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Evaluation of thermal processing variables for reducing acrylamide in canned black ripe olives

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ABSTRACT

Acrylamide, formed in certain plant foods at elevated cooking temperatures, has been identified as a probable carcinogen. A wide variation in acrylamide concentration has been reported for commercial canned black ripe olives, with some levels being quite high. Standards for olive sterilization were developed before acrylamide was discovered to be present in food. The objective of this study was to determine if alternative sterilization conditions during thermal processing could substantially reduce acrylamide levels in black ripe olives, while still maintaining safety and quality. Heat penetration tests for six thermal processes were used to evaluate process F_0 – a sterilization value –which was correlated to acrylamide formation and changes in quality attributes of black ripe olives. Acrylamide concentration followed a positively correlated second order polynomial regression with process F_0. This correlation was further demonstrated by two of the experimental processes with different initial temperatures and different processing times, but with ultimately similar F_0, that produced very similar levels of acrylamide. The amounts of solids leaching from olives in brine increased at higher temperatures of thermal processing, whereas pH variation in olives and brine was most likely related to the differences in lye-wash cycles. Skin color was unaffected, whereas increasing (time/temperature) thermal processing reduced the firmness of whole olives. Optimization of safe thermal processing conditions while lowering process F_0 is a practical and efficient strategy to reduce acrylamide formation and improve the safety of black ripe olives.

1. Introduction

Acrylamide [$(CH_2{=}CH{-}CO{-}NH_2$] is a conjugated reactive molecule that acts as a biological alkylating agent. It has been reported to induce numerous adverse effects in cells, animals, and possibly also humans, such as antifertility, carcinogenicity, neurotoxicity, and teratogenicity (Friedman, 2003). Acrylamide has been identified as a probable carcinogen (International Agency of Research on Cancer – IARC, 1994 – ; National Toxicology Program, 2012). The chemistry of acrylamide formation in food, and it's *in vivo* reactions with essential enzymes, DNA, and neurons that cause the mentioned adverse effects as well as methods to reduce formation in processed food and mitigation of *in vivo* toxicity are described in detail elsewhere (Friedman, 2003, 2015; Friedman and Levin, 2008; Friedman and Mottram, 2005).

Dietary acrylamide in most foods is derived from the heat-induced Maillard-type reaction between the amino group of the amino acid asparagine and the carbonyl group of a reducing sugar such as glucose and fructose. Both precursors and heat must be present for this route of formation, so acrylamide is generally found only in thermally processed plant-derived foods including cereals, coffees, almonds, olives, and potatoes (Friedman, 2003; Stadler et al., 2004; Tareke et al., 2002; Zyzak et al., 2003). Many reported methods to reduce acrylamide levels in foods depend on reducing the availability of the precursors, asparagine and sugars

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(Amrein et al., 2007; Friedman and Levin, 2008). The formation of acrylamide in olives is also heat-dependent, but appears to be by a different mechanism than the Maillard reaction (Casado et al., 2013; Charoenprasert and Mitchell, 2014). There is no correlation between asparagine and sugar concentrations in the raw olive, and subsequent acrylamide formation (Casado and Montaño, 2008). Acrylamide in olives is primarily found in the style known as California Black Olives, which are sterilized at high temperature, rather than pasteurized or preserved as are most other styles (Charoenprasert and Mitchell, 2014).

Acrylamide is formed in foods cooked at elevated and typical baking and frying temperatures. Studies with laboratory-heated foods revealed a temperature dependence of acrylamide formation, which is not detected in unheated or boiled foods (Tareke et al., 2002). Various levels of acrylamide have been found in black ripe olives. In a survey of black ripe olives in US market, a wide range of acrylamide (375–1925 μg/kg) was found (U.S. Food and Drug Administration – U.S. Department of Health and Human Services – Center for Food Safety and Nutrition, 2006 – –). Casado and Montaño (2008) screened 11 commercial black ripe olive samples (pitted, whole, and different cultivars) from Spain and found levels of acrylamide also ranged broadly from 176 to 1578 μg/kg of olive pulp. Similarly, we found 288–1192 μg/kg acrylamide in seven black ripe olives samples (unpublished results). Charoenprasert and Mitchell (2014) also reported relatively high concentrations of acrylamide in black ripe olives (226–1925 μg/kg).

It has been shown that acrylamide in black ripe olives is primarily formed during the sterilization step in the canning and bottling process, and that sterilization time and temperature can significantly influence the formation of acrylamide (Amrein et al., 2007; Casado and Montaño, 2008; Casado et al., 2010). Black ripe olives are processed through several lye immersion and water rinsing steps (Charoenprasert and Mitchell, 2014; Romero et al., 1995) and then canned or bottled and sterilized as a low-acid canned food (LACF) with a flesh pH around neutrality (Casado et al., 2007; Romero et al., 1995). LACF heat treatment standards require assurance of at least 12 log reduction of *Clostridium botulinum* spores at a 12D processing time at a constant lethal temperature, where D refers to time at a reference constant lethal temperature to achieve a one log cycle reduction (or 90% reduction) in the number of spores in the LACF (Stumbo, 1973).

California-style black ripe olives, as a LACF, usually require sterilization temperatures above 110 °C (Charoenprasert and Mitchell, 2014). In contrast Spanish- and Greek-style table olives are considered acidified foods (AF) and require less rigorous methods: either pasteurization temperatures below 65 °C, additives for preservation, or a combination of the two, and as a consequence, acrylamide content is negligible in these two types of olives (Charoenprasert and Mitchell, 2014). Although acrylamide formation has been reported as temperature dependent, it correlates better with the overall thermal load, which also considers processing time. Lowering the sterilization temperature and shortening the sterilization time could reduce the formation of acrylamide, while continuing to meet the necessary standards to assure a low hazard and quality product. It is hypothesized that the wide variation of acrylamide in commercial canned black ripe olives is a consequence of different thermal processes used by industry due to variable container sizes and retort types. Although it has been reported that acrylamide in canned black ripe olives is caused by high temperature processing (Casado and Montaño, 2008; Casado et al., 2010), there is a lack of systematic studies to define the effect of thermal processing during commercial sterilization of olives on the levels of acrylamide in the final product. For LACF, it is important to assure commercial sterility and then determine if it is still possible to reduce acrylamide concentrations below levels found while using commercial canning practices.

The objective of this study was to determine if different safe sterilization conditions during thermal processing could reduce substantially the acrylamide levels in canned black ripe olives. We adjusted the sterilization time and temperature for six different processing conditions. From heat penetration data, process lethality was calculated and correlated to acrylamide formation and to changes in physical quality attributes of black ripe olives.

2. Materials and methods

2.1. Reagents

For the olive preparation, sodium hydroxide solution (50%) was obtained from Loeffler Chemical Corp. (Atlanta, GA, USA). Sodium chloride (canning and pickling salt, food grade) was purchased from Morton Salt Inc. (Chicago, IL, USA). Ferrous gluconate (food grade) was purchased from VWR (Radnor, PA, USA).

For the acrylamide analysis, acrylamide (>99.9%) and methanol (HPLC grade, 99.9%) were purchased from Fisher Scientific (Pittsburgh, PA, USA). Acrylamide-d_3 standard solution (500 mg/L in acetonitrile) was purchased from Sigma-Aldrich (St. Louis, MO, USA). Reversed-phase solid phase extraction cartridge columns (Sep-Pak™ C18, 1 g and 6 cc) were obtained from Waters Corp. (Milford, MA, USA).

2.2. California-style black ripe olive preparation

Pilot-plant olive preparation was modified from the methods developed by Charoenprasert and Mitchell (2014) to mimic industrial California-style black ripe olive processing.

2.2.1. Harvest and washing

Olives were harvested in October 2014 and stored in 2% acetic acid solution by the processor. During May 2015 13 kg batches of olives were immersed in 1% sodium hydroxide solution (19 L) for 5 h for lye treatment, and then rinsed eight times before immersion in fresh water for 19 h to remove the residual sodium hydroxide. During the immersion steps, air was constantly bubbled into the water to oxidize the olives. The lye-wash cycle was performed a second time with the following changes: during the second cycle, the olives were soaked in lye for 2 h and immersed in fresh water for 20 h. The pH was measured after the final lye treatment to ensure the lye penetrated to the pit and the pH was between 8 and 9.5. Then the olives were treated with 0.15% ferrous gluconate solution (19 L) for approximately 3 h to fix the color. During the ferrous gluconate treatment, carbon dioxide was bubbled into the solution to neutralize olives. When the pH reached 7, the olives were rinsed to remove residual ferrous gluconate.

2.2.2. Blanching, canning and heat penetration testing

Cruess (1921) indicated that a tendency of the can to “buckle” was greatly reduced when olives in cans were thoroughly heated in an exhaust box to 90.6 °C before sealing. To achieve this, whole olives should be blanched under saturated steam conditions for 2–4 min. This additional processing step eliminates tissue gases and bulging of cans without increasing acrylamide levels. Whole olives were blanched for a residence time of 4 min, in single layers on stainless steel trays moving on a continuous belt on a custom built steam blancher at the University of California, Davis FST food processing pilot plant. Whole olives reached a maximum temperature of 96.7 ± 1.8 °C from an initial temperature of 25.0 ± 1.1 °C (n = 6).

Can filling was done by hand. Fill weights (790 g) for 401 × 411 (10.32 cm diameter, and 10.91 cm height) cans were 322.5 g (41%)