MEMBRANE CARBON DIOXIDE STERILIZATION OF LIQUID FOODS: SCALE UP OF A COMMERCIAL CONTINUOUS PROCESS

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INTRODUCTION

Producing safe, flavorful and nutritious liquid foods and beverages without using heat requires a revolution in food processing technology. Continuous sterilizing of liquid foods using only dissolved carbon dioxide and no destructive heat can provide the needed technology.

FRESH TASTE. The temperature used in the process is low, under 45^{o}C, so the taste of a fruit juice, for example, remains that of fresh squeezed.

MICROBE ELIMINATION. Dissolved carbon dioxide kills both pathogenic and spoilage microorganisms, obtaining the level of "pasteurization" or complete sterility.

NUTRIENT PRESERVATION. The low processing temperature assures this.

ENZYME INACTIVATION. The enzymes responsible for food browning or juice pulp precipitation are inactivated. The food stays fresh looking.

PRODUCT PRESERVATION. Carbon dioxide displaces the dissolved oxygen, giving foods a longer shelf life, both for taste and appearance.

NO PROCESSING ARTIFACTS. The process handles pulpy and cloudy juices without changing the texture or mouth feel. No excess carbon dioxide remains unless it is purposely left in to make a carbonated beverage.

First observations that carbon dioxide inhibits microbial growth date back 100 years. Jones [5] and Daniels [2] give comprehensive reviews of the publications on inhibition. More recent investigations of exposing foods to carbon dioxide show that microbes are killed at room temperature when carbon dioxide partial pressure is superatmospheric. Stahl [13] and Magnaradze [8] showed total kill of several microorganisms, including E. *coli*, at carbon dioxide pressure as low as 4 atm. The necessity of the presence of free water for the germicidal effect of carbon dioxide was shown by Taniguchi [14]. Since then, in over thirty publications, the necessity of water is clearly recognized as essential to the microbiocidal action of carbon dioxide. The work of Kumagai [7] is particularly compelling.

In only two publications was the concentration of dissolved carbon dioxide in the aqueous test fluid noted. Shimoda [10], killing bacteria and yeast, measured the concentration of dissolved carbon dioxide to be 90% of saturation at their highest sparging rate. Ishikawa [4], inactivating enzymes, measured a maximum concentration of 80% of saturation.

Many enzymes, but not all, are inactivated irreversibly by pressurized carbon dioxide when the environment surrounding the enzyme is above a critical water concentration, i.e. when there is free water. Weder [15] summarizes the effects of the carbon dioxide/water combination on proteins and enzymes. Shank [9], Friedrich [3] and Christianson [1] patented

processes using pressurized carbon dioxide/water to inactivate proteases in fresh meat, lipoxygenase in moist soy flakes and peroxidase in moist corn germ, respectively.
To make a commercially feasible continuous process we employ a membrane contactor to carbonate the flowing test liquids, i.e. buffered water or orange juice. A membrane contactor is a much more efficient carbonator than conventional dispersing devices. In a membrane contactor carbon dioxide is not mixed with the flowing liquid food but instead diffuses into it across the large surface area of a membrane only as needed for saturation. It mimics the natural, highly evolved efficiency of the lungs of air-breathing creatures. Karoor [6], working with carbon dioxide/water at 2 bars, showed that the carbonating efficiency of a membrane contactor was at least five times greater than a conventional packed tower.

MATERIALS AND METHODS: DISSOLVED CO_2 DETERMINATION

Water or orange juice exiting the apparatus through the product valve is collected in a filter flask with a hose from the side arm connected to an ASTM dry test meter. The liters of released carbon dioxide are converted to grams, and the vacuum degassed water is weighed. Results in g CO_2/100 g water are compared to the literature solubility of carbon dioxide in water of Teng [15] to determine percent saturation.

CONCLUSION

The objective of this work is to provide guidelines for scaling up a commercial continuous process. Sims [11] proposed such a process with partial recycle of carbon dioxide. It is most important to recognize that the kinetics of carbonation and of kill are separate and need to be optimized individually. The process sequence is shown in Figure 1.

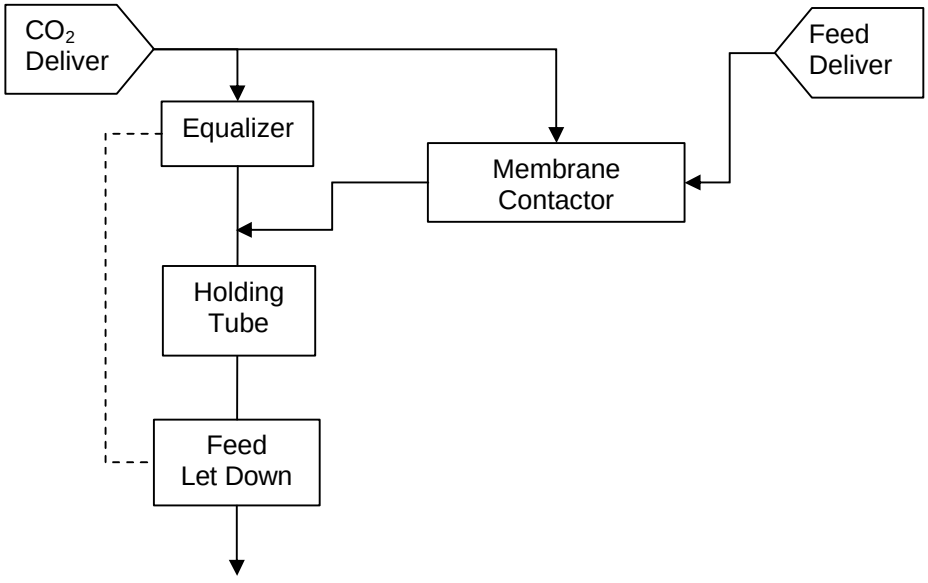


Figure 1 : Membrane Carbon Dioxide Continuous Sterilization of Liquid Foods

It was shown by Sims [12] that the highest microbial killing rate is achieved when the concentration of dissolved carbon dioxide is at saturation at a particular operating pressure and temperature in the holding tube.

The solubility of carbon dioxide in water, Figure 2, shows why an operating pressure of about 75 bar is near optimum. Solubility increase rapidly with increasing pressure up to 50 – 75 bar, but an increase of pressure beyond 75 bar gives little additional increase in solubility or of microbial killing rate.

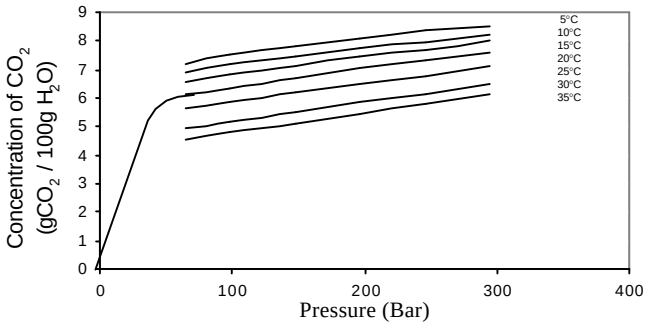


Figure 2 : Dissolved CO_2 Concentration as a function of pressure and temperature

The minimum operating temperature in the holding tube is set by the degree of sterility required and the type of microorganisms. Most pathogenic and spoilage microorganisms in their vegetative state are killed at 40°C, and their spores at 45°C [12].

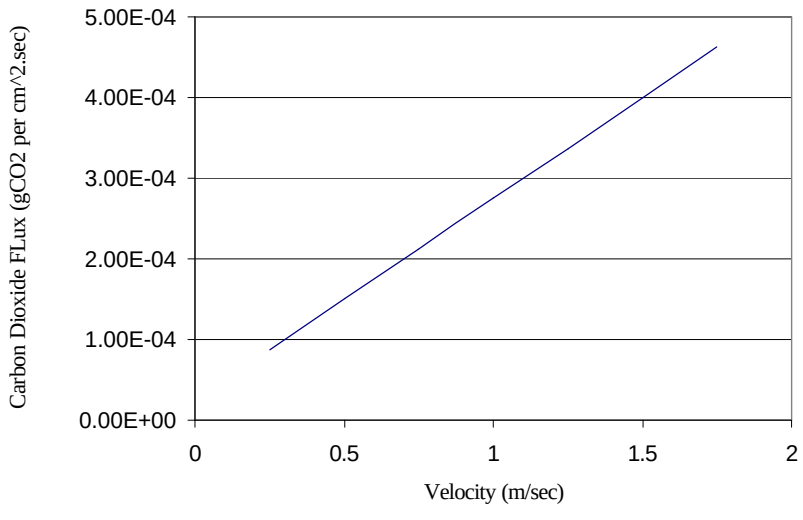
Optimizing the holding tube is simply a matter of choosing tubing size so that a residence time of about one minute and a plug flow velocity profile is achieved, i.e. a Reynolds Number of greater than 3000.

Because the cost of a membrane contactor is based on its active surface area ($/m^2), the optimized membrane carbonator achieves the following criterion:

$$\frac{flowrate}{area} \Rightarrow \text{maximum}$$

The optimum membrane contactor achieves the highest flux of carbon dioxide. The flux in a hollow fiber membrane increases with feed liquid velocity as shown in Figure 3.

Figure 3: Carbon Dioxide Flux Vs. Water Velocity in Lumen of 1.8 mm ID Microporous Hollow Fiber (Membrana/PPS6/2) at 150 bar, 20-40 Deg. C



The process should be operated at the fastest flow rate and velocity as possible while still obtaining near saturation of dissolved carbon dioxide. However, high velocity creates high pressure drop in the hollow fibers which requires higher pumping pressure relative to the system pressure. The higher pumping pressure translates to a high transmembrane pressure differential. The maximum velocity which can be used is set by the specifications of the membrane manufacturer for the maximum allowable transmembrane pressure differential. These differentials range from 0.9 to 3.0 bar, depending on wall thickness, pore size and operating temperature.

The most important simplification of the optimization and scale up procedure is: a single fiber can be used. The performance of one fiber in a bundle is identical to that of any other fiber in the bundle, provided that the distribution of liquid in each lumen is uniform. In carbonation, with liquid carbon dioxide in the pores of the hydrophobic membrane and with feed flowing in the lumen, the distribution and concentration of carbon dioxide are completely uniform over the entire length of the fiber as long as carbon dioxide supply pressure is maintained constant. Data obtained in one fiber characterize the entire bundle simply through multiplication by the number of parallel fibers in the bundle.

When pumping orange juice inoculated with 10^8 CFU/cc Lactobacillus *plantarum*, the optimum 1.8 mm ID fiber is 10 m in length and operates at 1.0 m/sec velocity. Residence time in the fiber is 10 sec. Contact with carbon dioxide at 75 bar provides saturated juice and a total kill of L. *plantarum* after a one minute residence in the holding tube operated at 40^{o}C.

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