BULK TANK TECHNOLOGY:
Acidification of Commercially Fermented Cucumbers in Bulk Tanks to Increase Microbial Stability

H. P. Fleming,* E. G. Humphries, R. L. Thompson, and R. F. McFeeters

U. S. Department of Agriculture, Agricultural Research Service,
and North Carolina Agricultural Research Service,
NC State University, Raleigh, NC 27695-7624, USA

ABSTRACT

Ten commercial fiberglass tanks (8,000 gal) of size no. 1 fermented cucumbers were acidified with 3.21N HCl to lower the brine pH to 3.5 from the pH that resulted at the end of fermentation (3.65-3.70). The volume of hydrochloric acid required to do this varied from 8.9-14.8 gal per tank. The rate of attainment of pH equilibrium after acid addition varied by tank location, but was complete within about 4 hr. Samples of brine-stock and cover brine from each of the 10 tanks were adjusted to pH 3.5, 4.0, and unadjusted and were stored in glass jars under laboratory conditions. Over a 14-month storage period at room temperature, the brines adjusted to pH 3.5 were more stable in chemical composition than the unadjusted, and much more so than the pH 4.0-adjusted samples, based on chemical changes. Chemical changes that indicated microbial instability were characterized by a rise in pH and CO_2 and acetic acid concentrations, and a reduction in lactic acid concentration. The data indicated that lowering the pH of the brine to 3.5 after fermentation from higher levels can help to increase microbial stability of brined cucumbers at relatively low salt concentrations without serious adverse effects on cucumber firmness.

INTRODUCTION

Commercially fermented cucumbers held in bulk tanks are subject to various types of quality loss, including bloater damage, softening, off-flavor development, and others. These problems can be greater with cucumbers which are held at low brine strengths, and is why high salt concentrations (8-16%) have been used by some commercial firms for many years. Regulatory pressures to limit food processing and other wastes have increased over the past 30 yr. Of particular concern to the pickle industry was the issuance of a guideline limit of 230 ppm chloride in freshwater bodies by the U.S. Environmental Protection Agency (EPA, 1987). Brine recycling and reduction of salt concentrations for cucumber fermentation and storage are being increasingly used by many pickle companies to meet regulatory pressures and to avoid surcharge expenses. The frequency of spoilage due to microbial instability may have increased in recent years due to efforts to meet regulatory guidelines.

Microbial instability of fermented cucumbers has been associated with a decrease in lactic acid and an increase in pH, acetic acid, carbon dioxide, and, in extreme cases, the production of propionic and butyric acids (Fleming et al., 1989; 1996; 2002a; 2002b). Carbon dioxide production during storage can cause serious bloater damage since purging to remove it is normally done only during the fermentation period (first 10 days after brining). In extreme cases where propionic and butyric acids appear, off-flavor development can cause the entire tank to require disposal. If the pH rises above 4.6, food safety of the pickles becomes an issue because of potential growth and toxin production by *Clostridium botulinum*. It has been reported that an optimum pH of 3.5 will help ensure microbial and textural stability of fermented cucumbers at relatively low (4.4%) concentration of salt (Fleming et al., 1996). Considerably lower concentrations of hydrochloric acid are required to lower the pH of fermented cucumbers, compared to either acetic or lactic acid (Fleming et al., 1996).

In this paper we demonstrate how the addition of hydrochloric acid (HCl, i.e., muriatic acid) can be used to acidify commercially fermented cucumbers to help increase microbial stability at relatively low concentrations of salt (4.6-5.6%).

MATERIALS AND METHODS

Hydrochloric Acid

Hydrochloric acid (also called muriatic acid) has many commercial applications, including some in the food industry, but, being a hazardous chemical, must be handled with due caution. Hydrochloric acid is considered to be a multiple purpose GRAS (Generally Regarded As Safe) food substance within the meaning of section 409 of the Federal Food, Drug, and Cosmetic Act under 21 CFR, paragraph 182.1057 (Vulcan Chemicals, 1996). Thus, the chemical does not pose a health concern when present in appropriate levels in food, but handling of it in concentrated form can be hazardous. It should be handled so as to avoid inhalation, skin contact, or ingestion by use of safety devices such as protective gloves and masks. Handling instructions supplied by the manufacturer or seller should be carefully followed.

Acidification of a Commercial Tank of Fermented Cucumbers—Concept

Conventional "side-arm" purging units, as employed by the industry, operate as gas-lift pumps and have been described in detail (Costilow et al., 1977). Generally, brine is pulled from the bottom of a tank into a 4-6 inch diameter PVC eduction pipe located near the tank wall, raised to the tank top by the action of the gas (air or nitrogen), and discharged through a short horizontal section at the tank top as illustrated in Figure 1. Thus, a pattern of liquid (brine)

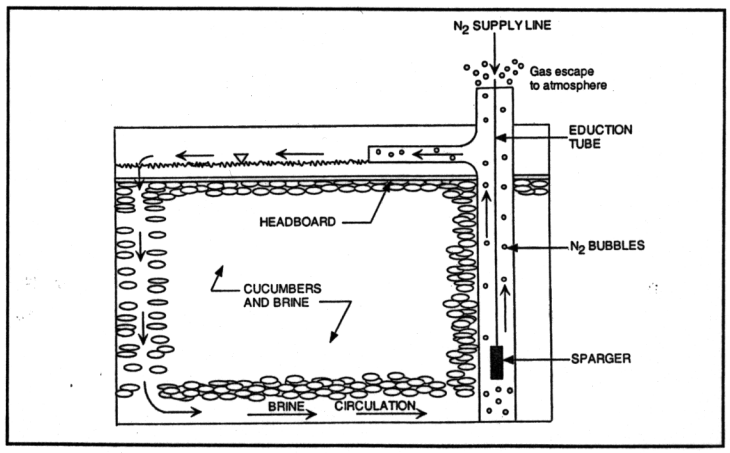

Figure 1. Model cucumber brining tank illustrating the brine circulation pattern when being purged.

circulation is established in the tank that may resemble that illustrated in Figure 1. The flow pattern at the tank top can be visually discerned, as represented in Figure 2A; however, the flow pattern at successive depth intervals is not visible and has not been measured. A liquid zone of 6-20 inches in depth is established at the tank bottom as a result of cucumber buoyancy in the brine. Liquid flow patterns in this zone of the intake of the eduction pipe are most likely as shown in Figure 2B. A transition shift in flow patterns between the top and bottom zones must exist. This paper deals with measurement of pH change at various tank locations (as a result of acid addition via the purging system) and the time required for uniform mixing throughout a tank.

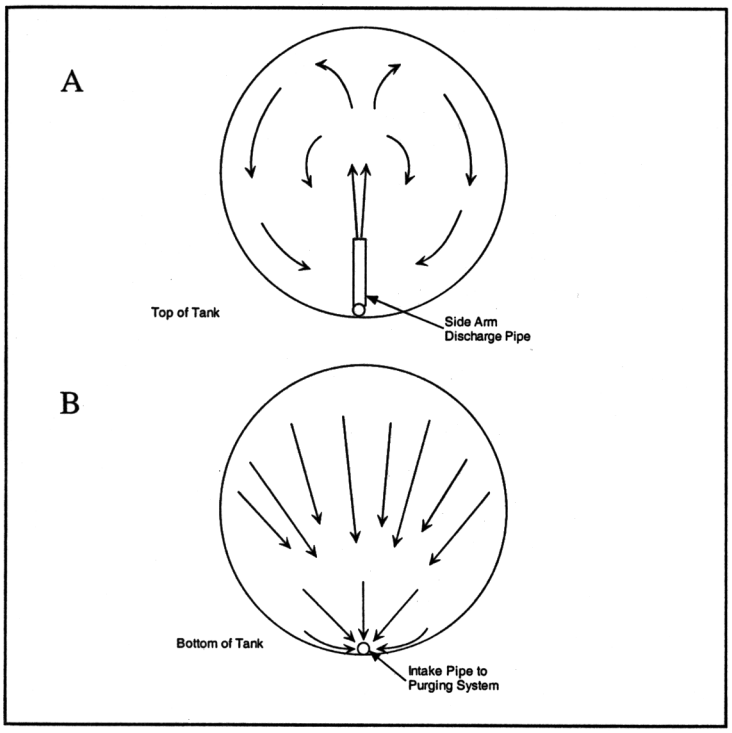

Figure 2. Theoretical brine circulation pattern at the top and bottom of the nitrogen-purged tank of brined cucumbers.

Procedure

A local processor's tank (~8,000 gal) containing no. 1 size (up to 1-1/16 inches diameter) cucumbers and brine was employed to measure mixing response and uniformity. The cucumbers were fermented to completion (no residual sugar) and 20° salometer 1 month prior to acidification on 7/28/1992. The tank purging system was operated at 35 SCFH for 24 hr prior to acidification to assure uniform conditions throughout the tank. The volume flow rate of brine circulated by the purging system was determined by placing a plastic bag over the purging discharge pipe and catching the brine flow for timed intervals. Replicate measurements indicated 46.2 and 45.5 gal/min. At a nominal pack-out of 50% brine and 50% cucumbers, the 4,125 gal of liquid in the 8,351 gal (actual volume of tank occupied by brine and cucumbers) tank would require only 90 min to turn over.

Twelve sampling positions were established throughout the tank at locations indicated in Figures 3-5. Each sample position was established by installation of an appropriate length of 3/8-inch stainless steel tubing with eight 1/8-inch diameter holes within its bottom 6 inches. The bottom end of each tube was plugged with a

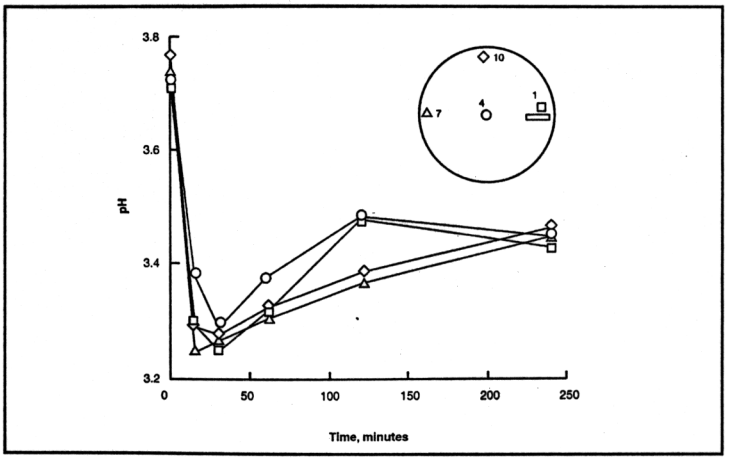

Figure 3. Acidification rate of the brine in the top of the brined cucumbers. The circular diagram represents a tank viewed from the top. The numbers within the diagram represent the locations from which brine samples were taken. The side-arm discharge pipe is illustrated under the □ sampling location.

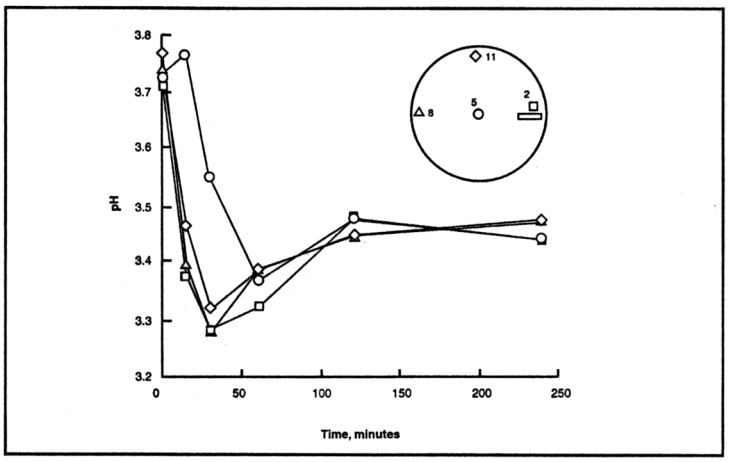

Figure 4. Acidification rate of the brine in the middle of the brined cucumbers. See Figure 3 caption for explanation of the circular diagram.

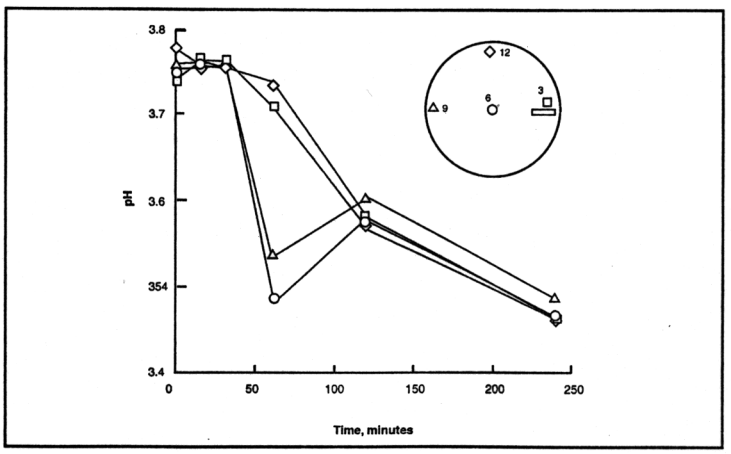

Figure 5. Acidification rate of the brine in the bottom of the brined cucumbers. See Figure 3 caption for explanation of the circular diagram.

bullet-shaped PVC dowel, and each tube was inserted through a hole drilled in the headboards. A flexible Tygon tube was attached to the top end of each sample tube so that all samples could be collected from a common location. The top layer tubes (position numbers 1, 4, 7, and 10) extended about 16 inches below the headboards, which were covered with 8 inches of brine. The middle layer tubes (position numbers 2, 5, 8, and 11) were 72 inches long and were inserted to the approximate mid-depth level of the cucumber mass. The bottom layer (position numbers 3, 6, 9, and 12) tubes extended to a depth about 6 inches from the tank bottom and should have been in a liquid-only zone. The tubes located on the outer perimeter were approximately 6 inches from the tank wall.

Hydrochloric acid diluted to 3.21N was placed in a 55-gal polyethylene drum positioned about 3 ft above the brine surface. The acid was gravity-fed into the elbow of the purging system by 1/4-inch diameter Tygon tubing over an 80-min period. The brine discharge of the purging system deposited and mixed the acid on the top level of the tank and, subsequently, circulated it throughout the tank. Brine samples for pH measurement from the 12 locations were taken after 0, 15, 30, 60, 240, and 360 min; additional samples were taken after 24 and 48 hr.

Acidification of 10 Commercial Tanks

Ten tanks of size no. 1 cucumbers were selected from the brine yard of the pickle company, including the one cited above. These tanks had undergone a normal primary fermentation and had no fermentable sugars left in the brine. The pH of the tanks ranged between 3.6-3.8, with the salt varying from 4.8-5.5% w/v. The lactic acid concentration ranged from 115-170 mM, with acetic acid varying from 15-25 mM. The unusually high lactic concentrations in some of the tanks were attributed to recycled brine being used, as indicated by the company. A normal lactic acid fermentation using fresh brine will generally not produce over 140 mM of lactic acid. The highest level of propionic acid was 7.4 mM, with no butyric acid being detected in any of the tanks. None of the tanks had been brined for more than 4 months, since all had been filled from the spring planting.

Aliquots of the tanks at the same ratio of cucumbers to brine (60:40% w/v) were slurried with a Tekmar Tissuemizer (model SDT 182 Super Dispax, Tekmar, Cincinnati, OH) and titrated with 6N HCl to a pH of 3.5. From the titration data, the quantity of commercial HCl necessary to adjust each of the 10 tanks to a pH of 3.5 was determined, as is summarized in Table 1. Equilibration of the pH was assumed to be complete within 48 hr after the addition of the HCl, based on circulation data collected at 12 different locations within the model tank referenced above. After the pH adjustment, there were no noticeable deleterious effects upon the brine-stock; odor, color, and texture were unaffected by the pH adjustment.

Laboratory Storage Studies

Two 1-gal jars of brine and salt-stock were brought back to the laboratory for controlled storage study. Six 46-oz jars of brine-stock were packed from each tank at a 60:40 (cucumbers:brine) ratio, with two of the jars having no pH adjustment, two adjusted to pH 3.5, and two set at pH 4.0. Estimated amounts of acid or base needed to adjust the brine-stock to the desired pH were calculated by blending 300 g of cucumbers with 200 g of brine and titrating the slurry with either 5.49N HCl or 6N NaOH to the desired pH. The calculated amounts of acid or base were added to the 46-oz jars of brine-stock, leaving a headspace of 5 mm, and the jars were then capped and inverted for storage. The caps were fitted with a rubber septum for sampling of the brine by syringe. After a 2-week equilibration period, the jars were scheduled to be sampled once every 3 months for up to 12 months. The final sample actually occurred after 14, rather than 12 months. Conditions of the sample jars, such as cap pressure, were noted at each sampling period.

Chemical Analyses

Brine samples were analyzed by HPLC with a Dionex system, using a conductivity cell and PAD detector in series, according to the method developed by McFeeters (1993). Lactic, acetic, propionic, and butyric acids were analyzed with the conductivity cell, and sugars and alcohols were analyzed with the PAD cell. Salt and titratable acidity were determined as described by Fleming et al. (2001). All pH and titration data were determined with a model 825 MP Fisher Accumet pH meter.

Brine-Stock Quality Evaluation

Cucumber brine-stock firmness was measured with a USDA Fruit Pressure Tester as described by Bell et al. (1955). Firmness was determined initially on 10 whole cucumbers from each of the 10 tanks from which samples of brine-stock were taken. The final firmness on lots of these cucumbers stored at the three pH values indicated was determined after storage for 14 months.

Table 1. Acidification of commercially brined cucumbers in bulk tanks.[1]

Tank no.	Brine pH			HCL added (gal, 3.21N)[3]	Salt (%)	Lactic acid (mM)
	Before HCl	24 hr after HCl	6 days after HCl			
915	3.75	3.52	3.52	46.8	5.0	114
992	3.65	3.50	3.53	28.1	4.7	143
1513	3.67	3.46	3.47	37.6	5.0	179
1517	3.66	3.48	3.47	34.1	5.6	159
1518	3.67	3.51	3.50	37.6	5.2	173
1521[2]	3.67	3.57	3.54	34.5	5.3	151
1522	3.65	3.51	3.49	30.0	5.5	164
1526	3.65	3.48	3.46	33.2	4.7	152
1527	3.70	3.50	3.48	44.0	4.6	156
1538	3.68	3.50	3.50	41.1	4.8	165

[1]Size no. 1 cucumbers were brined in 8,000-gal tanks.
[2]Tank used to demonstrate rate and pattern of HCl distribution.
[3]Diluted from 10.1N HCl to reduce hazard in handling.

Statistical Analyses

The experimental design for studying the effect of brine pH adjustment on chemical changes in the brine was a split-split plot. To clarify the statistical results, a series of linear regressions sorted by pH adjustment, tank, and rep over time was performed. The parameters, slope, and intercept were then analyzed in a simple one-way analysis of variance (ANOVA), with the pH adjustment as the independent variable. This way the essence of the experiment and chemical changes over time, as affected by the initial pH adjustment, could easily be statistically compared. The software package used in all statistical analyses was the SAS package for Windows (SAS Institute, Cary, NC). Since the texture was measured at the beginning and end of the experiment, a two-way analysis of variance sufficed for all statistical inferences concerning texture.

RESULTS AND DISCUSSION

Rate and Pattern of Acidification in a Model Tank

Acid addition resulted in rapid changes in pH, especially in the top layer of the tank, as illustrated in Figure 3. All four positions indicated a rapid decrease in pH from the initial 3.7 level within 15 min; after 30 min, the pH began to gradually increase, even though acid was still being added to the tank. Continued circulation resulted in the pH approaching an equilibrium value of 3.5 in less than 360 min. Apparently, position number 7 (on the tank wall opposite from the purger discharge) reacted most rapidly, while position number 4 (center of tank) was slowest to change. The differences are probably not significant.

The middle level responses (Fig. 4) show similar trends, i.e., an initial decrease followed by a gradual rise to the equilibrium value. In this case, it appears that the response lagged at the top level values by about 15 min. Again, it appears that the tank center was slowest to respond. The bottom level values (Fig. 5) required more than 30 min to register significant decreases in pH, with position numbers 6 (tank center) and 9 (opposite tank wall) exhibiting the most rapid changes. Position numbers 3 and 12 apparently approached their final values without "overshoot," as exhibited by the other 10 positions.

The average values of pH for the three layers (Fig. 6) illustrate the rapid response to the top and middle layers and much slower reaction of the bottom layer. Overall, it appears that complete and uniform mixing of tank additions requires a relatively short circulation period of 4 hr. Any measurement of tank response to additions introduced through the purging unit should be made at the tank bottom.

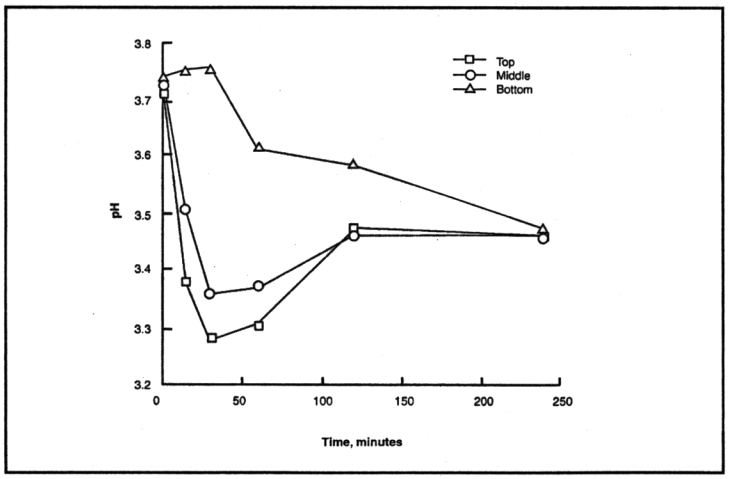

Figure 6. Average rates of acidification at the top, middle, and bottom of the brined cucumbers. Averages are for the four locations at each depth.

Storage Stability Under Laboratory Conditions

Microbial instability of brined cucumbers is initially evidenced by a rise in pH, production of CO_2 and acetic acid, and a reduction in lactic acid concentration. In more advanced stages of instability, propionic acid and later butyric acid also appear. Figures 7-10 summarize the rates of chemical changes that were observed over the 14-month storage period under laboratory conditions (held in 46-oz jars at room temperature). Note that data are presented for cucumbers/brine that had three pH adjustments: unadjusted, pH 3.5, and pH 4.0. Plotted are means of samples from 10 commercial tanks, with solid lines representing predicted values based on statistical inferences, as determined by ANOVA (Table 2).

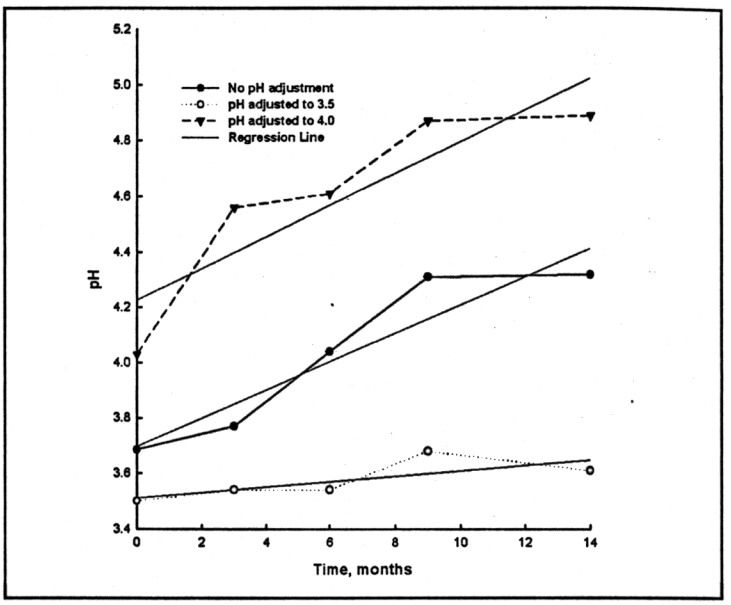

Figure 7. Changes in pH of the brine in fermented cucumbers stored at three inital pH values.

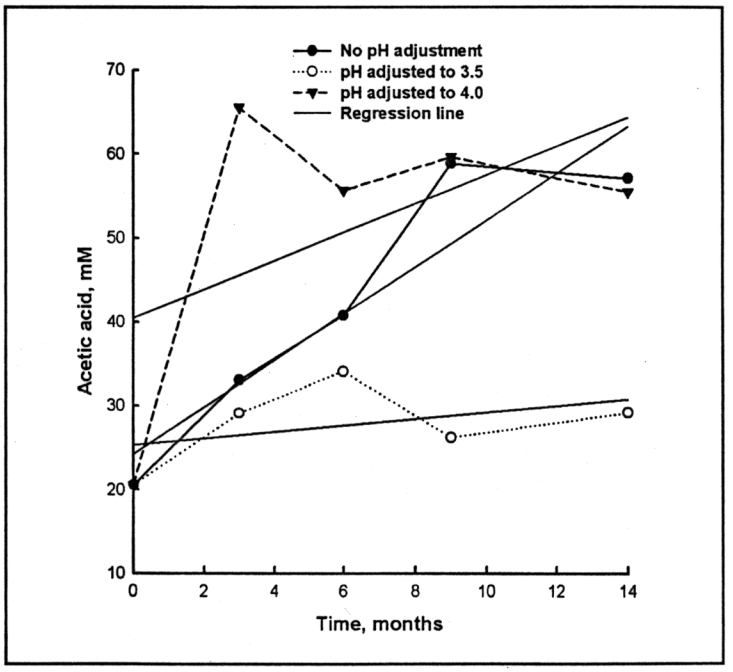

Figure 8. Changes in acetic acid concentrations in fermented cucumbers stored at three initial pH values.

An interesting overall observation relative to pH changes is that the ratios of pH increases were similar ($P > 0.05$) for unadjusted and pH 4.0-adjusted brines, based on predicted slopes (Fig. 7). However, pH change over time was significantly lower ($P < 0.05$) for pH 3.5-adjusted brines. The intercepts were all significantly different from one another, but this was due to the initial pH adjustment. It is interesting to note that the rate was not higher for the pH 4.0 treatment when compared to the no-adjustment treatment. In the pH 4.0 treatment, however, 3 of 20 samples did go into a butyric acid fermentation. We have observed that, when the fermentation goes into

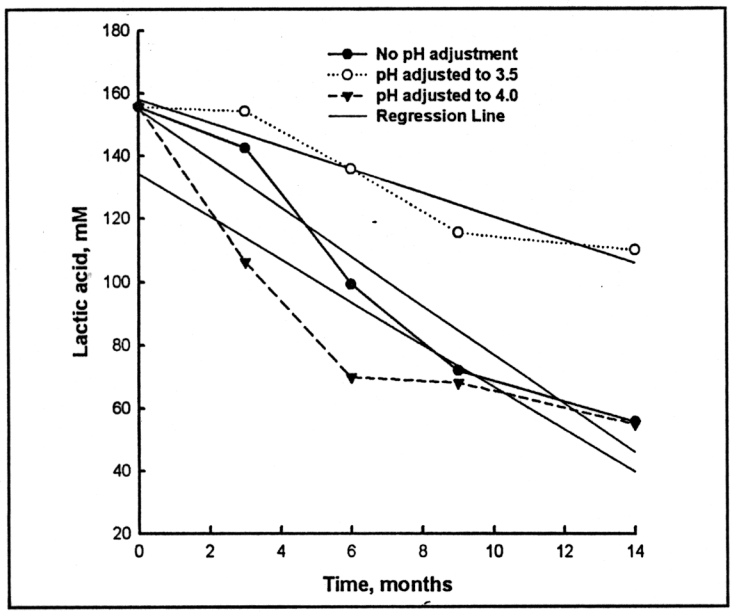

Figure 9. Changes in lactic acid concentrations in fermented cucumbers stored at three initial pH values.

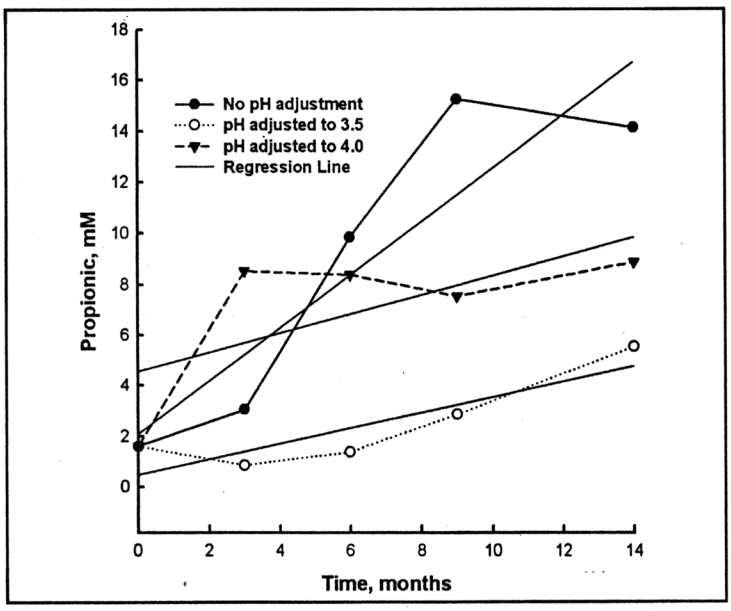

Figure 10. Changes in propionic acid concentrations in fermented cucumbers stored at three initial pH values.

the butyric stage, the rate of depletion of the lactic acid is more rapid than when only acetic acid, propionic acid, and perhaps propanol are produced from the lactic acid. None of the treatments which had no pH adjustment or were adjusted to 3.5 went into a butyric fermentation, as no traces of butyric acid were identified by HPLC on any of the analyses of these samples.

Figure 8 shows that the terminal amounts of acetic acid for the non-adjusted and pH 4.0-adjusted brines are similar, but the rates are significantly different ($P \leq 0.0003$). The production of acetic acid was greatly depressed by pH 3.5 adjustment, the slope not being statistically different from zero ($P > 0.054$). We have no explanation as to why the 3-month reading was 65 mM+ for the 4.0 treatment, dropped approximately 10 mM, and remained at that level for the rest of the storage period. Again, the pertinent finding here is that the pH 3.5 adjustment, while not completely stopping the chemical changes, did slow them down.

The lactic acid changes, as shown in Figure 9, again illustrate the benefits of the pH 3.5 adjustment treatment. The rate of depletion is approximately half that of the other treatments. The rates of lactic depletion are not different for the non-adjustment and pH 4.0 adjustment, while the pH 3.5 adjustment rate is significantly less ($P \leq 0.0001$).

The production of propionic acid (Fig. 10) in the pH 4.0-adjusted brine was virtually over within 3 months. At this time, the propionic acid concentration was 8.5 mM and 8.9 mM and was not appreciably higher at 14 months. Propanol was also formed (data not shown) in the 4.0 and non-adjusted treatments. In a similar manner to the propionic acid production for the 4.0 adjustment, propanol formation was over within 3 months at 17.8 mM, while at 14 months it was 19.8 mM. The non-adjusted brines continued to produce propionic acid and propanol at similar rates over the entire storage period, with the terminal concentrations ending up at 14.1 and 17.1 mM for propionic and propanol, respectively. It is interesting to note that in one treatment an almost 1:1 ratio of propionic acid and propanol was produced, while, in the 4.0 treatment, a 1:2 ratio of propionic to propanol was produced. The pH 3.5 treatment followed a similar pattern to the non-adjusted treatment, but, at a much slower rate. The concentrations of propionic acid and propanol at the 14-month period in these brines were 5.6 and 4.0 mM, approximating the 1:1 production ratio.

Over the 14-month storage period, the 3.5 pH-adjusted brines were more stable in terms of rate of chemical changes. The conversion of lactic acid, a product of the primary initial fermentation, into acetic acid, propionic acid, and propanol was approximately half that of the non-adjusted and pH 4.0 treatments. There were no uncharacteristic odors and noticeable effects upon internal or external color of the brine-stock. The pH 4.0 treatment illustrated what can happen when the pH rises to the point that allows growth of butyric acid-producing bacteria. When this happens, the protective lactic acid is rapidly converted into butyric acid. This happened in 15% of the samples representing the pH 4.0 treatment. The non-adjusted samples varied somewhat, but, for the most part, a "cheesy," non-characteristic, fruity odor was noticed in the majority of the samples by the 6-month storage

Table 2. Analyses of variance on the effects of pH adjustment and time upon the changes in pH and lactic, acetic, and propionic acids.[1]

Source	Df	pH	F-ratio		
			Acetic acid	Lactic acid	Propionic acid
pH adjustment	2	203.05**	57.54**	73.56**	19.61**
Tanks	9	2.77**	3.26*	12.58**	1.94
pH Adjustment x tanks (error a)	18				
Time	4	55.13**	33.58**	197.38**	39.36**
pH Adjustment x time	8	9.66**	8.28*	13.46**	11.33**
Time x tanks (pH adjustment)	108				

[1]*Statistical significance at the 0.05 probability level or less; **statistical significance at the 0.01 probability level or less.

period. All of the pH 4.0-adjusted samples had developed significant headspace pressure (as noted upon sampling by syringe) in the jars by the 3-month period, and 75% of the non-adjusted samples had headspace pressure by 6 months. In previous experiments (data not shown), the headspace gas was analyzed as CO_2. Although none of the non-adjusted samples went into a butyric fermentation, the final pH on some of the samples was as high as 4.5.

Firmness

Cucumbers from all 10 commercial tanks were sampled at zero time and at the end of the 14-month laboratory storage period for firmness evaluation by the Fruit Pressure Tester. Three jars from the 4.0 adjustment treatment samples, which had gone into a butyric fermentation, were omitted at the 14-month period. The initial means, representing 10 cucumbers per tank, ranged from 13.8-11.5 lbs, with an overall mean of 12.6 lbs. For the three treatments, pH adjustments were not statistically different in firmness retention (P >0.1503, Table 3). Time was a significant factor as the mean decreased from 12.6 to 11.5 lbs over the storage period. Although this was a statistically significant drop in firmness (P <0.0034), the decrease of only 1.1 lbs while stored at 26°C indicates the cucumbers maintained their firmness well. It should be noted that the initial firmness (12.6 lbs) is considered to be inferior according to Bell et al. (1955). At the 14-month period, the means of the treatment were 11.7, 11.4, and 11.2 for pH 4.0, no adjustment, and 3.5, respectively.

Table 3. Analysis of variance on the effects of pH and time upon firmness retention in no. 1 salt-stock cucumbers.

Source	Df	MSE	F-ratio[1]
pH Treatment	2	0.872	2.12
Tanks	9	4.187	10.2**
pH Treatment x tanks	17	0.41	
Time	1	7.226	15.6**
Time x Tank (pH Treatment)	9	0.465	

[1]**Statistical significance at the 0.01 probability level or less.

Although no statistically significant differences in cucumber firmness retention during brine storage were attributed to brine pH adjustment, reducing the pH much below pH 3.5 should be viewed with caution. Earlier research revealed that adjustment of size 2B brine-stock cucumbers to 3.0 resulted in a statistically significant reduction in firmness (Fleming et al., 1996). For this reason, pH 3.5 is considered an optimum pH to increase microbial stability without serious adverse effects on firmness retention of cucumber brine-stock.

CONCLUSIONS

This study demonstrated that the microbial storage stability of brined cucumbers can be increased by adjusting the brine pH to 3.5 from higher values with hydrochloric acid after the lactic acid fermentation is complete. Although the chemical changes (including a depletion in lactic acid concentration and rise in pH) were not stopped completely, they were slowed considerably. Firmness of the cucumbers was reduced slightly, but not importantly for all pH treatments over the 14-month storage period. Thus, pH control by acidification may be a favorable alternative to increase the stability of cucumbers brined at relatively low concentrations of salt (4.6-5.6% in this study). The addition of more salt after fermentation to 8-16%, as has been common commercial practice in the past, is becoming a less favorable alternative because of the waste problems generated during processing the brine-stock into finished pickle products.

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*Corresponding author: telephone 919-515-2979, fax 919-856-4361, E-mail: hfleming@unity.ncsu.edu.

Authors Fleming, Thompson and McFeeters are with USDA-ARS, Raleigh, NC; author Humphries is with the Department of Biological and Agricultural Engineering,

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"For Those Who THINK PICKLES"

ABOUT THE COVER:

Bulk storage in brine has been an economic means of extending the processing season of pickling cucumbers since before the 1930's (1). When larger sizes of cucumbers began to constitute a higher proportion of the crop in the 1960's, bloater formation resulted in buoyancy force sufficient to rupture tank heading timbers (2), but purging of CO_2 from the brine reduced bloater damage and buoyancy forces within the tank (3). However, use of high concentrations of salt in brine storage requires washing of the excess from the brine-stock before conversion to finished products, which requires the use of aeration ponds to biodegrade the organic matter (4), but still results in problems in the handling of salt and other non-biodegradable wastes. The use of fiberglass and polyethylene tanks (5) has reduced salt leakage that was prominent with wooden tanks (1-3), but relatively high salt concentrations are still used to serve as insurance against vagaries of nature due to tanks being open to the atmosphere. Closed tanks have been considered by the industry (6), but various factors have resulted in modernized brine yards of open-top, fiberglass and polyethylene tanks and a waste handling system (7). This issue of the journal is devoted largely to summarizing efforts to design and test a pilot system (8) for preserving "process-ready," brined cucumbers with improved quality and reduced wastes, and with intended benefits to the producer and processor of pickling cucumbers.

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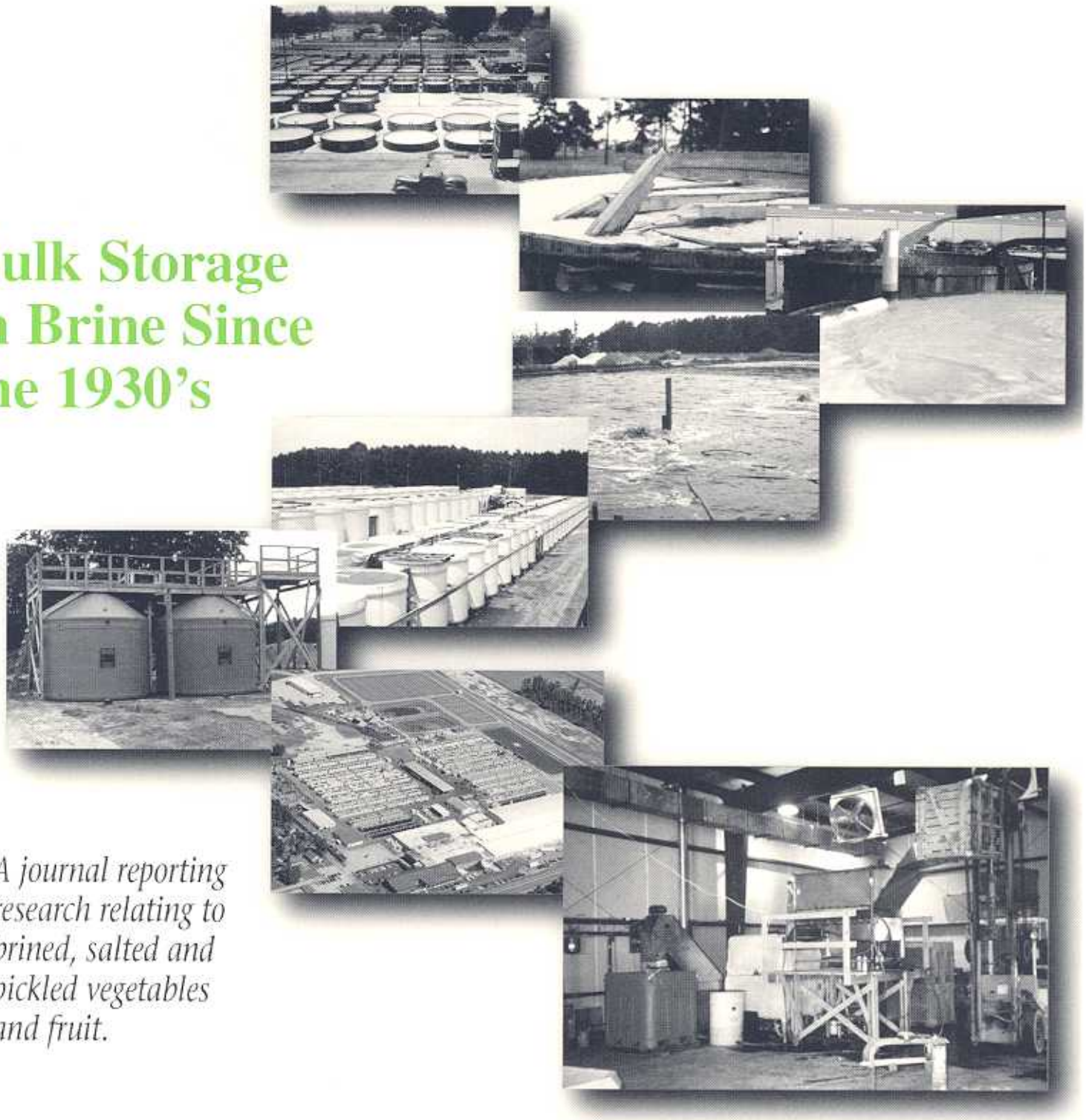

Bulk Storage in Brine Since the 1930's

A journal reporting research relating to brined, salted and pickled vegetables and fruit.