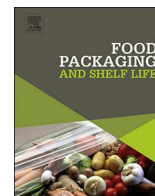




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Contents lists available at ScienceDirect

Food Packaging and Shelf Life

journal homepage: www.elsevier.com/locate/fpsl

Impact of migration test method on the release of silver from nanosilver-polyethylene composite films into an acidic food simulant

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ARTICLE INFO

Keywords:

Nanosilver
Polyethylene
Packaging
Migration
Release
Food simulant
ICP-MS

ABSTRACT

Impact of migration test method on the release of nanocomponent from nano-polymer composite films intend for food contact is seldom addressed. In this study, release of silver from nanosilver-polyethylene composite films into 3% acetic acid under different migration test methods were investigated, with the aim to understand the effect of exposure condition on silver release. The following test protocols were designed for the experiment: single-sided contact versus two-sided contact, more versus fewer cut edges, and glass migration cell versus metal one. The results show that samples under two-sided contact had higher silver release than that under one-sided contact. Besides, increasing the number of cut edges largely increased the release of silver, while using metal instead of glass migration cell significantly reduced the release. Furthermore, partially floated films have lower silver release than that of totally immersed films. As a conclusion, impact of migration test method on the release of nanocomponent is different from that on the migration of common migrants and therefore, exposure condition should be carefully taken into account in the design of migration test for nanosilver-polyethylene composite films to minimize any potential negative impact on obtaining comparable results.

1. Introduction

Plastic has been widely accepted as food packaging material to protect food from chemical and microbiological spoilage during production, transportation, and storage for their excellent properties, i.e. low weight, strong, lustrous, odorless, waterproof, gas-barrier and other characteristics (Kao, 2012). However, additives (Lin, Liang, Su, Shan, & Wang, 2017), residuals from raw materials (Torres et al., 2014), neo-forming molecules (Koster et al., 2014) like by-products created during the synthetic process within plastic food packaging may have the possibility to migrate into food when in contact with food. Except for toxicity, risks posed by those potential migrants are also related to their exposure to human beings. The exposure is the result of their migration into food and the consumption of that food (Rijk & Veraart, 2010). Migration test is therefore of great importance to check the safety of plastic food packaging and to check whether or not it complies with the legislations.

Detailed migration test method have been described in different standards and guidelines, such as *Materials and articles in contact with foodstuffs-Plastics* (BS EN 1186-1:2002), *Materials and articles in contact*

with foodstuffs-Plastics substances subject to limitation (BS EN 13130-1:2004), *Technical guidelines for Migration Testing Guideline (Draft)* (European Commission Joint Research Centre, 2014), and *Guidance for Industry Preparation of Premarket Submissions for Food Contact Substances Chemistry Recommendations* (U.S. Department of Health and Human Services, 2007). In general, common migrants (additives, residuals, and neo-forming molecules) are generally small and their migration is usually controlled by their diffusion within packaging materials (Lee, Yam, & Piergiovanni, 2008). If a polymer is characterized by low diffusivity, like polystyrene, and the exposure time is short, migration only occurs from a thin surface layer and total immersion goes for two-sided testing. Linssen et al. (Linssen, Reitsma, & Roozen, 1991) have investigated the effect of migration test methods on migration of styrene monomer from polystyrene packaging materials. It was shown that total immersion exposure resulted in the highest migration, while the use of sample cell (less cut edge) gave the lowest migration. Compared to polystyrene, diffusion within polyethylene is faster and within a rather short time also one-sided testing may include material from the other side within an appropriate thickness. Migration of nano-materials from nanocomposites, however, is different from that of common migrants. It

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<http://dx.doi.org/10.1016/j.fpsl.2017.08.012>

Received 14 June 2017; Received in revised form 9 August 2017; Accepted 17 August 2017
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is not only controlled by diffusion but also dissolution and desorption (Duncan & Pillai, 2015). Hence, migration test methods may have different effects on the migration of nano-materials compared to that of common migrants.

With the properties of a broad-spectrum antibacterial activity, high temperature stability and low volatility, nanosilver is well received to be incorporated into food packaging with the aim to realize antimicrobial function (Echegoyen & Nerín, 2013). However, nanosilver particle itself and silver ion may release into foodstuffs during the contact. They bring about antibacterial effect on foodstuff, however, also toxic effect on human health. What's worse is that the cytotoxic level of nanosilver or silver ions is even much higher than the antibacterial level (Guo, Yuan, Lu, & Li, 2013). Therefore, Migration test is so crucial that it must be performed during the introduction process of nano-packaging (Cushen, Kerry, Morris, Cruz-Romero, & Cummins, 2013).

Up to now, there are many studies on release of silver from nanosilver-polymer composite materials. Some researchers conducted migration test by filling the packaging with food simulants (single-sided contact) (Echegoyen & Nerín, 2013; Mackevica, Olsson, & Hansen, 2016; Metak, Nabhani, & Connolly, 2015) or soaking a piece of packaging film into food simulants (two-sided contact) (Addo Ntim, Thomas, Begley, & Noonan, 2015; Artiaga, Ramos, Ramos, Camara, & Gomez-Gomez, 2015; Bott, Störmer, & Franz, 2014; Song, Li, Lin, Wu, & Chen, 2011); others, however, designed the experiment by soaking several small pieces of specimen into food simulants (Fernández, Soriano, Hernández-Muñoz, & Gavara, 2010; Su et al., 2015), which increased the number of cut edge in contact with food simulants. It is reasonable to believe that those different exposure conditions will affect the release of nanosilver from food contact materials into food simulants as mentioned above. Still, as far as we know, no experimental data about effect of migration test method on silver release from food contact materials is available to the public now. The aim of the present study is to provide valuable experimental data in this field.

2. Materials and methods

2.1. Instrumentation

Neat polyethylene film was blown using a lab scale blowing machine (FYC-25; Jinfangyuan Co. Ltd., Guangzhou, China). An electronic analytical balance with 0.1 mg resolution (AG 204; Mettler Toledo, Zurich, Switzerland) was employed to weight samples in determining total Ag content in samples. Thickness of all films was measured by thickness gauge (DRK203B; Drick, Jinan, China). A microcontroller digital electric heating plate (XMTD-701; Xuanyuan, Yancheng, China) was employed to digest plastic films after adding 15 mL mixed acid. After the digestion, an inductively coupled plasma atomic emission spectrometry (ICP-AES; iCAP 6500; Thermo, California, USA) was applied to determine total Ag content in plastic films. The ICP-AES was operated under the following conditions: plasma power 1150 W, cool gas flow 14 L/min, auxiliary gas flow 0.6 L/min, nebulizer gas flow 0.5 L/min, plasma view axial, analytical wavelength of Ag 328.68 nm.

Two incubators (DHG 9140A; Jinghong, Shanghai, China) were used to conduct the migration test. Determination of the Ag in the food simulant (3% acetic acid) was carried out by inductively coupled plasma mass spectrometry (ICP-MS; iCAP Qc; Thermo, California, USA) which was equipped with a polytetrafluoro ethylene (PFA) nebulizer, a quartz spray chamber and Peltier-cooled cyclonic chamber. The ICP-MS was operated under the following conditions: plasma power 1548.6 W, cool gas flow 13.9 L/min, auxiliary gas flow 0.8 L/min, nebulizer gas flow 1.06 L/min, dwell time 0.2 s, measurement mode: kinetic energy discrimination (KED).

2.2. Reagents and samples

Nitric acid (HNO₃), perchloric acid (HClO₄), and acetic acid (CH₃COOH) of analytical grade were purchased from Guangzhou Chemical Reagent Factory (Guangzhou, China). All solutions were prepared with distilled water obtained from a Milli-Q water purification system (Advantage A10; Millipore, Boston, USA). Stock standard solutions of silver (Ag), rhodium (Rh; internal standard) and other 25 elements including antimony (Sb), aluminium (Al), arsenic (As), beryllium (Be), cadmium (Cd), chromium (Cr), mercury (Hg), lead (Pb), manganese (Mn), nickel (Ni), selenium (Se), thallium (Tl), barium (Ba), cobalt (Co), copper (Cu), iron (Fe), molybdenum (Mo), thorium (Th), uranium (U), vanadium (V), zinc (Zn), Bismuth (Bi), strontium (Sr), tin (Sn), lithium (Li) of 1000 µg mL⁻¹ were obtained from National Nonferrous Metals and Electronic Materials Analysis and Testing Center (Beijing, China). Working solutions of Ag for ICP-AES and ICP-MS were prepared with 5% nitric acid and 3% acetic acid, separately. Working solutions of those 25 elements for ICP-MS were prepared with 2% nitric acid. All of those working solutions were stored at 4 °C (in a refrigerator).

Two kinds of nanosilver-polyethylene composite film were provided by Anson Nano-Biotechnology Co., Ltd (Zhuhai, Guangdong, China). The thicknesses of those films were 0.011 ± 0.001 mm (named thin film) and 0.057 ± 0.005 mm (named thick film), separately. Besides, neat polyethylene granule was purchased from China Petroleum & Chemical Corporation (2420H; Maoming, Guangdong, China), and neat polyethylene film was blown by the small size blowing machine with the thickness of 0.048 ± 0.06 mm.

2.3. Determination of Ag in samples

0.1 g film samples cut into small pieces were weighted into Erlenmeyer flask. After being carbonized by putting them on the microcontroller digital electric heating plate (set at 400 °C), they were cooled for about 30 s in room temperature (about 20 °C) and 15 mL mixed acid (HNO₃: HClO₄ = 3:1, volume ratio) was then added. Finally, they were put on the heating plate (set at 250 °C) until all films were digested completely. The digested solutions were then diluted to 50 mL with distilled water after being cooled for 2 h and analyzed by ICP-AES. Unless otherwise specified, all samples were analyzed in triplicate.

For the determination of Ag in plastic, linearity and correlation coefficient were obtained by analyzing six Ag standard solutions (0.01, 0.05, 0.1, 0.2, 0.5 and 1 µg mL⁻¹) diluted with 5% HNO₃ directly by ICP-AES. Limit of detection (LOD) and limit of quantitation (LOQ) were determined as the corresponding concentrations of blank response plus 3 or 10 times standard deviation of 11 blank measurements (digested solutions taken from the PE-reference films), respectively.

2.4. Migration test

In the present experiment, five migration test methods (Fig. 1) were designed and compared to investigate impact of exposure condition on silver release from nanosilver-polyethylene composite films into a food simulant (surface-to-volume ratio: 6 dm² of surface area in contact with 1000 mL of simulant). Only 3% acetic acid was selected as the food simulant due to its higher potential to release silver from silver-nanocomposites compared to 50% ethanol observed in our previous research (Su et al., 2015). Method 1 (Two-sided contact: randomly dispersed specimens) was designed as follows: a great amount of cut-outs specimens (1 cm × 1 cm) were taken from different parts of the nanocomposite, mixed together and then 28 pieces were selected randomly. The selected specimens were put into 50 mL centrifuge tube and 47.5 mL food simulant was then added. In this case, nanosilver-polyethylene films partially float upon simulant because of its low density.

Method 2 (Two-sided contact: strung specimens) was designed as

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