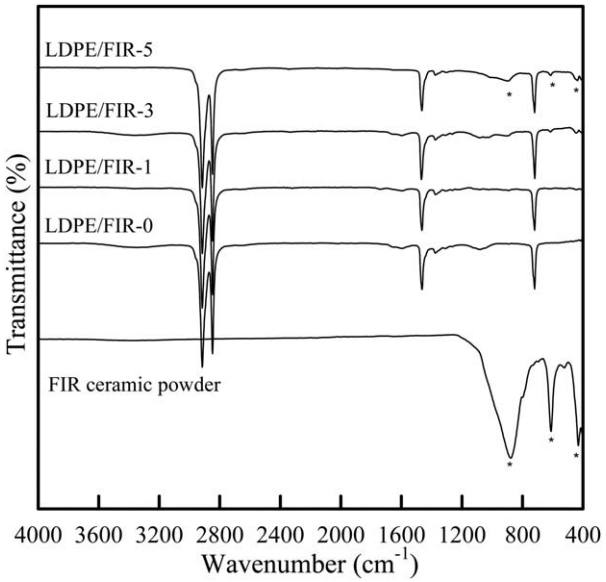


Figure 1. FTIR spectra of FIR ceramic powder and LDPE/FIR composite films.

RESULTS AND DISCUSSION

Preparation of LDPE/FIR Composite Films

To examine the effectiveness of FIR ceramic powder as a filler for active antimicrobial packaging materials, four different LDPE/FIR composite films with 0–5 wt % FIR ceramic powder were prepared by melt-compounding and extrusion processes.[3,4,10] The interfacial interaction between the LDPE matrix and FIR ceramic powder was investigated by FTIR analysis, as shown in Figure 1. Pure LDPE showed several characteristic peaks at 2847 cm^{-1} (—CH_3 symmetric stretching), 2913 cm^{-1} (—CH_2— asymmetric deformation), 1470 cm^{-1} (—CH_2— bending deformation), 1376 cm^{-1} (—CH_2— symmetric deformation), and 729 cm^{-1} (—CH_3 rocking vibration). The LDPE/FIR composite films showed similar FTIR spectra except for three characteristic bands of the FIR ceramic powder in the lower frequency range. Other than characteristic bands of the FIR ceramic powder, however, there were no appreciable changes in the intensities or positions of the characteristic LDPE peaks, suggesting low interfacial interaction between the LDPE and the FIR ceramic powder.[10,11]

Morphology

To investigate the morphology of LDPE/FIR composite films, both SEM and WAXD analyses were performed, which are depicted in Figures 2 and 3, respectively. The FIR ceramic powder in this study consisted of multiple mineral oxides and mineral salts and had particle sizes in the range of 0.3–10 μm, as shown in Figure 2(a). The pure LDPE film showed a relatively smooth appearance both on the top and fractured surfaces. With increasing FIR ceramic powder content, however, the appearance of white dots seemed to increase, and their arrangement in the LDPE became increasingly irregular. Additionally, there existed some agglomerations of FIR ceramic powder. This poor dispersion of FIR ceramic powder in LDPE may be related to the microsized FIR ceramic powder and low interfacial interactions between LDPE and FIR ceramic powder, as described in the FTIR analysis.

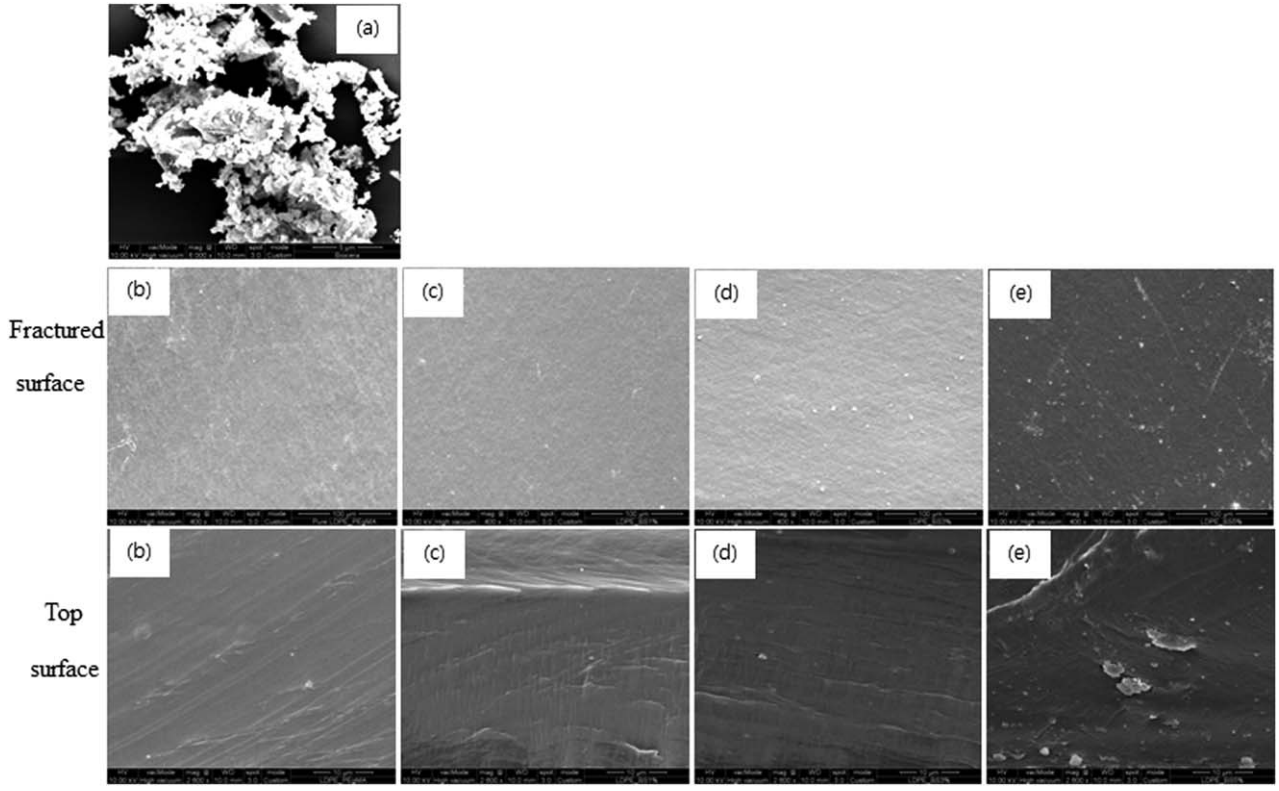


Figure 2. SEM images of (a) FIR ceramic powder, (b) LDPE/FIR-0, (c) LDPE/FIR-1, (d) LDPE/FIR-3, and (e) LDPE/FIR-5.

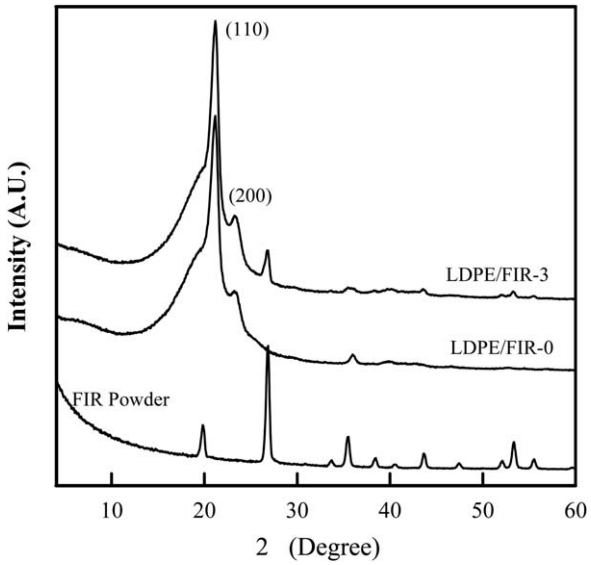


Figure 3. WAXD patterns of (a) FIR ceramic powder, (b) LDPE/FIR-0, and (c) LDPE/FIR-3 films.

Figure 3 shows WAXD patterns of (a) FIR ceramic powder, (b) pure LDPE (LDPE/FIR-0), and (c) LDPE/FIR-3 composite film. The FIR ceramic powder showed several peaks at 20.0°, 26.6°, 33.7°, 35.4°, 38.4°, 40.6°, 43.5°, 47.5°, 52.1°, 53.3°, 55.5°, and 59.7°. As mentioned above, the FIR ceramic powder was a mixture of SiO_2, Al_2O_3, P_2O_5, TiO_2, K_2O, and Na_2O, and consequently it showed relatively complex WAXD patterns. However, the major SiO_2 component showed characteristic peaks at 20.0° and 26.6°, corresponding to the (100) and (101) crystal planes, respectively, which can be readily identified in Figure 3(a). Pure LDPE showed an amorphous halo in the range of 15–30° (2θ), one sharp diffraction peak at $2\theta = 21.3°$, one slight indication at $2\theta = 23.5°$, and several small diffraction peaks in the range of 25–60°. Two diffraction peaks observed at $2\theta = 21.3°$ and 23.5° were attributed to the diffraction peaks of the (110) and (200) crystal planes of pure LDPE, respectively.[12–14] Compared to pure LDPE, the LDPE/FIR-3 composite film showed nearly the same diffraction pattern in the range of $2\theta = 5–25°$, a slight increase in the peak at $2\theta = 21.3°$, and several peaks in the range of $2\theta = 25–60°$, likely due to the loading of FIR material, which may indicate that although the FIR material did not significantly change the morphological structure of pure LDPE in the film, the crystalline structure of the pure LDPE film slightly increased with the incorporation of FIR ceramic powder.

Thermal Properties

To investigate how the content of FIR ceramic powder affects the thermal properties of LDPE film, DSC and TGA analyses were performed in a nitrogen atmosphere. As shown in Figure 4(a), pure LDPE showed an endothermic peak at 106.3°C corresponding to the melting of pure LDPE, which remained constant with varying compositions of the composite films. However, the melting enthalpy apparently increased with increasing FIR ceramic powder, resulting in an increase in the crystallinity of the LDPE/FIR composite films. As shown in Table I, the calculated crystallinity of the composite films increased from 35.3% to 54.4%, likely due to the addition of FIR powder. The TGA thermograms in Figure 4(b) show that all of the composite films had a similar one-step decomposition pattern occurring at approximately 430–530°C, which indicates that the presence of FIR ceramic powder does not significantly influence the thermal-degradation pattern of LDPE composite films. However, the thermal-decomposition temperatures ($T_{5\%}$ and $T_{10\%}$) apparently increased, indicating improvement in the thermal stability of the LDPE matrix from incorporation of FIR ceramic powder.

Mechanical Properties

The mechanical properties of the four LDPE/FIR composite films were investigated using a universal testing machine, as shown in Figure 5. The introduction of FIR ceramic powder into pure LDPE greatly changed the mechanical properties of the pure LDPE film. The tensile strength of pure LDPE increased with increasing content of FIR ceramic powder,

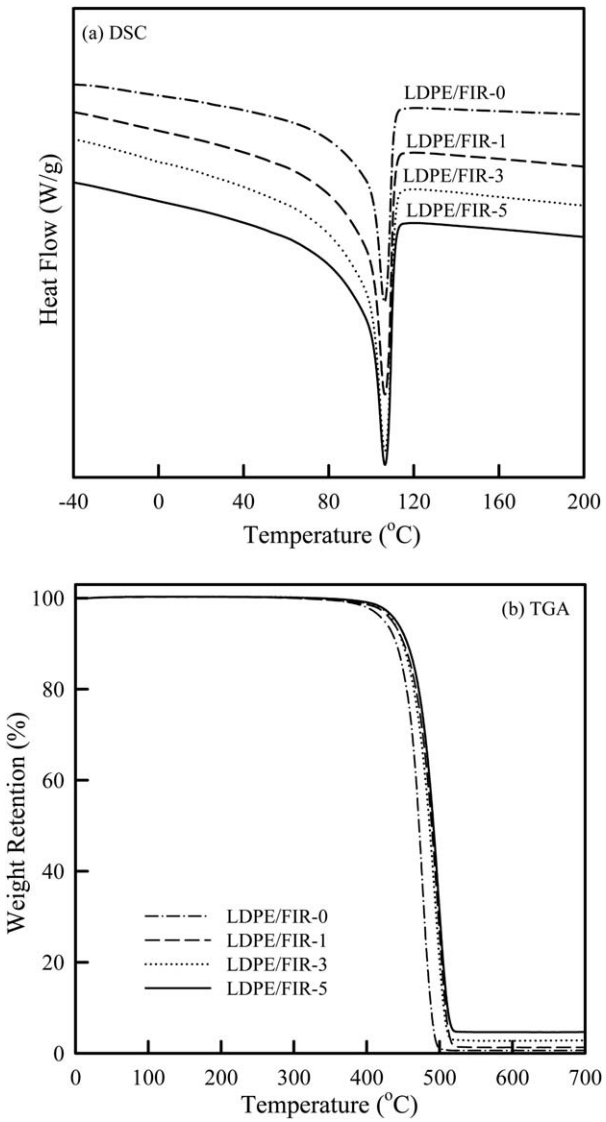


Figure 4. (a) DSC and (b) TGA thermograms of LDPE/FIR composite films.

Table I. Thermal Properties of LDPE/FIR Composite Films

	TGA			DSC		
Sample code	$T_{5\%}$ (°C)[a]	$T_{10\%}$ (°C)[a]	Residues (%)[b]	T_m (°C)[c]	ΔH_m (J/g)[d]	X_c (%)[e]
LDPE/FIR-0	424.0	440.7	0.8	106.3	90.1	30.7
LDPE/FIR-1	434.5	451.2	1.4	106.8	119.1	40.6
LDPE/FIR-3	435.9	449.5	3.0	106.5	125.7	42.9
LDPE/FIR-5	440.2	455.9	4.8	106.5	135.1	46.1

[a] 5% and 10% weight loss temperature measured by TGA.
[b] Residue percent at 650°C measured by TGA.
[c] Melting temperature measured by DSC.
[d] Melting enthalpies of the composite films measured by DSC.
[e] % crystallinity of the composite films: % crystallinity = $\Delta H_m/\Delta H_m^\circ \times 100$, where ΔH_m is the melting enthalpy of the blend films and ΔH_m° is the melting enthalpy for the 100% crystalline LDPE sample (293 J/g).

whereas the elongation at break decreased. The tensile strength was 1.0 MPa for pure LDPE film, which increased substantially with the 3 wt % FIR loading and then decreased slightly or stayed constant with increasing content of FIR ceramic powder. However, the elongation of the LDPE/FIR composite films showed the trend opposite to tensile strength, a substantial decrease to 3% FIR content and then a slight increase with higher FIR ceramic content. This result might be ascribed to the introduction of FIR ceramic powder and the interfacial interactions between the LDPE and FIR ceramic. Similar to our results, a reduction in elongation at break of composites with increasing inorganic filler content, irrespective of filler particle size and shape, has been reported by Ismail *et al.* and Onuegbu *et al.*[15,16]

However, the introduction of a relatively low concentration of FIR ceramic powder would endow the composite films with a reinforcing effect compared to the pure LDPE matrix, whereas a high content of FIR ceramic powder induces poor dispersion in the LDPE matrix, resulting in an adverse effect on film tensile strength and elongation.

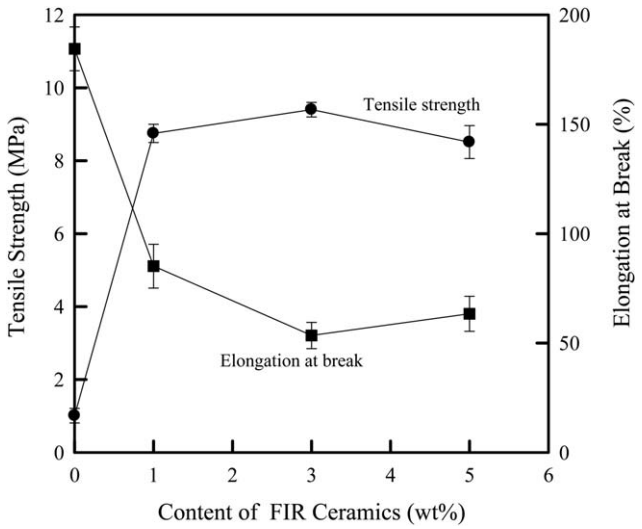


Figure 5. Tensile strength and elongation at break of LDPE/FIR composite films.

Barrier Properties

One of the critical parameters for determining the utility of a material for food packaging applications is the barrier property.[1,2] Good barrier properties against oxygen and moisture can extend the shelf life of food. LDPE, as a representative thermoplastic, is well known and widely applied in flexible food packaging, due to its good water vapor barrier properties. In composite films of polymer and inorganic filler, the barrier properties are strongly related to the chemical structure and morphology that basically originate from the composition and dispersion of filler in the polymer. To examine the effect of FIR ceramic powder on the water vapor and oxygen barrier properties of the LDPE/FIR composite films, WVTR and OTR were measured, as shown in Figure 6.

As shown in Figure 6, the LDPE/FIR-0 film had a WVTR value of 10.5 g/m^2/day, and the LDPE/FIR composite films exhibited values in the range of 8.9–10.0 g/m^2/day, indicating relatively lower values than pure LDPE film. It was observed that the OTR values of the composite films were more strongly dependent upon the FIR ceramic content. The pure LDPE film showed an OTR value of 8404 cc/m^2/day, and the composite films exhibited values in the range of 4149–4934 cc/m^2/day. Thus, the

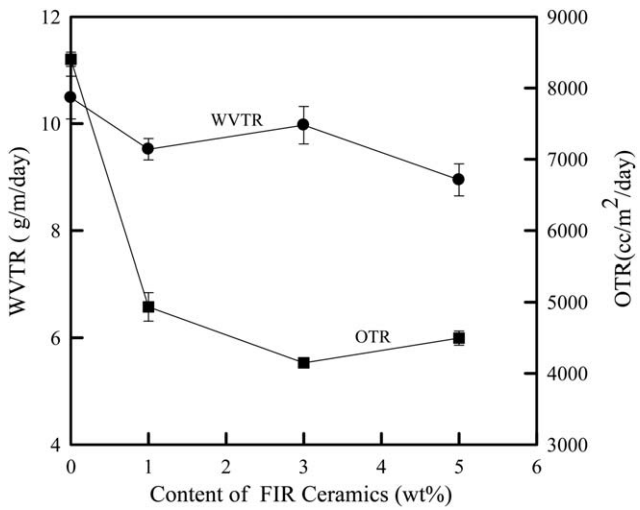


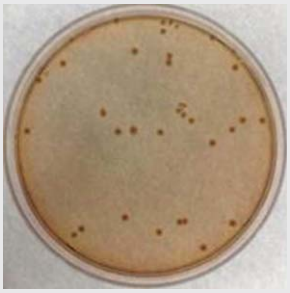
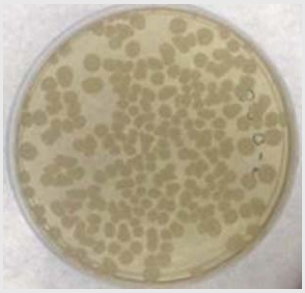
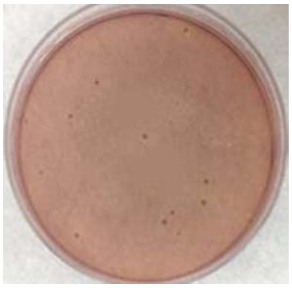
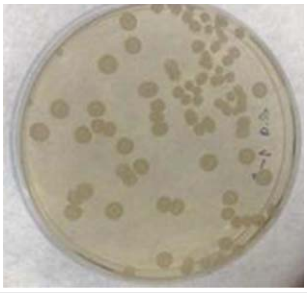
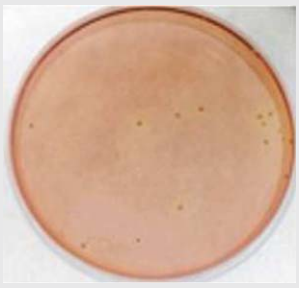
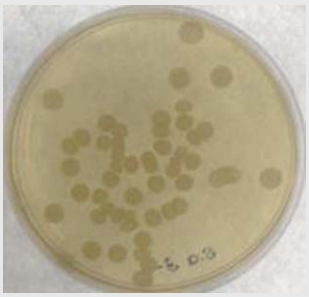
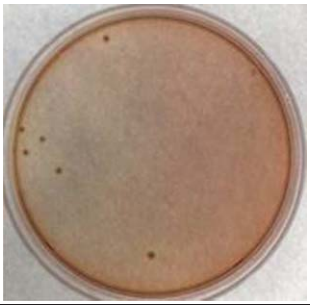
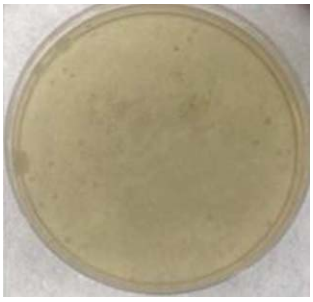
Figure 6. WVTR and OTR of LDPE/FIR composite films.

Table II. FIR Emissivity and Emissive Power of FIR Ceramic Powder and LDPE/FIR Composite Films

Sample code	Emissivity of FIR	Emissive power (W/m^2)
LDPE/FIR-0	0.87	3.34×10^2
LDPE/FIR-1	0.87	3.37×10^2
LDPE/FIR-3	0.88	3.40×10^2
LDPE/FIR-5	0.89	3.42×10^2
FIR ceramic powder	0.91	3.50×10^2

incorporated FIR ceramic powder acts as an effective barrier filler to water vapor and oxygen. The OTR of the composite film with 3 wt % FIR ceramic powder was greatly suppressed by 44.9% relative to pure LDPE film. Compared to pure LDPE, the enhanced barrier properties of the LDPE/FIR composite films originate with the loading of FIR ceramic powder, which may affect the morphology of the composite films. As described in the DSC and WAXD analyses, composite films with a relatively high content of FIR materials exhibited an increased crystalline phase and a more ordered film structure. This molecular ordering and dense structure in the composite films would act as a barrier to oxygen and water vapor. Additionally, introducing

Table III. Antimicrobial Activities of LDPE/FIR Composite Films

Sample code	*E. coli*	%*R*	*S. aureus*	%*R*
LDPE/FIR-0		—		—
LDPE/FIR-1		99.9		2.6
LDPE/FIR-3		99.9		52.6
LDPE/FIR-5		99.9		91.8

[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

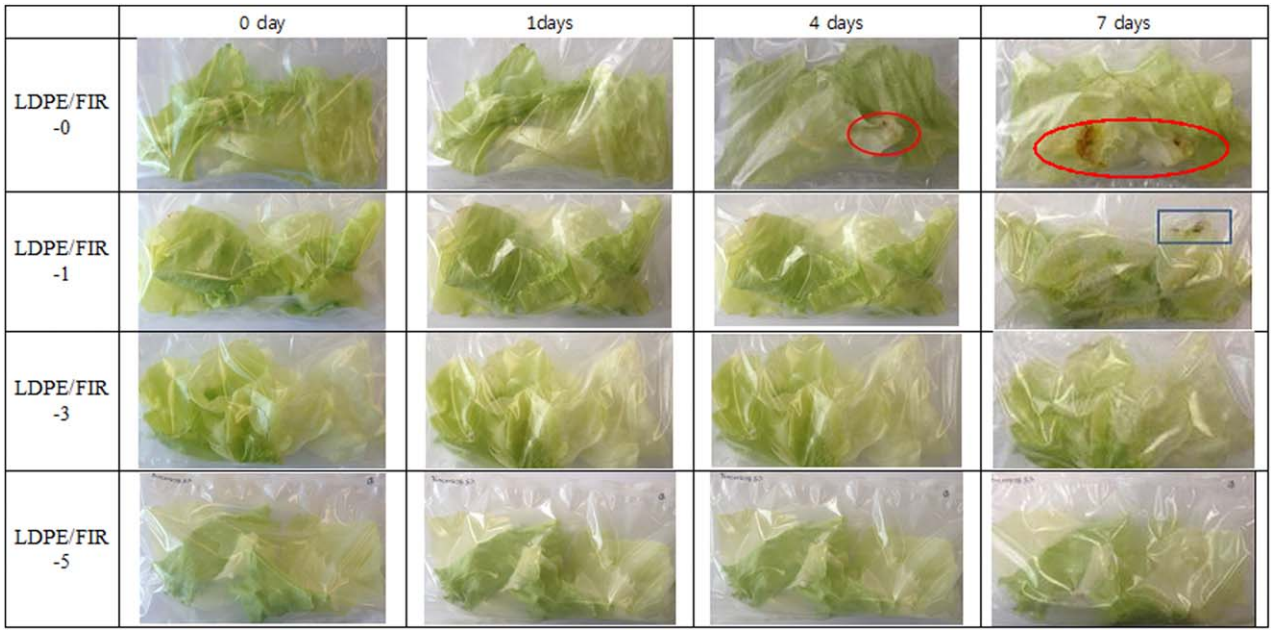


Figure 7. Photographs of lettuces packaged with composite films during storage at 20°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

FIR ceramic powders as inorganic filler into an LDPE film somewhat complicates the paths of diffusing molecules, resulting in enhanced barrier properties for the composite films.[2,17,18]

FIR Emission and Antimicrobial Activities

The FIR radiation emissivity and emissive power of the FIR ceramic powder and composite films were determined in the wavelength range of 5–20 μm at 37°C using an FTIR spectrometer, and the results are summarized in Table II. The FIR ceramic powder exhibited a FIR emissivity of 0.91 and an emissive power of 3.51×10^2 W/m^2. The FIR radiation properties in the composite films were dependent upon their composition. As expected, all of the composite films showed lower values than the FIR powder; however, as the content of FIR ceramic powder was increased, the emissivity and emissive power of the composite films increased. This result implies that LDPE/FIR composite films may have high potential in food-packaging applications that demand antibacterial activity.

The antimicrobial activities of as-prepared LDPE/FIR composite films were investigated according to the JIS Z 2801 method, and the results are summarized in Table III. *E. coli* and *S. aureus* were chosen as the target Gram-negative and Gram-positive microorganisms, respectively. Each sample was compared with a control sample, LDPE/FIR-0, that contained no FIR ceramic powder. According to Table III, the percent reduction (%*R* value) of microorganisms was strongly dependent on the FIR ceramic content and the type of microorganism; the %*R* value increased with increasing content of FIR-emitting material. Pure LDPE film showed a 0% *R* value for the two different microorganisms. In comparison, LDPE/FIR composite films exhibited better antibacterial activity than did pure LDPE. In addition, the antibacterial activities of the composite films were definitely more prominent against *E. coli* than *S. aureus*. Although the LDPE/FIR composite films with 1–5% FIR ceramic exhibited a 99.9% reduction in *E. coli*, the LDPE/FIR-5 composite film with the highest content of FIR ceramic powder gave merely a 91.8% reduction in *S. aureus*. This result may be related to the thicker peptidoglycan cell membrane of the Gram-positive bacteria *S. aureus*, which makes it more difficult to be attacked and penetrated.[12,19] The FIR ceramic powder, which is composed of SiO_2, Al_2O_3, TiO_2, and so on, can act as an antibacterial agent to kill bacteria by direct contact with the microorganism and indirectly by FIR radiation.[3–5] FIR materials have a wavelength around 10 μm, encouraging antibiosis and deodorization and potentially extending shelf life.[3] However, the exact mechanism behind the excellent antimicrobial activity of FIR materials and their efficacy for food quality have not yet been comprehensively tested or understood.[3] Lee *et al.* demonstrated the effectiveness of an FIR ceramic powder in paperboard in maintaining the freshness of mandarin oranges.[8] Lin also showed the prolonged shelf life of meat by using an FIR ceramic sheet package.[4]

Freshness of Lettuce

To demonstrate the effectiveness of the as-prepared LDPE/FIR composite films for food packaging, the freshness of lettuce was evaluated by measuring its appearance. In this study, the change in quality of lettuce packaged using composite films containing different amounts of FIR ceramic powder was tested by appearance for triplicate samples at 20°C. Figure 7 shows representative photographs of each sample change by day. In the deterioration in quality that rapidly advances due to cutting and handling, the browning of lettuce appears to be the most important factor depreciating consumer interest in the product.[20] Samples packaged with pure LDPE film (LDPE/FIR-0) showed an apparent color change to brown during the 7 days. Compared to the pure LDPE film, the LDPE/FIR-1 composite film showed slight changes in color, while the LDPE/FIR-3 and LDPE/FIR-5 composite films showed no significant differences in color as compared to the initial visual quality of the lettuce. No color change

to brown was observed in samples packaged in the LDPE/FIR composite films with a relatively high content of FIR material, which indicates that incorporation of FIR ceramic powder could be an effective way to minimize quality change and to extend the shelf life of lettuce. However, further, more extensive studies are required to examine the effect of FIR materials for extending shelf life and product quality with respect to color, weight retention, pH, decay rate, and sensory evaluation.[4,20]

CONCLUSIONS

Four different LDPE/FIR composite films with 0–5 wt % FIR ceramic powder were prepared, their physical properties and antimicrobial activity were examined, and storage tests were performed. While LDPE/FIR composite films exhibited good dispersion at relatively low FIR levels, the interfacial interaction between LDPE and FIR material was weak, which induced some agglomeration of FIR ceramic powder. TGA and DSC analyses showed that the thermal stability and crystallinity of the films improved with FIR ceramic powder content. Compared to pure LDPE film, the composite films showed an apparent enhancement in moisture and oxygen barrier properties. Specifically, the OTR of the 3 wt % FIR ceramic powder composite film was greatly suppressed by 44.9% relative to pure LDPE film. A higher content of FIR ceramic powder in the LDPE/FIR composite films led to higher FIR emissivity and increased emission power of the composite films. Antimicrobial evaluation showed that LDPE/FIR composite films exhibited good antimicrobial activities against both Gram-negative and Gram-positive microorganisms through direct contact or FIR radiation. The properties of the composite films were strongly related to the altered chemical structure and morphology that arose from the addition of FIR ceramic powder. The enhanced thermal stability, moisture/oxygen barrier properties, and antimicrobial properties make LDPE/FIR composite films potential candidates for versatile, antimicrobial food-packaging applications. However, further studies are required to increase the compatibility and dispersion of FIR ceramic powder in the LDPE matrix in order to maximize their performance and expand their applications to medical packaging applications.

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