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Formulating essential oil microemulsions as washing solutions for organic fresh produce production

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ABSTRACT

Applications of plant-derived organic essential oils (EOs) as antimicrobials for post-harvest produce operations are limited by their low water solubility. To dissolve EOs in water, microemulsions were studied using two surfactants permitted for organic production, sucrose octanoate ester (SOE) and soy lecithin that were mixed at various mass ratios before dilution with water to 40% w/w. EOs were then mixed with the surfactant solution by hand shaking. Based on visual transparency, intermediate lecithin:SOE mass ratios favoured the formation of microemulsions, e.g., up to 4.0% clove bud oil at ratios of 2:8 and 3:7, and 4.0% cinnamon bark oil and 3.0% thyme oil at ratios of 2:8 and 1:9, respectively. Microemulsions with intermediate lecithin:SOE mass ratios had a relatively low viscosity and better ability to wet fresh produce surfaces. The microemulsions established in this work may be used as washing solutions to enhance the microbial safety of organic fresh produce.

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1. Introduction

Fresh produce is an important group of food products ([Rico,](#page--1-0) [Martin-Diana, Barat, & Barry-Ryan, 2007\)](#page--1-0). At the same time, outbreaks of foodborne illnesses linked to fresh produce contaminated by pathogens have also increased, most likely due to the increased consumption of prepackaged fresh products ([Doyle & Erickson,](#page--1-0) [2008; Harris et al., 2003\)](#page--1-0). Since fresh produce has many chances of being contaminated by pathogens from farm to fork and many are consumed with minimal processing, they have become commonly associated with outbreaks of foodborne illnesses [\(Sivapalasingam,](#page--1-0) [Friedman, Cohen, & Tauxe, 2004](#page--1-0)).

Washing with sanitizers is often used to enhance safety and prolong shelf-life of fresh produce. To reduce water consumption and wastewater discharge rates, water used in the pre-washing and washing operations for produce is recycled and reused ([Ölmez & Kretzschmar, 2009](#page--1-0)). This recycled water has decreased sanitizer concentrations and thus may have viable pathogens that have detached from fresh produce and transfer to non-contaminated produce [\(Luo, 2007; López-Gálvez, Gil, Truchado, Selma, &](#page--1-0) [Allende, 2010](#page--1-0)). Furthermore, the use of sanitizers on organic produce is strictly regulated. For example, a maximum of 4 ppm

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residual chlorine in waste water discharge is allowed by the National Organic Programme [\(Suslow, 2011](#page--1-0)) which can limit the effectiveness of sanitization. The organic produce industry would benefit from sanitizers which have greater antimicrobial capacity and increased activity over time in post-harvest washing applications.

In recent years, plant-derived essential oils (EOs) have been extensively studied as naturally occurring food preservatives because of their broad antimicrobial activities [\(Davidson, Critzer,](#page--1-0) [& Taylor, 2013\)](#page--1-0). Since EOs can be prepared from organic plants, organic EOs may be prepared as alternative sanitization agents. Generally, an EO concentration of $0.01-1\%$ v/v is applicable as washing solutions for fresh vegetables and fruits [\(Wan, Wilcock,](#page--1-0) [& Coventry, 1998\)](#page--1-0). Due to the limited solubility of EOs in water, strategies are needed to dissolve EOs in water for use as washing solutions.

Emulsions are commonly used to incorporate hydrophobic compounds in aqueous systems in the food industry and could be used to disperse EOs. Nanoemulsions, with droplet diameters smaller than ca. 200 nm, can be stable against gravitational separation and aggregation [\(Uson, Garcia, & Solans, 2004](#page--1-0)). However, the major antimicrobial compounds in EOs have some water solubility, which causes the growth of droplet particles during storage due to Ostwald ripening [\(McClements & Rao, 2011](#page--1-0)). In contrast, microemulsions are composed of a mixture of oil, surfactants, and water, also possibly co-surfactants, that self-assemble into structures that minimise the system free energy and therefore

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are thermodynamically stable [\(Lawrence & Rees, 2000\)](#page--1-0). The transparent and shelf-stable features of microemulsions make them good candidates for preparation of EO-based sanitization wash solutions.

When developing EO-based wash solutions, the accessibility of EO components to microorganisms is important because the latter can be located in protected sites like cut surfaces, stomata, and crevices ([Takeuchi, Matute, Hassan, & Frank, 2000](#page--1-0)). The hydrophobicity of plant surfaces varies significantly and hydrophobic surfaces make it difficult for sanitizing solutions to contact/wet surface structures ([Bhushan & Jung, 2006](#page--1-0)). This can result in significant differences in the effectiveness of sanitizers when tested in growth media versus applied to fresh produce [\(Xiao et al., 2011](#page--1-0)). Contact angle between air, liquid (sanitizer solution) and solid (surface of fresh produce) phases is commonly used as a parameter to measure the wettability of fresh produce ([Juniper, 1959](#page--1-0)). Surfactants can lower the interfacial tensions and thus decrease the contact angle and increase the wettability of sanitizer solutions on hydrophobic produce surfaces ([Choi, Park, Ahn, Lee, & Lee, 2002](#page--1-0)). Microemulsions, therefore, have potential for use as washing solutions because surfactants are used to dissolve EOs and can improve the wetting properties.

The major objective of the present work was to formulate microemulsions of organic EOs applicable to the production of organic produce. Lecithin and sucrose octanoate ester (SOE) are the two surfactants permitted for use on organic fresh produce ([Suslow, 2011\)](#page--1-0). These two surfactants were used to prepare microemulsions of three organic EOs (clove bud, cinnamon bark, and thyme oils). The second objective was to characterise the viscosity of surfactant solutions and their contact angles on organic fresh cherry tomato, carrot, cantaloupe, romaine lettuce and spinach because these two properties are important to the application of microemulsions as washing solutions.

2. Materials and methods

2.1. Materials

Organic certified clove bud oil, cinnamon bark oil and thyme oil were procured from Sigma–Aldrich Corp. (St. Louis, MO). Soybean lecithin was purchased from Fisher Scientific (Pittsburgh, PA). SucraShield™ (containing 40% SOE) was purchased from Natural Forces LLC (Davidson, NC). Organic cherry tomato, carrot, cantaloupe, romaine lettuce and spinach were purchased from a local retailer.

2.2. Preparation of surfactant solutions

Soybean lecithin and SucraShield™ were mixed at different lecithin:SOE mass ratios to form a homogenous mixture. Water was then added to the surfactant mixture to an overall surfactant (SOE and lecithin) mass concentration of 40% w/w. Net content of SOE in SucraShield™ was used in formulations.

2.3. Viscosity measurement

Viscosities of surfactant solutions were studied with an AR 2000 rheometer (TA Instrument, New Castle, DE) using a Searle setup (bob outer diameter = 28 mm and cup inner diameter = 30 mm). A shear rate ramp from 1 to 100 s^{-1} was applied at a constant temperature of 25 °C. Each sample was measured three times and the averages were reported.

2.4. Preparation of emulsions

Each of the three EOs was added to the surfactant solutions to an overall concentration of 0.5–5.0% w/w, followed by hand shaking till the mixture appeared visually homogenous. The samples were incubated at room temperature (21 \degree C) overnight before use.

2.5. Contact angle measurement

Contact angles were measured using a goniometer (model 100-00-230, Ramé-Hart Instrument Co., Mountain Lakes, NJ). Romaine lettuce and spinach leaves were cut into $2 \text{ cm} \times 2 \text{ cm}$ square pieces and fixed on a glass slide using double-sided tape, and both the adaxial and abaxial surfaces were evaluated. Carrot and cantaloupe were peeled to take the outer layer which was similarly taped on a glass slide. A cherry tomato was cut into two pieces along the major axis before being placed on a glass slide. One drop $(1 \mu l)$ of water or surfactant solution was placed on the surface of a fresh produce sample. For the halved cherry tomato, the droplet was placed on the top of the curvature. Photos of solution droplets on the surface of fresh produce were taken using a CCD camera. Contact angles were measured and recorded for 20 droplets on each kind of produce.

2.6. Statistical analysis

All emulsion samples were prepared in duplicate, and all measurements were repeated at least twice. Means and standard deviations of replicates were reported. Analysis of variance was used to determine significant differences (p-value 0.05), followed by mean separation (least significant difference – LSD). All statistical analysis was conducted using Statistical Analysis Software (V9.2, SAS Institute, Cary, NC).

3. Results and discussion

3.1. Viscosity of surfactant solutions

Preliminary experiments were performed by mixing 10%, 20%, 30%, and 40% mass of surfactant mixture in water, with various SOE:lecithin mass ratios from 1:0 to 1:1. Only samples with 40% w/w surfactant were transparent at all studied SOE:lecithin mass ratios and were chosen for further characterisations. The viscosities of these surfactant solutions are shown in Fig. 1. Solutions with

Fig. 1. Viscosities of 40% w/w surfactant mixture solutions at 25 °C. Surfactants were a mixture of lecithin and sucrose octoanate ester (SOE) with mass ratios shown in the legend.

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