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Effect of solvent addition sequence on lycopene extraction efficiency from membrane neutralized caustic peeled tomato waste

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ABSTRACT

Lycopene is a high value nutraceutical and its isolation from waste streams is often desirable to maximize profits. This research investigated solvent addition order and composition on lycopene extraction efficiency from a commercial tomato waste stream (pH 12.5, solids ~5%) that was neutralized using membrane filtration. Constant volume dilution (CVD) was used to desalinate the caustic salt to neutralize the waste. Acetone, ethanol and hexane were used as direct or blended additions. Extraction efficiency was defined as the amount of lycopene extracted divided by the total lycopene in the sample. The CVD operation reduced the active alkali of the waste from 0.66 to <0.01 M and the moisture content of the pulp increased from 93% to 97% (wet basis), showing the removal of caustic salts from the waste. Extraction efficiency varied from 32.5% to 94.5%. This study demonstrates a lab scale feasibility to extract lycopene efficiently from tomato processing byproducts.

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1. Introduction

Lycopene is a lipid soluble phytochemical that is responsible for the red color in various fruits and vegetables, including tomatoes. Consumption of a diet rich in tomatoes has been correlated with decreased risk for a number of chronic diseases, including heart disease and cancer, specifically prostate cancer (Story, Kopec, Schwartz, & Harris, 2010; Zu et al., 2014), and this decrease risk is often attributed to lycopene Processed tomato products are a worldwide commodity that yielded over 210 million metric tons in 2012 (FAOSTAT, 2012). The use of a strong base (KOH or NaOH) as chemical peeling agents is a common practice in many commercial operations. The effluent created from a chemical peeling operation consists of skins, seeds and tomato flesh with a very alkaline pH (11.5–12.5). Research has shown that pretreatment of tomato with strong base prior to lycopene extraction increases extraction efficiency due to its destructive effect on cellular structure (Cuccolini, Aldini, Visai, Daglia, & Ferrari, 2013), making an effluent from a chemical peeling tomato operation ideal for lycopene extraction. The same research found that subsequent treatment of the tomato with enzymes allowed for lycopene extraction without use of organic solvents. Although this process is devoid of organic solvents, pretreatment with 4% (w/w) NaOH and then acidification (with HCl) is chemically intensive, and alternative methods to neutralization have yet to be investigated.

Solvent extraction of lycopene is a common method for extraction both on a commercial (Ben-Yehuda, Garti, Hartal, Raveh, & Zelkha, 2004) and laboratory scale (Kopec et al., 2010). This can be attributed to several factors. First, certain solvents have the ability to disrupt and dehydrate plant cellular structure, making the lycopene more available for extraction. Once these solvents have penetrated the cellular structure, hexane is typically used to extract the lycopene due to its relatively high solubility, making it the primary component in most extraction blends. It has been demonstrated that a blend of hexane, ethanol and acetone in a 30:35:35 (vol%) ratio can optimize lycopene extraction in a single step extraction method, which has been patented (Lavecchia & Zuorro, 2007). However, multi-step extraction procedures are often used to extract lycopene from tomato. In the first step,

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acetone and/or ethanol are used to disrupt cellular structure. In the second step, hexane is added to allow partitioning of the lycopene in to the non-polar fraction. Water is added as a third step to create two distinct phases: the upper phase consists of lycopene (and other carotenoids) dissolved in hexane, and the lower/aqueous phase contains water, acetone and/or ethanol and water soluble compounds from the plant material. In this procedure, effects of addition order on extraction efficiency have yet to be investigated.

Advanced processing technology for the extraction of lycopene from tomatoes has emerged in the past several years. Supercritical CO_2 has been used and shows promise as a commercial extraction procedure (Egydio, Moraes, & Rosa, 2010; MacHmudah et al., 2012). The strongest advantage to supercritical CO_2 is its ability to extract lycopene without the use of organic solvents, although research using supercritical CO_2 with other solvents also demonstrates promise (Saldana, Temelli, Guigard, Tomberli, & Gray, 2010). Detrimentally, it takes a large amount of energy to operate commercial scale supercritical fluid extractors and extraction yields of 80% or more require a large amount of CO_2 (>100 g CO_2/g sample). Researchers have recently begun to investigate if enzymatic treatment in tandem with supercritical CO_2 can reduce the cost of operation on a commercial level (Lenucci et al., 2015). Other advanced processing technologies include the Naviglio Extractor® (Naviglio, 2003) and high hydrostatic pressure extractions (Jun, 2006). Both of these technologies are modifications of solvent based extractions and evaluations to optimize extraction parameters are still being assessed. The goal of this study was to investigate the effects on solvent type and order on lycopene extraction efficiency from a commercial tomato waste stream.

2. Materials and methods

2.1. Commercial waste

Commercial caustic tomato slurry was obtained from Hirzel Canning Co. (Ottawa, OH). This slurry originated from Roma tomatoes processed first through two caustic (NaOH) baths followed by a mechanical peeler/scrubber. The material extracted from the peeler/scrubber was further processed using a horizontal decanter creating two streams. The solids fraction included seeds and skin having a wet basis moisture content (MC_{wb}) of 77.9 ± 0.03% and the liquid fraction contains tomato pulp and caustic solution with a MC_{wb} of 93.3 ± 0.01%. The liquid fraction from the decanting operation was the caustic tomato slurry used in these experiments.

2.2. Chemical free neutralization

2.2.1. Pilot scale membrane filter design

Chemical free neutralization was completed using a constant volume dilution (CVD) method via membrane filtration. A pilot scale membrane filtration unit was used (Model BRO/BUF Membrane Specialists, Hamilton OH). The unit consists of a Hydra-cell high-pressure pump (Model D15EASTHFEHF, Wanner Engineering, Minneapolis MN) connected to a variable frequency drive controller. A touch screen human machine-interface communicates with a downstream pressure controller to modulate target membrane inlet pressure. A 1.2 m tubular membrane housing (Model B1, PCI Membranes, Hampshire UK) was used with a pH tolerant ultra-filter with a 200 kDa molecular weight cutoff (Model FPN-200, PCI Membranes). A total of eighteen 1.2 m long membranes with a diameter of 1.0 mm were fitted into the housing for a total of 0.9 m^2 surface area. The coarse filter was selected to retain the colloidal components of the tomato pulp. The material retained by the filter (retentate) was returned to the feed tank while the material that was passable across the membrane (permeate) was collected and evaluated for moisture content, pH, active alkalinity and electrical conductivity. As permeate was removed, reverse osmosis water (pH 7.2, electrical conductivity 0.155 mS/cm) was added to the feed tank (with attached mixer) at the same volumetric flow rate (i.e., constant volume dilution). Fig. 1 shows the unit operation for the CVD process.

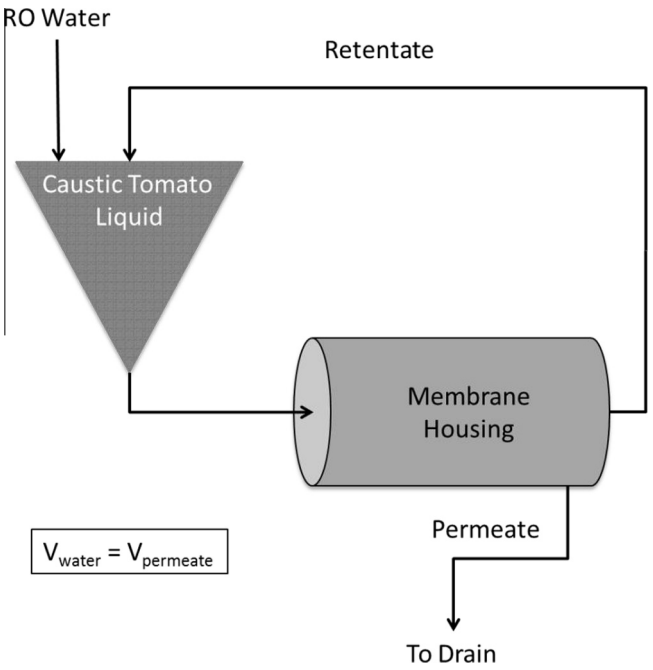

Fig. 1. Unit operation diagram for the constant volume dilution operation used to achieve a chemical free neutralization of the caustic tomato slurry.

2.2.2. Moisture content, active alkali, electrical conductivity and pH

Moisture content of the permeate and retentate streams throughout the CVD process were determined using an oven drying method. Briefly, approximately 3 g of sample was weighed on an aluminum pan and placed in a drying oven at 110 °C and heated for 18 h (Liu, Liu, Chen, Liu, & Di, 2010). The loss in mass was attributed to the loss of water from the sample and moisture content was calculated.

The active alkali of the permeate was measured throughout the CVD process. Active alkali is a direct titration method for determining the quantity of ionic hydroxyl species within a strong caustic solution (Mercadé-Prieto, Paterson, Chen, & Wilson, 2008; Scandinavian Pulp, 1985). Specifically, thirty milliliters of permeate sample was pipetted into a 50 mL flask. One drop of phenolphthalein was added to the flask, causing the sample to turn pink. The solution was titrated with 0.1 M HCl until the pink color disappeared. The consumed volume of titrant (V_{HCl}) was read and recorded and the molarity of the permeate was calculated as being equal to the molar mass of HCl used to titrate the sample.

The electrical conductivity and pH of the permeate were measured using a Hanna Instrument Edge® Multiparameter meter (Hanna Instruments, Woonsocket RI; model HI2020) with an electrical conductivity electrode (model HI763100) and a pH electrode (model HI11310).

2.3. Solvent extraction of lycopene from tomato waste

2.3.1. Experimental design

Solvent extraction was performed in three sequential steps: step 1 aimed to disrupt the cellular structure to aid extraction, step 2 aimed to extract lycopene from the matrix into a hexane