THERMAL AND RHEOLOGICAL PROPERTIES OF BRINE FROM FERMENTED AND SULFITE-PRESERVED CUCUMBERS[1]

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ABSTRACT

Pickling cucumbers may be temporarily preserved by fermentation in brine (6-8% NaCl) or without fermentation in salt-free, sulfite solution (300 ppm sulfite, pH 3.5). Brines obtained from preservation processes are often discarded. Due to environmental concerns, there is increasing consideration for further use of the brine solutions by recycling for use in bulk storage or filtration and incorporation into finished products. Thermal and rheological properties are fundamental to the reuse of the brine. The effect of temperature was determined on the rheological (5-45C) and thermal properties (5-75C) of brine. The properties of the brine samples were found to be significantly different (P < 0.05) from each other and from water. Salt content was the most important factor affecting the thermal and rheological properties of brine. At the same conditions, the values of the properties (thermal conductivity, specific heat, and thermal diffusivity, viscosity) were about 5 to 23% less than the corresponding values for water.

INTRODUCTION

Approximately 40% of the pickling cucumbers produced annually (550,000 tons) in the United States are preserved by brine fermentation (Fleming *et al.* 1995a). Fermentation of cucumbers is typically carried out with 5 to 6% NaCl.

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The salt concentrations may be increased to 15% in cold climates to prevent freeze damage, or to only 8 to 12% to ensure textural and microbial stability during extended storage. The salt concentration must be reduced to 2 to 3% during processing of the brined cucumbers into finished products. This has created disposal and environmental problems for the pickle industry. In addition, many companies cannot meet the 230 ppm chloride limit recommended by the U.S. Environmental Protection Agency for discharge of water into fresh water bodies into which processing waste is discharged (EPA 1987; Humphries and Fleming 1989).

The pickle industry currently recycles about 40% (that exogenous to the cucumber) of the salt used in brining by incorporating it into new brines for fermentation. The salt within the cucumbers must be leached from the fruit during further processing to finished products. This salt-containing leach water is too diluted to be economically reused. Strategies that are now being considered by our laboratory to alleviate this problem include: (1) controlled fermentation of cucumbers (i.e., cucumbers blanched and inoculated with a selected lactic acid bacteria culture) at lower salt levels and recycling or incorporating diluted brine into finished products (Fleming *et al.* 1987, 1995b), and (2) storage of pickling cucumbers in 300 ppm of sodium metabisulfite (McFeeters 1998). With sulfite storage, calcium chloride and HCl are added to the brine for product stability during the storage period. Fermentation does not take place and, thus, the sulfite-preserved cucumbers are free of microbial cells, salt, lactic acid, and other components that are associated with vegetable fermentation.

Brines that are to be recycled or incorporated into finished products have to be reclaimed from the stored pickles. Some of the unit operations that may be carried out on the brine samples include filtration or removal of microbial cells, pumping of the brine, and storage in appropriate containers. The thermal properties (specific heat, thermal conductivity, thermal diffusivity, and density) are needed in the design of suitable storage systems (Mohsenin 1980) due to diurnal and seasonal variations in environmental temperature during brine storage. Brine rheological properties of the brine are important in the design of pumps and handling and filtration systems (Rao 1999; Holdsworth 1971). To our knowledge, information on the thermal and rheological properties of brine from fermented and sulfite-preserved cucumbers is not available in literature. It is expected that the properties of fermentation brines will be different from those of sulfite-preserved brines.

The objective of this study was to quantify and compare the thermal and rheological properties of brines from fermented and sulfite-preserved cucumbers.

MATERIALS AND METHODS

Size 3A cucumbers (diameter of 42.0 to 45.5 mm) of unknown cultivar were obtained from a local processor. The cucumbers were washed in a reel washer and were free of obvious physical damage and disease. Brining of the cucumbers was carried out in duplicate in 1-gal (3.8 L) glass jars containing 50% cucumbers and 50% brine by weight. The cucumbers were preserved by fermentation in 6 and 2% salt levels and in sulfite solution (300 ppm of sodium metabisulfite).

In addition to salt, the cover brine for the fermented cucumbers contained 18 mM of CaOH and 53 mM of acetic acid. The brines were inoculated to contain 10^6 CFU/mL of *Lactobacillus plantarum* MOP-3 MRS broth (Difco Laboratories, Detroit, MI). The cover brines for sulfite treatment contained 30 mM of $CaCl_2$ and 100 mM of acetic acid. HCl was used to adjust the pH of the sulfite-preserved samples to 3.5.

Brined cucumbers were stored in an incubator at 30C for 2 months to allow the fermentation process to occur. Acidified samples preserved with sulfite were also stored for 2 months at 30C. The brine samples were collected after the 2-month storage period and frozen until the time for thermal and rheological properties determination. The samples were thawed and allowed to equilibrate to room temperature (~25C).

The pH, salt level and turbidity (indication of microbial cell population) of the brine samples were also determined. NaCl was measured by titration with standard $AgNO_3$ using dichlorofluorescein as an indicator (Fleming *et al.* 1992). Before rheological and thermal properties measurements, the optical density of the brine samples were determined by reading absorbance of samples at a wavelength of 640 nm in a UV-visible spectrophotometer (model 8452A, Hewlett Packard, Avondale, PA).

Thermal Properties

The measured thermal properties were thermal conductivity (k), specific heat (c_p) and density (ρ). Thermal diffusivity was calculated from the relation below:

$$\alpha = \frac{k}{\rho c_p} \quad (1)$$

Symbols are defined in the Notation section.

Thermal conductivity was determined by the line-heat source probe method. The probe apparatus, constructed according to the recommendations of Sweat

(1986), consisted of a type E thermocouple (0.051 mm diameter), constantan heater wire (0.077 mm diameter), a 23 gauge stainless steel hypodermic needle (houses the heater wire and thermocouple), and a type E thermocouple connector.

To test a sample, beakers containing the 150 mL brine samples were placed in a circulating water bath and allowed to equilibrate to the desired temperature. The probe was inserted into the sample and the power for the heater wire in the probe was turned on. The time and temperature data were recorded by a datalogger (model OM-3000, Omega Engineering, Stamford, CT) at the rate of five readings per second. Thermal conductivity was calculated from the relation (Sweat 1986):

$$k = \frac{Q}{2\pi} \frac{\ln(t/t_o)}{(T - T_o)} \tag{2}$$

The initial time, t_o, in the above equation was set equal to the time when the semi-log plot of time-temperature plot starts to become linear. The power level in the heater was 5.5 W/m. For each time-temperature plot, the slope was found using simple linear regression.

Since preliminary testing showed that the viscosity of brine was close to that of water, data collection was carried out for 25 s to minimize the development of convection currents that may arise due to probe heating (Mohsenin 1980; Sweat 1986). The accuracy of the thermal conductivity measurement procedure was verified by measuring the thermal conductivity of water at 25C. This was measured to be 0.618 W/m K. This is close to the published value of 0.606 W/m K for thermal conductivity of water at 25C (Singh and Heldman 1993).

Specific Heat and Density

Specific heat was measured by means of a Perkin-Elmer DSC 7 differential scanning calorimeter equipped with intracooler II refrigeration unit and dry box (Perkin Elmer Corp., Norwalk, CT). The DSC was calibrated with indium (temperature and enthalpy) and dodecane (temperature) before use. Samples were pipetted (55 μL) into the manufacturer's stainless steel pans and run from 5 to 45C at a heating rate of 3C/min using an empty pan as the reference. Specific heat was calculated by the software provided by the DSC manufacturer. To ascertain the accuracy of the measurements, the specific heat of HPLC-grade water was measured and found to be within 2% of published values.

To obtain density, the weight of brine sample needed to fill a 50 mL volumetric flask was measured. Density was taken as the ratio of mass to volume (50 mL).

Rheological Properties

A controlled stress rheometer (StressTech, ATS Rheosystems, Bordentown, NJ/Rheological Instruments, Lund, Sweden) was used to measure the rheological properties of the brine samples. A temperature-controlled unit connected to the rheometer ensured correct and stable temperature control. The rheometer and the temperature unit were controlled by means of a Windows-based software provided by the manufacturer of the rheometer. Data acquisition, analysis, storage, and retrieval were also done with the software.

A couette attachment was used to evaluate brine viscosity. The "bob" had a diameter of 25 mm, and sheared solutions occupying the 1-mm fluid gap. Shear rates were ramped from 5 to 100 s^{-1} at temperatures of 5, 15, 25, 35 and 45C.

Statistical Testing

Statistical evaluation of collected data on the rheological and thermal properties of the brine samples was carried out by using t test in the ANOVA procedure of Statistical Analysis System (SAS 1996). Statistical testing was performed at the 95% confidence interval.

RESULTS AND DISCUSSION

Visual observation of the cucumbers after 2 months of storage indicated that the fermented and sulfite-preserved cucumbers were of good quality and without any significant bloater damage. Sodium chloride concentrations and pH of the brine samples equilibrated to the desired levels (Table 1). The low turbidity (absorbance at 640 nm) of brine from sulfite-preserved cucumbers confirmed that preservation occurred without the growth of microbial cells. Sample B (2% salt fermentation) was more turbid than Sample A (6% salt fermentation). This is probably indicative of more microbial cells in Sample B. This is consistent with the results of Passos *et al.* (1993a, b). These authors found that growth rate of *L. plantarum* in cucumber juice was highest at salt level of 2% when fermentation was carried out at salt levels that varied from 0 to 12%. The specific growth rates found by these authors at 2 and 6% salt levels were 0.43 h^{-1} and 0.35 h^{-1}, respectively.

Density and Thermal Properties

Densities of the brine samples (Samples A, B and C) were respectively measured to be 1046.1, 1018.0, and 1010.1 kg/m^3, respectively. The values for Samples A and B are close to the published values (Wolf *et al.* 1982) for aqueous solutions containing 2 and 6% NaCl (1014.3 and 1043.1 kg/m^3, respectively). The presence of other products of fermentation (lactic acid and microbial cells) might have contributed to the slight increase in measured values over the literature values.

TABLE 1.
ABSORBANCE, NACL, AND pH OF BRINE SAMPLES USED IN THIS STUDY

	Sample[a]		
	A	B	C
NaCl (%)	6.1	1.9	0.1
Optical density (640 nm)	0.970	1.596	0.0349
pH	3.54	3.64	3.84

[a]A - fermentation at 6% salt level; B - fermentation at 2% salt level; C - sulfite preservation.

The influences of temperature on the thermal conductivity, specific heat and thermal diffusivity of the brine samples are respectively shown in Fig. 1, 2 and 3. Similar to water, thermal conductivity increased with increase in sample temperature. Thermal conductivities for the brine samples were about 7 to 15% less than that of water. Statistical testing ANOVA procedure (SAS 1996) showed that the thermal conductivity values for each sample were significantly different and were significantly less than that of water ($P < 0.05$) within the temperature range tested in this study. The thermal conductivity values at each temperature for the samples were related to temperature using simple linear regression (Eq. 3). Values of estimated parameters a and b are given in Table 2.

$$k=p+qT \tag{3}$$

Similarly, the specific heat and thermal diffusivities (calculated from Eq. 1) of brine samples were significantly different from each other and from water (3 to 10%). Second-order polynomial and linear equations were respectively used to fit the specific heat and thermal diffusivity of the samples to temperature (Eq. 4 and 5). Table 3 gives the values for the estimated parameters from Eq. 4 and 5 and the statistics describing the fit of the equations to experimental data.

$$c_p = v + wT + xT^2 \tag{4}$$

$$\alpha = y + zT \tag{5}$$

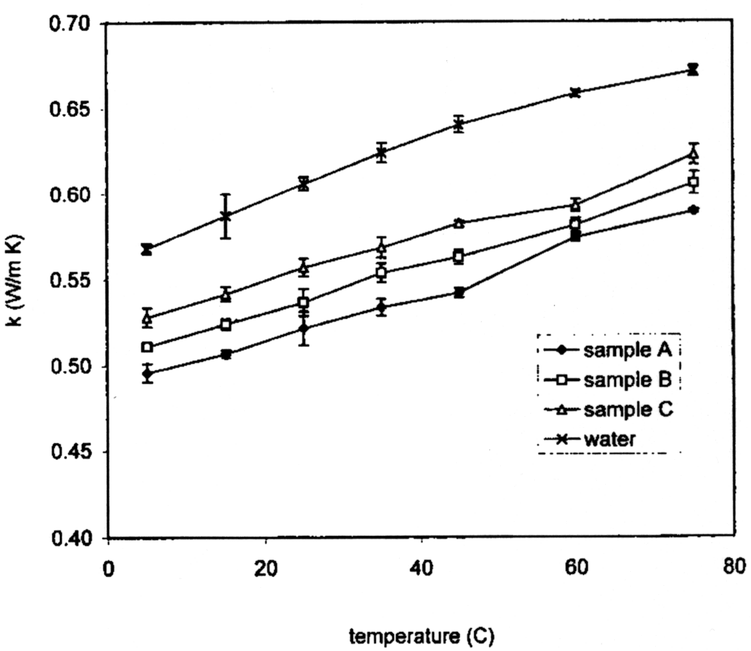

FIG. 1. TEMPERATURE EFFECT ON THE THERMAL CONDUCTIVITY OF BRINE SAMPLES
Sample A - 6% NaCl fermentation,
Sample B - 2% NaCl fermentation,
Sample C - sulfite preservation.

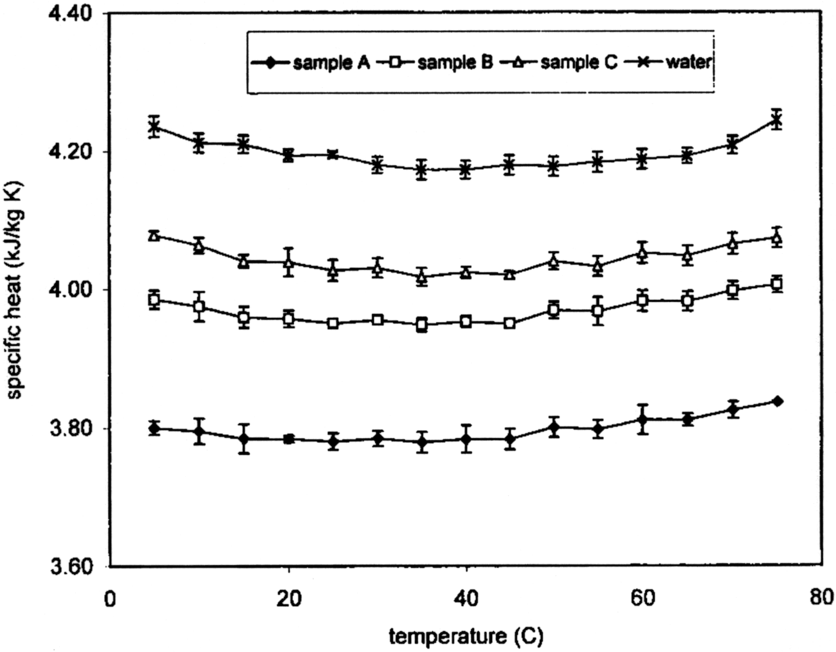

FIG. 2. TEMPERATURE EFFECT ON THE SPECIFIC HEAT OF BRINE SAMPLES
Sample A - 6% NaCl fermentation, Sample B - 2% NaCl fermentation,
Sample C - sulfite preservation.

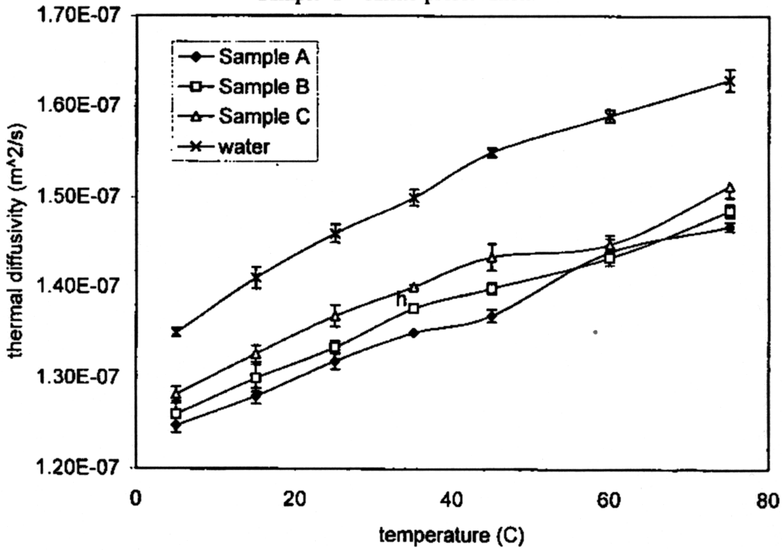

FIG. 3. TEMPERATURE EFFECT ON THE THERMAL DIFFUSIVITY OF BRINE SAMPLES
Sample A - 6% NaCl Fermentation, Sample B - 2% NaCl fermentation,
Sample C - sulfite preservation.

TABLE 2.
VALUES OF REGRESSION PARAMETERS IN EQ. (3)

Sample[a]	Regression Parameters			
	p	q	R^2	s.e.
A	0.487	1.37×10^{-3}	0.990	0.0038
B	0.504	1.33×10^{-3}	0.997	0.0020
C	0.523	1.30×10^{-3}	0.988	0.0038

[a]A - fermentation at 6% salt level; B - fermentation at 2% salt level; C - sulfite preservation.

TABLE 3.
VALUES OF REGRESSION PARAMETERS IN EQ. (4) AND (5)

Regression parameters	Sample[a]		
	A	B	C
Specific heat (Equation 4)			
v	3.81	3.99	4.08
w	1.66×10^{-3}	2.37×10^{-3}	3.30×10^{-3}
x	2.76×10^{-5}	3.46×10^{-5}	4.22×10^{-5}
R^2	0.96	0.94	0.91
s.e.	0.0037	0.0048	0.0063
Thermal diffusivity (Equation 5)			
y	7.37×10^{-6}	7.46×10^{-6}	7.35×10^{-6}
z	-1.60×10^{-8}	-1.57×10^{-8}	-1.51×10^{-8}
R^2	0.987	0.980	0.962
s.e.	4.99×10^{-8}	6.12×10^{-8}	8.15×10^{-8}

[a]A - fermentation at 6% salt level; B - fermentation at 2% salt level; C - sulfite preservation.

Rheological Properties

The temperature dependence of the flow curves for the brine sample from 6% salt fermentation is shown in Fig. 4. Similar curves were obtained for the other brine samples. The points on the curve are the averages of two measurements, and the maximum coefficient of variation between any two points was 3%. It is obvious from the curves that brine samples behave as Newtonian fluids, and, as expected, increasing temperature reduced brine viscosity. The data points were fitted to Eq. 6 (Rao 1999) using the simple linear regression feature of Excel 97.

$$\sigma = \eta \dot{\gamma} \tag{6}$$

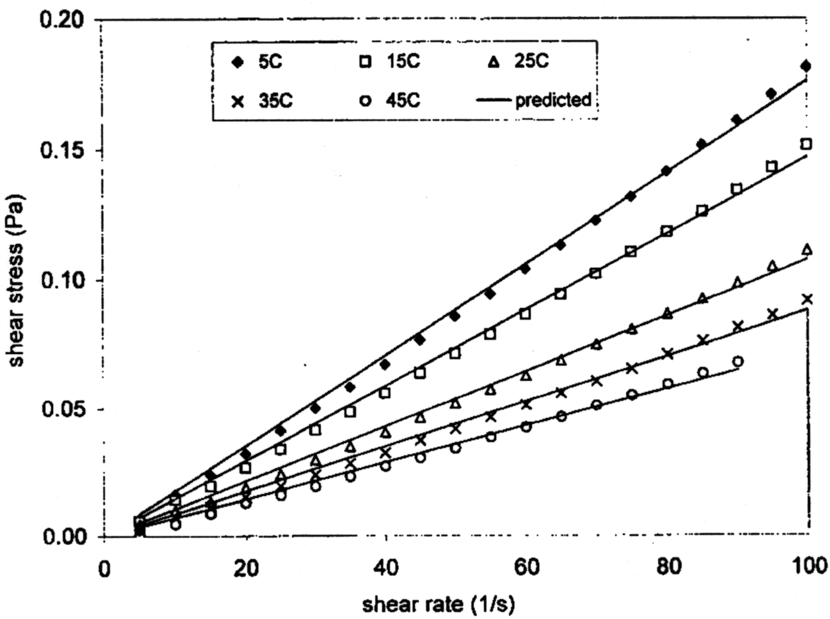

FIG. 4. TEMPERATURE EFFECT ON THE FLOW BEHAVIOR OF BRINE FROM CUCUMBER FERMENTED AT 6% NaCl (SAMPLE A)

Symbols are defined in the Nomenclature section. Calculated viscosities of the brine samples and that of water (measured using the StressTech Rheometer and were within 3% of literature values, Rao 1999) are given in Table 4. All the brine samples were significantly ($P<0.05$) more viscous than water. Sample A was significantly ($P<0.05$) higher in viscosity than the other samples (Table 4). Sample C had the lowest viscosity values. Samples A and B have microbial cells, salt, and other by-products of fermentation present in them. These constituents might have contributed to the higher viscosity values of Samples A and B in comparison to Sample B. It is hypothesized that the viscosity of Sample A was higher than that of Sample B due to the salt level and microbial cell level. This hypothesis was tested and the results presented in the next section.

TABLE 4.
MEAN* VISCOSITY (mPas) OF BRINE SAMPLES AND WATER[a] AT TEMPERATURES OF 5 TO 45C

Temperature (C)	Sample[b]			Water
	A	B	C	
5	1.760[d] ± 0.0052[c]	1.733[e] ± 0.0025	1.640[f] ± 0.0030	1.541[g] ± 0.0074
15	1.467[d] ± 0.0062	1.427[e] ± 0.0013	1.346[f] ± 0.0028	1.264[g] ± 0.0052
25	1.072[d] ± 0.0059	1.040[e] ± 0.00062	0.971[f] ± 0.00090	0.911[g] ± 0.0011
35	0.877[d] ± 0.0033	0.853[e] ± 0.0020	0.786[f] ± 0.0043	0.738[g] ± 0.00040
45	0.714[d] ± 0.0027	0.708[d] ± 0.025	0.633[e] 0.0032	0.598[f] ± 0.0012

[a] measured
[b] A - fermentation at 6% salt level; B – fermentation at 2% salt level; C - sulfite preservation.
[c] standard error of estimate
* means with the same letter in each row are not significantly different ($P<0.05$)

The viscosity values in Table 4 were related to absolute temperature using the Arrhenius-type relationship (Eq. 7). Standard error of estimate and the coefficient of determination obtained from the fit are given in Table 5. There was negative correlation between frequency factor and activation energy.

$$\eta = \eta_{\infty} \exp\left(\frac{E_a}{RT}\right) \tag{7}$$

TABLE 5.
PARAMETERS OF THE ARRHENIUS EQUATION (EQ. 7)

	Sample[a]		
Parameter	A	B	C
Frequency factor (mPa.s)	0.00128	0.00120	0.00081
Activation energy (kJ/mol)	16750	16875	17654
s.e.[b]	0.0498	0.0450	0.0450
R^2	0.991	0.995	0.996

[a]A - fermentation at 6% salt level; B - fermentation at 2% salt level; C - sulfite preservation.
[b]s.e.- standard error of estimate (stress was in mPa during regression).

Despite the significant higher viscosity of Sample A in comparison to Sample C and water, the difference in viscosities is less than 1 mPa s. Calculations from typical brine pumping systems (Fig. 5) indicate that this difference does not significantly affect the power required to pump the brine samples from one location to the other. Therefore, for practical purposes the viscosity of brine from fermented and sulfite preserved cucumbers can be assumed to be numerically equal to that of water. Further calculations indicate that the viscosity of a solution has to be at least 100% higher than water before obtaining a 16% increase in pumping power requirements.

Influence of Microbial Cells and Salt on Rheological and Thermal Properties

We have provided evidence in the previous section that the rheological and thermal properties of the brine samples are different. In an attempt to find a reason for the differences, the rheological properties and specific heat of the following samples were determined at 25C. Microbial cells in Sample A (fermented with 6% salt) were removed by centrifuging at 8,000 rpm for 30 min.

(a) Microbial cells in Sample B (fermented with 2% salt) were removed by centrifuging at 8,000 rpm for 30 min.
(b) Microbial cells in Sample A increased by 500-fold. Cells obtained through centrifuging were resuspended in supernatant.
(c) The salt concentration of the supernatant from centrifuged Sample B was increased to 6%.
(d) The salt concentration of Sample C (brine from sulfite preservation) was increased to 2%.
(e) The salt concentration of Sample C (brine from sulfite preservation) was increased to 6%.

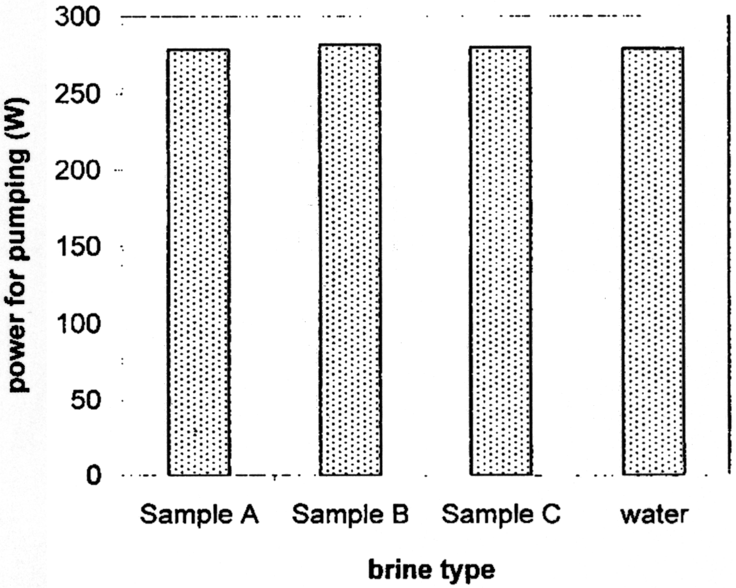

FIG. 5. POWER REQUIRED FOR PUMPING BRINE THROUGH A 1.5" (0.0381 m) DIAMETER PIPE (45 m IN LENGTH)
Sample A – 6% NaCl fermentation, Sample B – 2% NaCl fermentation, Sample C - sulfite preservation.

The results obtained are presented in Table 6. Viscosity and specific heat of samples (a) and (b) show that microbial cells do not significantly contribute to the rheological and thermal properties of brine used for cucumber preservation. The values of viscosity and specific heat for (a) and (b) (Table 6) are similar to the values obtained from noncentrifuged Samples A and B. This was confirmed from the results obtained (c) when the microbial cell concentration in sample A was increased by 500-fold. Increasing the salt content of brine from 2% salt fermentation to 6% decreased their specific heat and viscosity to within the range of values obtained for 6% salt fermentation brine. Therefore, salt concentration is the predominant factor that affects the thermal and rheological properties of brine from cucumber fermentation.

TABLE 6.
INFLUENCE OF SALT AND MICROBIAL CELLS ON VISCOSITY AND SPECIFIC HEAT OF BRINE SAMPLES

Sample Type[1]	Specific Heat (kJ/kg K)	Viscosity (mPa s)[2]
a	3.819	1.071
b	3.970	1.038
c	3.858	1.073
d	3.839	1.071
e	4.006	1.044
f	3.746	1.075

[1] a - centrifuged brine from 6% salt fermentation; b - centrifuged brine from 2% salt fermentation; c - 6% salt fermentation brine with 500% microbial cell concentration; d - salt content of b increased to 6%; e - salt concentration in sulfite preserved brine increased to 2%; f - salt concentration in sulfite preserved brine increased to 6%.

[2] Viscosity at 25C

When the salt contents of the brine from sulfite preservation were increased to 2% (e) and 6% (f), the values of viscosity and thermal properties were higher than those obtained for fermentation brine with 2 and 6% salt level, respectively. Apart from microbial cells and salt, fermentation brine contain solutes such as lactic acid and sugars that were not fermented by microbial cells (Lu 1999). These solutes might contribute to the differences obtained between the properties of the fermented and sulfite preservation brine samples.

CONCLUSIONS

It can be concluded from this study that:

(1) The rheological and thermal properties of brine from fermented and sulfite-preserved cucumbers are significantly different from each other and from water.

(2) Salt content was found to be the main factor influencing the rheological and thermal properties of the brine solutions.
(3) The Arrhenius equation can be used to describe the relationship between brine viscosity and temperature. Thermal conductivity and thermal diffusivity were increased linearly with temperature, while a polynomial relationship existed between specific heat and temperature of brine samples.

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NOMENCLATURE

c_p	Specific heat (kJ/kg K)
E_a	Activation energy (kJ/kg mol)
k	Thermal conductivity (W/m K)
Q	Energy (W/m)
R	gas constant (8.314 kJ/mol K)
T	temperature (C)
T_K	Absolute temperature (K)
T_t	time (s)
p,q,v,w,x,y,z	Constants

Symbols

$\dot{\gamma}$	Shear rate (s^{-1})
ρ	Density (kg/m^3)
η	Apparent viscosity (Pa s)
σ	Shear stress (Pa)
η_∞	frequency factor (Pa s)
α	thermal diffusivity (m^2/s)

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