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Research of biocidal effect of corona discharges on poly(lactic acid) packaging films

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ABSTRACT

Investigation of the effect of corona discharges on the mortality of the strains of bacteria and fungus: *Salmonella enteritidis*, *Escherichia coli*, *Bacillus subtilis*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Penicillium chrysogenum* is presented. The microorganisms were placed onto a polylactide (PLA) packaging film. The surface layer of that film, with the microorganisms deposited on it, was subjected to modification by the corona treatment in the air, using the modifying unit energy (E_j) ranging from 1 to 20 kJ/m^2, and then the count of dead bacteria was determined. Microscopic images were taken to show the effect of the corona discharges on the individual microorganisms.

1. Introduction

The method of modifying polymeric materials by corona discharges and the analysis of the state of knowledge in that field have been demonstrated in many scientific publications (Żenkiewicz, 2000, 2008; Vasile, 2000; Dobrzańska-Danikiewicz et al., 2011; Żenkiewicz et al., 2012). The first reports describing the corona discharge effect on parasites and microorganisms were published in the 1980s and 1990s (Peyrous, 1986; Morar et al., 1997; Benstaali et al., 1998). The successive papers showed that the microorganism mortality caused by the corona discharges was influenced by various factors, e.g., the type of gas used while generating the discharges, modification process parameters, namely the power of the corona discharges and values of the unit energy (E_j) used for the modification (Kuzmichev et al., 2000; Pointu et al., 2005; Stepczynska et al., 2013).

However, despite a vast knowledge on modification of the surface layer of polymeric materials (mostly polyolefins) by using the corona discharge method, on the methods for sterilisation of plastic products, as well as on the effect of the corona discharges on the strains of various bacteria, the mechanism of the biocidal effect of the corona discharges is not fully understood. In the first part of the research (Stepczyńska et al., 2013), the authors tried to determine whether the corona discharges indeed caused the mortality of various strains of bacteria. Since the results of that research gave positive results, the author of the present paper has decided to investigate the effect of the corona discharges on the bacterial strains placed onto the PLA film.

Polylactide is an interesting biodegradable polymer, synthesised from renewable raw materials. It is mostly applied in medicine, however, due to its biodegradation properties, it is also used in the packaging industry as a disposable material. PLA is a polymer undergoing total biodegradation within a period ranging from half a year to two years while the biodegradation time of synthetic polymers, such as polyethylene (PE) or polystyrene (PS), ranges from 500 to 1000 years (Błędzki and Fabrycy, 1992; Doi and Steinbüchel, 2002; Pluta, 2004).

Due to favourable properties and rapid biodegradability, PLA attracts much attention among scientific and industrial communities who expect a mass application of that polymer for manufacturing food packaging. Necessary conditions of the PLA application for such purposes are adequate preparation of its surface layer for printing, gluing, or decorating processes as well as its sterilisation since the produced food packaging (not only from PLA) must be free of microorganisms.

The preparation of the surface layer of polymer films for printing, gluing, or decorating processes consists in the layer modification by, e.g., the use of the corona discharge method that is commonly applied in industry. The corona discharges occur due to the difference in the potentials created in the inter-electrode gap filled with gas (most often air) under atmospheric pressure. The generated electrons, moving within the inter-electrode gap with the kinetic energy ranging from a few to a dozen or so eV, hit the polymer chains, thus breaking some chemical bonds. Radicals created that way trigger chemical reactions, mostly oxidation, which lead to modification of the polymer surface layer. The radicals, reacting with, e.g., oxygen, ozone, OH groups, and water molecules, form polar compounds that induce changes in the surface properties of the products being modified (Żenkiewicz, 2008; Stepczyńska and Żenkiewicz, 2009).

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Scientific research is also being developed in relation to the application of the corona discharges as a method for sterilisation of packaging and medical materials (Doi and Steinbüchel, 2002; Radetić et al., 2008; Khattak and Simpson, 2010; Scholtz et al., 2010; Tallentire et al., 2010; Stepczyńska and Żenkiewicz, 2011a,b; Stepczyńska et al., 2013). A rapidly growing PLA application, mostly as a material for production of food packaging that must be sterilised, stimulates the necessity of a closer look at that phenomenon.

A dynamic development of the design of activators and no need to apply hermetic discharge chambers reduce the costs of the activators and enable the polymeric film modification to be carried out in a continuous way (Żenkiewicz and Gołębiewski, 1998; Brzeziński et al., 2009; Żenkiewicz et al., 2011). The corona discharge method can be applied to sterilise the food packaging without the need to introduce changes in the design of the already existing devices used for the corona treatment.

The unit energy (E_j) applied in the modification procedure is a basic factor that affects changes in the surface layer of PLA being modified. Its value determines the extent of modification of the PLA surface layer (Stepczyńska and Żenkiewicz, 2010, 2011a,b). Following the research performed so far, selection of an adequate E_j value was made based on the results of the experimental determination of its effect on the rate of mortality of respective bacterial strains (Stepczyńska et al., 2013).

The aim of the present paper was to show the results of the author's own research on the modification of the PLA film with deposited bacterial strains, using the corona discharge method, as well as to ascertain that the method can be utilised as a biocidal agent applied for the sterilisation of packaging materials made from that polymer.

2. Experimental

2.1. Material

The research involved polylactide (PLA) 2002 D provided by Cargill Down LLC, with the melt flow index of 4.2 g/10 min (2.16 kg, 190 °C) and density $d = 1.24$ g/cm^3. The polymer was used to form a film about 100 μm thick.

To study the biocidal activity of the corona discharges, the following bacterial strains were applied: *Salmonella enteritidis*, *Escherichia coli*, *Bacillus subtilis*, *Staphylococcus aureus*, and *Pseudomonas aeruginosa*, as well as the *Penicillium chrysogenum* fungus.

2.2. Apparatus

The PLA film to be investigated was made by applying a single-screw extruder type PlastiCorder PLV 151 (Brabender, Germany). The characteristic features of the extruder were as follows: the screw diameter of 19.5 mm, *L*/*D* ratio of 25, and flat head with the nozzle of an adjustable gap size and of the 170-mm width. The extruder was coupled with a calender consisting of three cylinders 110 mm in diameter, cooled with water. The temperatures of the cylinder heating zones I, II, and III and of the extruder head were 180, 190, 200, and 200 °C, correspondingly. The rotational speed of the extruder screw was 60 min^{-1} and the temperatures of the upper, middle, and lower rollers were 46, 37, and 27 °C, respectively.

To modify the PLA film, the AF2 film activator (IPTS Metalchem, Toruń, Poland) was used. Its diagram is given in Fig. 1 (Brzeziński et al., 2009). The main characteristics of the activator include a generator with the power of 2 kW (Energoelektronika, Bydgoszcz, Poland), discharge frequency of 50 kHz, inter-electrode voltage of 14 kV, single-tip high-voltage electrode (HVE) for discharges in the air (0.25 m long, made of aluminium due to its resistance to the oxidising effect of ozone), roller electrode (RE), also known as the earthed electrode (EE), accuracy of the inter-electrode gap adjustment of 0.1 mm, and film feed velocity of 0–100 m/min. The width of the inter-electrode gap should be as small as possible and should not exceed 3 mm (Żenkiewicz, 2000). Thus, in the present research, the distance between HVE and RE was chosen to be 1.8 mm. Due to the PLA film thickness of 0.1 mm, it was the smallest distance possible to carry out the modification process uniformly.

To determine the count of the cells of bacteria placed onto the PLA film, a Densilameter II (Pliva-Lachema, the Czech Republic) densitometer was used.

The microscopic images demonstrating the effect of the corona discharges on the microorganisms were taken with the use of an ECLIPSE E200 (Nikon, Japan) epifluorescence microscope equipped with a 50-W mercury high-pressure bulb and appropriate (00 and 10) filter sets. The PLA film specimens with the deposited bacterial cells were analysed using the lens of the 1000× magnification.

2.3. Methods

Each culture of a strain was prepared in the medium made up of a nutrient broth. Having the medium inoculated, the cultures were maintained at 37 °C for 24 h. Then, the count of cells was determined by a standard method of evaluation of optical density of the prepared cell suspension with the use of a densitometer based on the McFarland's scale (McFarland, 1907). The determined optical density of the studied suspension was on the order of magnitude of 0.5, which corresponded to 1.5×10^8 cells per 1 ml of the suspension, according to the McFarland scale. There were prepared three specimens of the PLA film to be modified with each E_j value and three control samples of the non-modified PLA film to compare

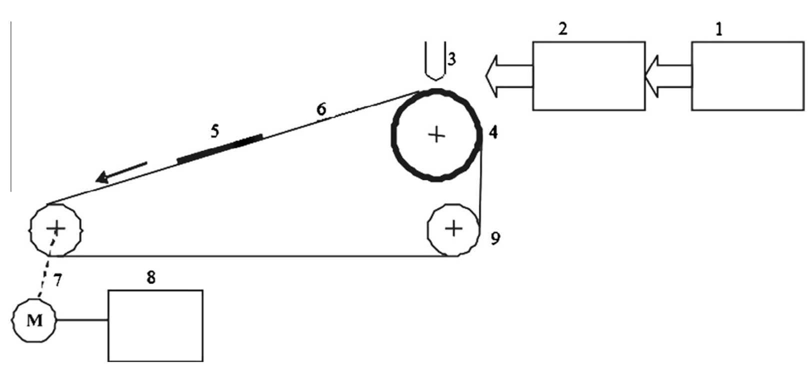

Fig. 1. Scheme for a film activator: voltage generator (1), high voltage transformer (2), high voltage electrode (3), roller/earthed electrode (4), film specimen (5), conveyer belt (6), conveyer drive (7), speed reducer (8), and idler pulley (9).