



Migration assessment of silver from nanosilver spray coated low density polyethylene or polyester films into milk



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ABSTRACT

The release of silver (Ag) from an experimental nanosilver spray coating on surface treated polyester (PT) and low density polyethylene (LDPE) packaging into milk was determined. Spherical nanoparticles (NPs) with a high electron density and crystalline face centred cubic structure characteristic of silver nanoparticles (AgNPs) were characterised in milk by cryogenic transmission electron microscopy (TEM). Total Ag migration into milk over time was determined by inductively coupled plasma – atomic emission spectrometry (ICP-AES). This study identified coating treatments such as the diblock copolymer, polystyrene-block-polyethylene oxide (PS-*b*-PEO), which can be used to reduce potential migration.

1. Introduction

To feed the world's growing population in the future, developments in food production, processing, packaging and transportation are of utmost importance to reduce food waste and increase production (Floros et al., 2010). Food packaging incorporating engineered nanoparticles (ENPs), which are discrete quantities of matter with at least one dimension in the size range 1–100 nm, have the potential to satisfy these requirements by prolonging food shelf life and improving food safety (Duncan, 2011). The unique properties lent to ENPs by their small size merits their inclusion in a number of food applications (Maynard & Michelson, 2017); however, ENPs increased mobility, driven by their reduced size, raises concerns regarding potentially heightened ENP release from packaging into food and increased human exposure (Simon, Chaudhry, & Bakos, 2008). These concerns are worsened by insufficient data on ENP mode of toxicity, persistence and possible bioaccumulation (Hansen & Baun, 2012).

To fill the existing knowledge gaps, research has been directed at quantification and characterisation of migrant ENPs from commercially available and experimental food packaging nanocomposites into food simulants and real food matrices (Hannon, Kerry, Cruz-Romero, Morris, & Cummins, 2015). The migration of ENPs has been assessed for a number of nanocomposites into a range of real food matrices including;

nanosilver and nanocopper from an antimicrobial ENP filled polyethylene nanocomposite into chicken meat (Cushen, Kerry, Morris, Cruz-Romero, & Cummins, 2014), nanosilver from ENP filled PVC nanocomposite into chicken (Cushen, Kerry, Morris, Cruz-Romero, & Cummins, 2013), nanoclay from a biodegradable starch/clay nanocomposite into spinach and lettuce (Avella et al., 2005), and nanosilver and zinc oxide ENPs from antimicrobial ENP filled low density polyethylene (LDPE) nanocomposites into orange juice (Emamifar, Kadivar, Shahedi, & Soleimani-Zad, 2010). Nanocoatings provide many benefits compared to ENP filled films and multilayer nanocomposites which includes; simplified film conversion into nano-enabled packaging, reduced material usage and enhanced antimicrobial performance due to improved contact area between the antimicrobial and food (Mihindukulasuriya & Lim, 2014). Despite the benefits associated with improved contact between antimicrobials and food provided by nanocoatings, there is also the risk that the improved proximity of the ENPs to food may result in increased ENP migration. To the best of the author's knowledge only one study (Jokar & Abdul Rahman, 2014) has investigated the migration of ENPs from surface coatings into real food matrices. Milk is a food product where nano packaging could have industry applications for product safety and shelf life extension (Bayani Bandpey, Aroujalian, Raisi, & Fazel, 2017).

In the past, Ag coins were placed in milk to extend its shelf life

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(Alexander, 2009). In many countries there is a considerable cost to both the consumer and the environment due to milk spoilage. In 2009, approximately 3% of the 13 million tonnes of milk produced for domestic consumption in the United Kingdom was wasted during the consumer phase (Reay et al., 2012). The most common bacteria responsible for milk spoilage are psychrotrophs, predominantly *Pseudomonas* species (Ledenbach & Marshall, 2009). The antimicrobial efficacy of AgNP coatings has been demonstrated against *Pseudomonas fluorescens* (Azlin-Hasim, Cruz-Romero, Cummins, Kerry, & Morris, 2016) which makes the packaging of milk a suitable candidate for the application of novel nanosilver spray coatings.

Before products incorporating nanotechnology can be accepted for use in the global food market, gaps in knowledge regarding human exposure and toxicity must be elucidated to protect the consumer and allow nanomaterials to be regulated by competent authorities. The European Union and Switzerland have been identified as the only world regions to incorporate nano-specific provisions in food legislation (Amenta et al., 2015). Other regions such as the United States of America have provided guidance on the definition and use of ENMs in the food area in the form of guidance documents. Many products have appeared on the global market making claims on the use of nanotechnology for improved properties (Maynard & Michelson, 2017). In the European Union, only a limited number of products have been accepted for use in food contact materials on the basis of limited or negligible human exposure supported by migration studies (EFSA, 2011) and predictive migration modelling (EFSA, 2012; EFSA, 2014).

The objective of this study was to a) investigate the effect of different treatment methods on the attachment of nanosilver in an experimental food packaging coating and b) investigate the migration of Ag from an antimicrobial nanocoating into milk.

2. Materials and methods

2.1. Nanocomposite manufacture

The nanosilver spray coated LDPE and polyester (PT) films were manufactured as outlined by Hannon, Azlin-Hasim et al. (2016). The cleaning of the LDPE and PT films was carried out by immersing the films in absolute ethanol for 3 min, followed by draining and drying in an oven (Mettler, Germany) at 60 °C. In order to modify the surface of one side of the cleaned commercial LDPE (Fispak, Ireland) or PT films (GBC EZTM, ACCO Brands Europe, U.K.) of 0.04 mm thickness, surface treatments involving UV and/or surfactant were used and included: UV treatment only (PSD Pro Series, UV Source: 180–254 nm); UV + 0.4 wt.% PS-*b*-PEO; UV + 0.85 wt.% PS-*b*-PEO; UV + 0.2 wt.% Pluronic + 0.4 wt.% PS-*b*-PEO, 0.2 wt.% Pluronic + 0.4 wt.% PS-*b*-PEO, 0.85 wt.% PS-*b*-PEO + 0.2 wt.% Pluronic and UV + 0.85 wt.% PS-*b*-PEO + 0.2 wt.% Pluronic (Table 1).

Table 1

List of films and surface treatments applied.

Sample name	Film	Ag precursor concentration	No. of coatings	Treatments (Acronym)
PE/T ₀ + 3 × 2%Ag	LDPE	2% Ag	3	No treatment (T ₀)
PE/T ₁ + 3 × 2%Ag	LDPE	2% Ag	3	UV (T ₁)
PE/T ₂ + 3 × 2%Ag	LDPE	2% Ag	3	UV + 0.4% PS- <i>b</i> -PEO (T ₂)
PE/T ₃ + 3 × 2%Ag	LDPE	2% Ag	3	UV + 0.85% PS- <i>b</i> -PEO (T ₃)
PE/T ₄ + 3 × 2%Ag	LDPE	2% Ag	3	0.4% PS- <i>b</i> -PEO + 0.2% Pluronic (T ₄)
PE/T ₅ + 3 × 2%Ag	LDPE	2% Ag	3	UV + 0.4% PS- <i>b</i> -PEO + 0.2% Pluronic (T ₅)
PE/T ₆ + 4 × 3%Ag	LDPE	3% Ag	4	0.85% PS- <i>b</i> -PEO + 0.2% Pluronic (T ₆)
PT/T ₀ + 3 × 2%Ag	PT	2% Ag	3	No treatment (T ₀)
PT/T ₁ + 3 × 2%Ag	PT	2% Ag	3	UV (T ₁)
PT/T ₂ + 3 × 2%Ag	PT	2% Ag	3	UV + 0.4% PS- <i>b</i> -PEO (T ₂)
PT/T ₃ + 3 × 2%Ag	PT	2% Ag	3	UV + 0.85% PS- <i>b</i> -PEO (T ₃)
PT/T ₄ + 3 × 2%Ag	PT	2% Ag	3	0.4% PS- <i>b</i> -PEO + 0.2% Pluronic (T ₄)
PT/T ₅ + 3 × 2%Ag	PT	2% Ag	3	UV + 0.4% PS- <i>b</i> -PEO + 0.2% Pluronic (T ₅)
PT/T ₇ + 3 × 0.04%Ag	PT	0.04% Ag	3	UV + 0.85% PS- <i>b</i> -PEO + 0.2% Pluronic (T ₇)

To obtain the untreated (T₀) coated films, 2 wt.% AgNO₃ was sprayed directly onto cleaned PT and LDPE. For the UV-treated samples, the films were treated with UV/Ozone for 30 min with 0.5 l min⁻¹ flow of O₂ using a Digital UV Ozone System (PSD Pro Series, UV Source: 180–254 nm). In this study, UV/Ozone was used as a physical treatment to increase the wettability of LDPE or PT films. The LDPE or PT films were surface modified by spray coating the film surfaces using non-ionic copolymer surfactant (a Pluronic polypropylene oxide-polyethylene oxide triblock system) and/or diblock copolymer (BCP) PS-*b*-PEO to allow satisfactory adhesion of AgNO₃ for the formation of AgNPs. The Pluronic™ solution (0.2 wt.%) was spray coated on PT or LDPE film surfaces using an air brush compressor (Model AS 18-2, Air Brush Pro, U.K.) fitted with an Airbrush BD-128P Kit. The Pluronic™ coated films were then dried in an oven for 2 h at 60 °C. The PS-*b*-PEO surface modified films were created by spray coating solutions of PS-*b*-PEO in toluene (0.4 wt.% or 0.85 wt.%). All surface modified (T₁–T₅) and unmodified films (T₀) were then coated with 3 cycled coatings of an ethanolic solution of AgNO₃ (2 wt.%) followed by exposure to UV light (UV surface dose of 1.2 J cm⁻²) in an environmental chamber (Binder, Germany) for 2 h at 50% RH and 25 °C. To enable diffusion and rearrangement of the PS-*b*-PEO coated films, films were incubated in an oven for 18 h at 60 °C.

In addition to the surface modified and unmodified films coated with 2 wt.% AgNO₃, different concentrations of AgNO₃ (3 wt.% and 0.04 wt.%) and coating numbers (3 or 4 application cycle coatings) were investigated. A film representing a high AgNP concentration was obtained when 4 coatings of 3 wt.% of AgNO₃ in an ethanolic solution was spray coated on Pluronic + 0.85 wt.% PS-*b*-PEO surface modified LDPE (PE/T₆ + 4 × 3%Ag film) which was optimised for higher antimicrobial activity. A film representing a low AgNP concentration was obtained when 3 coatings of 0.04 wt.% AgNO₃ was spray coated on UV + Pluronic + 0.85 wt.% PS-*b*-PEO surface modified PT (PT/T₇ + 3 × 0.04%Ag). To reduce ionic silver (Ag⁺) to AgNPs via a photo-reduction process, all coatings were exposed to UV light (UV surface dose of 295.5 mJ cm⁻²) for 18 mins in a laminar flow (Airclean 600 PCR Workstation STAR LAB).

2.2. Inductively coupled plasma – atomic emission spectrometry

The concentration of total Ag (Ag⁺ and AgNPs) in the fully digested spray coated films or food simulants from migration studies was determined by ICP-AES analysis using the operating conditions as outlined previously by Hannon, Kerry et al. (2016). Before each analytical run the ICP-AES was calibrated using a blank and four standards diluted from a stock Ag standard (1000 mg l⁻¹ AgNO₃ in HNO₃, Elementec, Ireland). One of two calibration ranges were used depending on the predicted Ag concentrations; 10, 100, 1000 and 5000 μg l⁻¹, or 1, 5, 10 and 20 μg l⁻¹ for the migration studies or total digestions, respectively.

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