

THE VOLATILISATION AND CONDENSATION OF ACETIC ACID DURING CANE JUICE EVAPORATION

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Abstract

Severe corrosion problems associated with vapour condensates have been experienced at Felixton Sugar Mill over the past few seasons. Routine analyses from the 1989/90 season onwards have pinpointed the problem as being due to acetic acid in the condensate. The acetic acid has been shown to originate from the acetyl hydrolysis of the hemi-cellulose fraction of bagasse during liming in the diffuser. Laboratory trials were devised to investigate the amount of volatilisation and condensation of acetic acid from juice under varying conditions of brix, temperature and impurity concentrations. The results showed that a combination of high brix and high temperature increased the volatilisation of acetic acid. This explained why Felixton, with 2nd effect Kestner evaporators operating above atmospheric pressure, was experiencing corrosion problems in the vapour 2 condensate. Experiments on the condensation of the vapour showed that the acetic acid was concentrated in the first part of the vapour to condense, explaining the severe localised corrosion found in the factory. A computer model was developed using ionic equilibrium and relative volatility constants for comparison with the experimental data. This model can explain several of the experimental observations.

Introduction

Corrosion caused by acidic vapour 2 (V2) condensates has been an ongoing problem at Felixton. Schäffler *et al.* (1988) showed that the corrosive condensates were due to high levels of acetic acid (HAc) and that these were as a result of acetyl hydrolysis of the bagasse in the diffusers. They felt that modifying the liming procedure would reduce the levels of acetate in the mixed juice and that this would overcome the V2 condensate problem. Beckett and Graham (1989) investigated the factors causing acetate production and found that it correlated with the strength of the milk of lime and fibre retention time. However, while these findings and recommendations are undoubtedly correct, factory results over the past three seasons show that the required improvements have not been achieved. Acetate levels in both the mixed juice and condensates are increasing at Amatikulu and Felixton. This is shown in Table 1.

Table 1

Factory results 1990-92

	Felixton					Amatikulu				
	Acetate* (ppm)			pH		Acetate* (ppm)			pH	
	MJ	V1	V2	V1	V2	MJ	V1	V2	V1	V2
1990	53	7	20	7,3	6,1	30	1	3	7,0	7,0
1991	65	24	25	7,5	5,9	31	3	7	8,0	7,4
1992	92	14	42	7,9	6,0	37	7	19	8,0	7,0

* Values obtained for acetate concentration were determined as acetic acid by gas chromatography and are reported in the paper in terms of mass of acetic acid.

Tests done during the 1990 season comparing corrosion rates measured in the Felixton diffusers with and without liming clearly showed that liming to a juice pH of 5,5-5,8 reduced corrosion to 10-20 MPY from 40-50 MPY (MPY = mils per year, one mil is one thousandth of an inch). This confirmed the results of Beckett and Graham who found that liming was associated with a drop in corrosion rate to between ¼ and ½ of that when the diffuser was unlimed. On the other hand, they established that corrosion in the vapour space was not reduced by liming. On the whole, these results indicate that liming mild steel diffusers to prevent corrosion of the diffuser is necessary and will be a standard operating practice for the foreseeable future. This will however result in increased levels of acetate in juice, resulting in corrosive condensates from evaporation.

Corrosion due to V2 condensate has been severe at Felixton and is most noticeable wherever the first part of the vapour starts condensing. For example, the steam chest inlet on the A1 continuous pan calandria had to be completely replaced. Various coatings have been tried inside the steam lines and calandrias with mixed success and commercial mixtures of amines and ammonia have been dosed into the vapour lines with dubious benefits.

In the light of the work done previously and the fact that liming of diffusers will be ongoing, it was decided to investigate conditions that influence the volatilisation of HAc from clear juice during evaporation. The objective was to be able to recommend changes to the operating conditions that would minimise acid volatilisation. If this was not possible then a method of removing or neutralising the effects of the HAc in the condensate would be sought. The work was done in three parts.

- Condensates were collected at different points and under different conditions in the factory and were analysed for pH and acetate concentration. This was in addition to the routine analysis of V1 and V2 condensates and mixed juice.
- Trial evaporations were run in the laboratory using factory juices and synthetic solutions. Conditions were varied and the condensates and feedstock were analysed.
- A computer model was derived using ionic equilibrium and relative volatility constants and this was fitted to the experimental data obtained in the factory and laboratory trials.

Methods

Factory work

The main purpose of the factory work was to establish the difference between the first material to condense which has been called *initial condensate* and the *bulk condensate* containing all the condensed vapours. A sample point was fitted at the inlet to the steam chest of the A1 continuous pan and was designed to collect initial condensate. The bulk condensate from the pan calandria was collected simultaneously. These samples were then analysed for pH and acetate concentrations.

Tests were also done on the same pan using a small surface condenser which had a variable cooling water feed. Vapour was taken from the steam entry chest and passed through the condenser. The cooling water flow was varied to condense different percentages of the vapour. The condensate samples were analysed for acetate concentration in relation to the percentage of vapour condensed.

Laboratory work

Benchtop evaporations. Initial laboratory evaporation tests were carried out on artificial solutions to determine the effect of calcium, ammonia and sugar on the volatilisation of acetic acid in solution. Various artificial solutions (2 l, each containing 200 ppm acetic acid plus other components) were batch evaporated at atmospheric pressure. Aliquots (200 ml) of condensate were collected and analysed. The pH values of all solutions were measured at the start and each solution was evaporated to approximately 35°Bx. Since evaporation rates were slow, the equipment was modified to increase these. Further tests were then performed on factory juices to determine the effect of various process conditions, i.e. brix, temperature and impurity concentrations on volatilisation of acetic acid. Evaporations were performed in the laboratory in a 5 l reaction flask on FX juice from the 1st effect at atmospheric pressure and also under vacuum, i.e. 80 kPa and 50 kPa. (Note: all pressures quoted in this paper are absolute pressures). To determine the effect of partial condensation, the equipment was modified to collect the initial condensate as well as bulk condensate. Condensate samples were collected in 200 ml aliquots and corresponding juice samples were also taken.

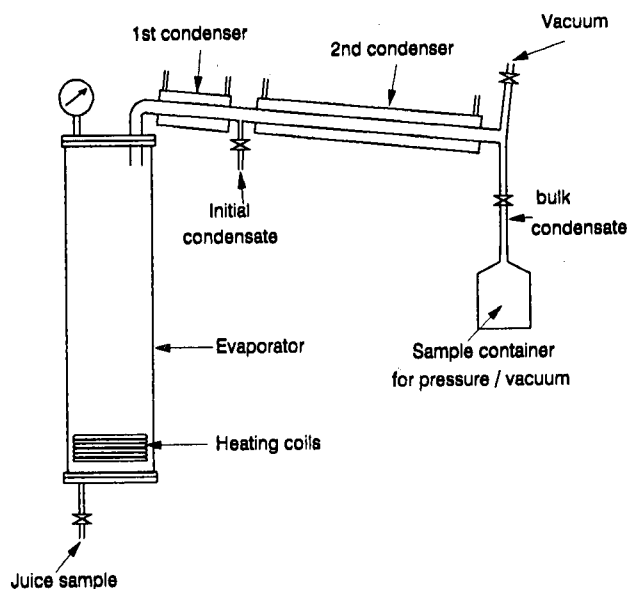


FIGURE 1 Laboratory evaporation apparatus.

Pilot evaporator. To obtain data for the effect of pressure and higher temperatures on volatilisation of acetic acid, evaporations were performed at 120 kPa in a 15 l pilot evaporator. Tests were repeated at atmospheric pressure and at 80 kPa using the same feed material to obtain data for comparison. Evaporations were also performed on AK juice dosed with increasing amounts of calcium acetate.

In all cases the condensate samples were analysed for pH and acetate and the juice samples for brix. Values obtained for acetate ion concentration were determined as acetic acid

by gas chromatography and are reported in terms of mass of acetic acid on sample. The results shown in graphs were obtained on analyses of consecutive 200 ml aliquots of condensate taken during evaporation, and corresponding juice samples taken from the evaporator.

Factory Results

Initial and bulk condensate samples were collected from the A1 continuous pan during three separate periods. The first was during normal running conditions, the second during attempts to dose NH₄OH into the juice and the third involving dosing amines into the V2 line. The results in Table 2 show that the pH of the initial condensate is considerably lower than that of the bulk due to the high acetate and probably higher acetic acid concentrations. Secondly, dosing has no measurable effect on the pH of either the initial or bulk condensates.

Table 2

Average acetate concentration and pH of Felixton V2 condensate

Date	Dosing	No. Samples	Initial condensate		Bulk condensate	
			Acetate ppm	pH	Acetate ppm	pH
30/09	None	4	110	5,4	35	7,3
24/10	NH ₄ OH	5	117	5,5	32	7,4
21-28/11	Amines	35	92	5,5	24	7,0

Figure 2 is a plot of all the individual results for initial and bulk condensates and shows that corrosive condensates, i.e. condensates with a pH < 7 can be expected to start occurring when the acetate level exceeds 20 ppm.

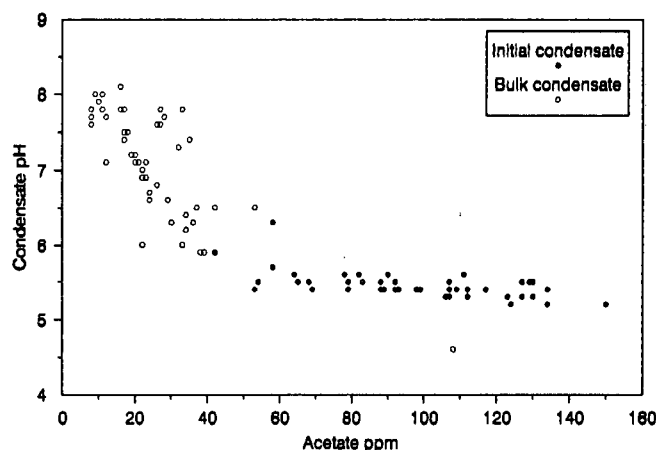


FIGURE 2 pH vs acetic acid concentration for V2 condensate.

The factory trial using the variable condenser yielded a number of samples of condensate which ranged in composition from 100% of the vapour condensed down to 4% condensed. The effect on the acetate concentration and pH of the condensate is shown in Figure 3. It is clear that the equilibrium condensate, i.e. the first drop to condense must contain high concentrations of acetate giving a pH of ± 5 which is in line with the previous factory results.

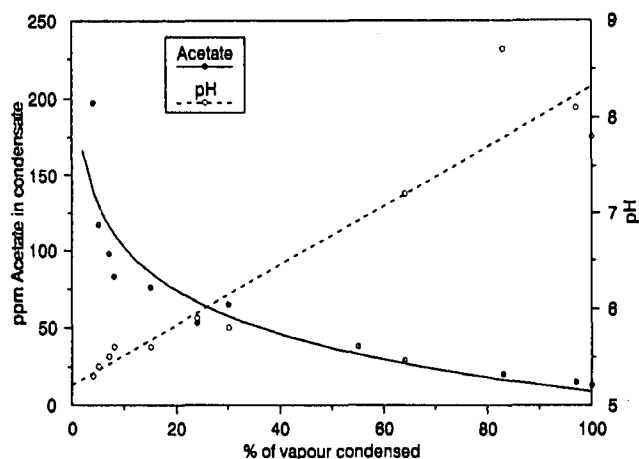


FIGURE 3 The effect of the portion of vapour condensed.

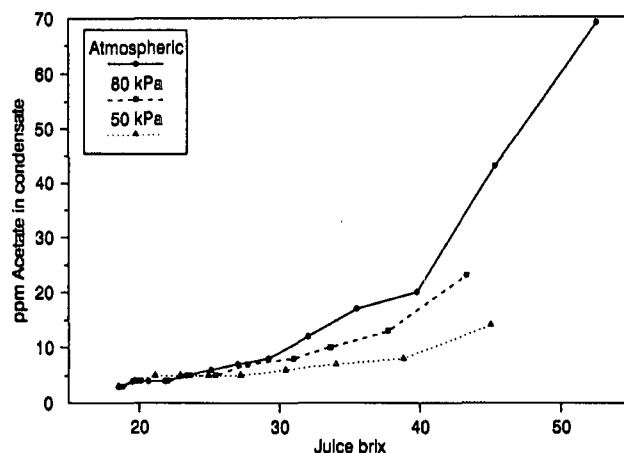


FIGURE 4 Acetate concentration in condensate vs juice brix.

Laboratory Results

Artificial solutions

The results in Table 3 show that the amount of acetic acid volatilised increases in consecutive condensate aliquots. This is to be expected as acetic acid has a higher boiling point (118,1°C) than water. The addition of either calcium hydroxide or ammonia increases the pH and suppresses volatilisation. Sugar however appears to increase volatilisation. Tests 2 and 3 show that the effects of both calcium hydroxide and ammonia (at stoichiometric levels) on volatilisation are similar. This suggests that the volatilisation is more dependent on pH than on specific ions.

Effect of brix

Juice from FX 1st effect evaporator at 18°Bx was evaporated, using the 5 litre glass benchtop evaporator at atmospheric pressure, 80 kPa and 50 kPa. Figure 4 shows that acetic acid volatilisation increases with increasing brix, the rate increasing significantly at ± 40° brix. It is also higher with increasing absolute pressure and consequently higher temperatures during evaporation.

Effect of absolute pressure/temperature

FX juice from the 2nd effect evaporator was diluted to approximately 23°Bx and evaporated in the 15 litre pilot plant evaporator at 120 kPa, atmospheric pressure and 80 kPa. The results in both Figures 4 and 5 show a higher degree of volatilisation as the pressure at which evaporation is carried out is increased.

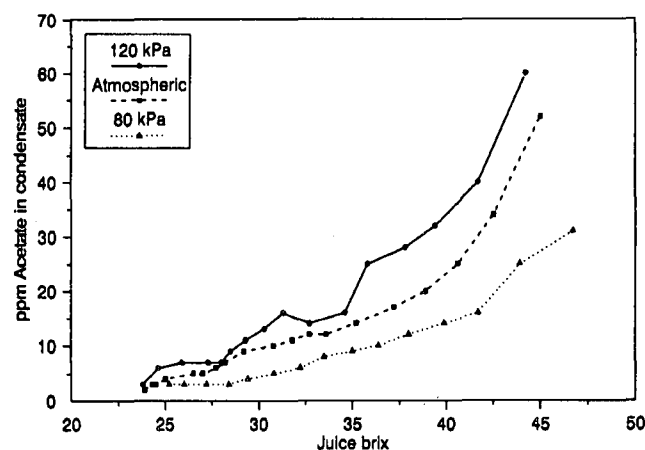


FIGURE 5 Acetate concentration in condensate vs juice brix.

pH of bulk condensates

pH values show a decrease with increasing pressure which corresponds with the higher acetic acid levels at higher pressures. A number of the laboratory evaporations showed an initial steep rise in pH before the expected drop. The reason for this increase has not yet been established and is probably a peculiarity of the batch evaporation procedure. Figure 6 shows the condensate pH values for the same trial as Figure 5. The initial pH increase is most noticeable in the atmospheric pressure evaporation.

Table 3
Artificial solutions containing 200 ppm Acetic acid

Test	Other constituents	pH	Acetate in condensate (ppm)						
			a	b	c	d	e	f	g
1	H ₂ O	3,7	120	120	125	140	140	160	
2	66 ppm Ca + H ₂ O	5,1	30	40	40	40	45		
3	60 ppm NH ₃ + H ₂ O	5,2	20	30	30	40	50		
4	12°Bx sugar	3,6	130	140	155	165	180	200	250
5	30 ppm Ca + 12°Bx	4,4	95	100	105	110	120	140	140
6	80 ppm Ca + 12°Bx	5,4	15	15	15	15	20	30	40

Samples a – g were consecutive 200 ml aliquots of condensate collected during evaporation. In tests 2, 5 and 6 Ca(OH)₂ is added but is expressed as Ca

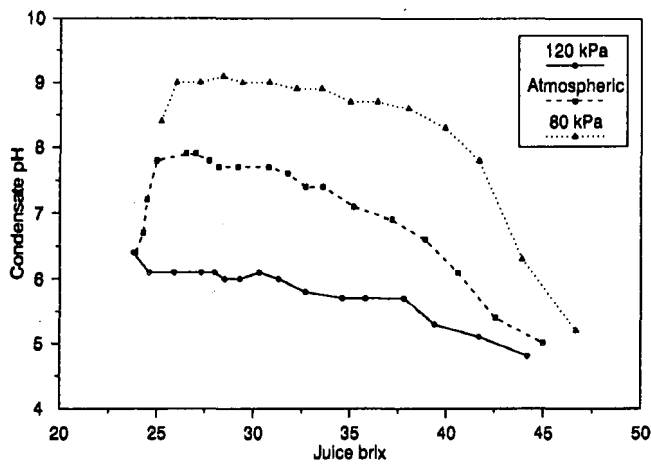


FIGURE 6 Condensate pH vs juice brix.

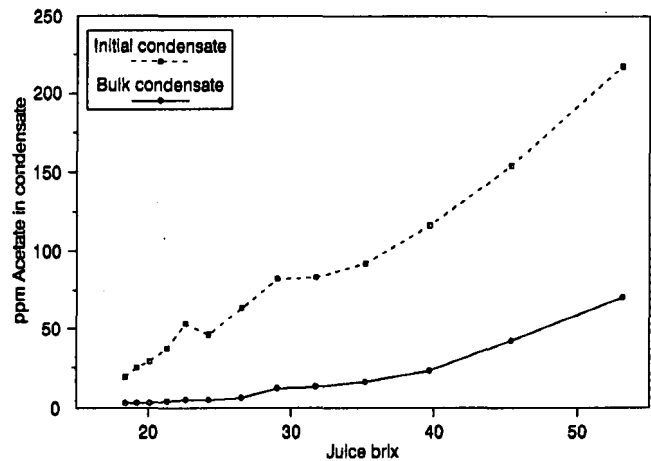


FIGURE 8 Comparison between initial and bulk condensate.

Effect of acetate levels in the juice

AK juice from the 1st effect was analysed and found to contain 100 ppm acetate. Calcium acetate equivalent to an additional 100 and 300 ppm was added to two samples of the juice. The samples now contained 100, 200 and 400 ppm acetate and the respective pH values measured were 6,7; 6,5 and 6,7 indicating that the addition of calcium acetate had no effect on the juice pH. The three samples of juice were evaporated in the pilot plant evaporator at atmospheric pressure. The results are shown in Figure 7 and although acetic acid volatilisation does increase with increasing initial acetate concentration, the effect of brix appears to be of far greater significance. Had the acetate ion concentration been increased by adding acetic acid and no extra calcium ions, the effect on acetic acid volatilisation would have been far greater.

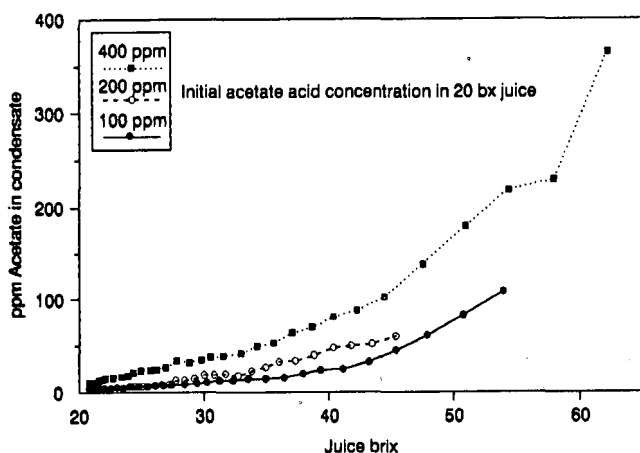


FIGURE 7 Effect of acetate in feed on acetate in condensate.

Effect of initial condensation of vapour

FX juice from the 1st effect was evaporated in the bench top evaporator modified to collect initial condensate, at atmospheric pressure. Initial condensate and the corresponding bulk condensate were sampled and analysed throughout the evaporation. The results in Figure 8 confirm those found in the factory, showing a much higher concentration of acetate in the initial condensate when compared with the bulk condensate.

Simplified Model

In the juice there are many interactions between the undissociated molecules and the ions. In most cases, analytical techniques cannot give the respective concentrations of molecules and ions, particularly under operating conditions but it is important to know these compositions, especially when it comes to distillation, because it is only the non-dissociated molecules which exert a vapour pressure. For this reason, a mathematical model was constructed to calculate the concentrations of those components of interest and to help explain some of the laboratory and factory observations.

Basic assumptions

- The system is in equilibrium. This would appear to be a fair assumption, as ionic equilibria in solution usually proceed rapidly, particularly at temperatures around 100°C, and because of the relatively long residence times of the solution, both in the laboratory tests and in the evaporators in the factory.
- It is not possible to take into account all the components of the juice. For the model, the juice was assumed to be a mixture of the following components in addition to water:

Sucrose: Because this component is present at high concentration, it might interact with other components. Here it was assumed only to reduce the available water component with increasing brix.

Acetic acid: This will be partly in molecular (non-ionised) form, and partly in the ionised form.

Ammonia: Similarly this will be partly in the molecular form and partly in the ionised form.

Ca(OH)₂: To balance all the foregoing to the correct acidity or pH, there has to be a certain amount of alkaline cation which, for convenience, was assumed to be the Ca⁺⁺ of Ca(OH)₂. Most of the total cations (Ca⁺⁺, K⁺, etc) in the juice will be associated with anions of a strong acid, e.g. Cl⁻, and will therefore have essentially no effect on the ionic equilibrium of the aforementioned components. Therefore, in what follows, Ca⁺⁺ concentration will refer only to the alkaline cations which are in surplus to those which belong to the salt of a strong acid-strong base combination. It is assumed that the mass of these ions in the solution remains constant during evaporation. In reality, buffer action from various other weak acids and bases in the juice, as well as degradation reactions in the juice during evaporation, could

alter the acid-base equilibrium and thus the amount of 'balancing' Ca^{++} ions.

Once the components are mixed, they will no longer be identifiable in their original form, due to the interactions.

- (c) The solution is assumed to be ideal, particularly in that the constituents behave as though it were dilute, when the activity of a component can be considered as equal to its concentration. This should be true for the above-mentioned ions. However, the total dissolved salts are present in a fairly high concentration, and even more so the sucrose, and this could cause deviations from ideality by interfering, particularly at higher brixes.

Ionic equilibria

Water has the following dissociation relationship:

$$[H^+][OH^-] = K_{H_2O} \quad (1)$$

where the brackets [] designate the concentration of the substance in question, in g-equivalent/litre.

It is however important to note that, in the case of water, the equilibrium constant is strongly dependent on temperature. At 25°C $K_{H_2O} = 1,0 \times 10^{-14}$, but at 100°C it is, according to Schäffler (1987), as high as $K_{H_2O} = 5,8 \times 10^{-13}$

The equilibrium relationship for acetic acid is:

$$\frac{[H^+][Ac^-]}{[HAc]} = K_{HAc} = 1,75 * 10^{-5} \text{ at } 25^\circ\text{C} \quad (2)$$

The equilibrium for ammonia in solution is:

$$\frac{[NH_4^+][OH^-]}{[NH_3]} = K_{NH_3} = 1,74 * 10^{-5} \text{ at } 25^\circ\text{C} \quad (3)$$

The total cations should balance the total anions, i.e.

$$[Ca^{++}] + [H^+] + [NH_4^+] = [Ac^-] + [OH^-] \quad (4)$$

In these four equations, there are four unknowns, namely, $[NH_4^+]$, $[Ac^-]$, $[OH^-]$ and, depending on the particular calculation, either $[H^+]$ or $[Ca^{++}]$. The given concentrations are: total ammonia [N], total acetate [A] and, again depending on the particular calculation, either $[Ca^{++}]$ or $[H^+]$.

Liquid-vapour equilibria

In this simplified model, the only substances distilled, besides the predominating water vapour component, will be the vapours of HAc and NH_3 .

The concept of relative volatility is used:

$$\alpha = \frac{\text{Fraction of component in vapour}}{\text{Fraction of component in liquid}}$$

Here the "component" in question would be either HAc or NH_3 , the fraction of which in each case is considered relative to the predominating water. The definition of 'fraction of component in vapour' is quite straightforward, but for the case of the liquid (juice), one also has to consider the significant amount of sucrose in the juice. Sucrose contains 11 oxygen atoms and according to Allen *et al.* (1974) and Kelly and Mak (1975), each sucrose molecule can be surrounded with up to 11 water molecules which satisfy its active sites. This bound water together with the sucrose were considered as unavailable when calculating the fractions of components in juice.

Thus:

$$\begin{aligned} &\text{Available Mass of } H_2O \\ &= \text{Mass of juice} * \frac{100 - Bx - n * Bx * MW_{H_2O} / MW_{Suc}}{100} \end{aligned}$$

where

MW_{H_2O} = Molecular mass of water (= 18)

MW_{Suc} = Molecular mass of sucrose (= 342)

n = Number of "unavailable" molecules water/molecule sucrose

A value of n = 10 was found to give good results, particularly in accounting for the steep rise in HAc distilling over at higher juice brixes.

Evaluation of data obtained from Ohe (1989) provides a relative volatility of HAc of $\alpha_{HAc} = 0,7$ for dilute solutions. In this work, better fits were obtained with $\alpha_{HAc} = 0,6$. Possibly the value of α changes due to the extremely low HAc contents and/or the interference of sucrose and other substances.

Using data from Perry and Chilton (1973), a relative volatility for NH_3 of $\alpha_{NH_3} = 17$ was calculated.

The following equations thus arise:

$$\frac{\text{Mass HAc in vapour}}{\text{Mass } H_2O \text{ in vapour}} = \alpha_{HAc} * \frac{\text{Mass non-dissociated HAc in juice}}{\text{Mass available } H_2O \text{ in juice}} \quad (5)$$

$$\frac{\text{Mass } NH_3 \text{ in vapour}}{\text{Mass } H_2O \text{ in vapour}} = \alpha_{NH_3} * \frac{\text{Mass non-dissociated } NH_3 \text{ in juice}}{\text{Mass available } H_2O \text{ in juice}} \quad (6)$$

At the low concentrations of HAc and NH_3 encountered, fraction and ratio of component can be used interchangeably.

Operation of the model

The equations (1) to (4) for ionic equilibrium and (5) and (6) for the liquid-vapour equilibrium are applied to the model to calculate the compositions of the following:

Juice. The balancing effective $[Ca^{++}]$ concentration cannot be determined analytically, but the pH of the initial sample can be measured at 25°C, and from that the hydrogen ion concentration $[H^+]$, i.e. $[H^+]$ becomes a given value. The other unknowns, including $[Ca^{++}]$, can be solved from the above equilibrium relationships (1) to (4), using the water equilibrium constant K_{H_2O} applicable to 25°C. This $[Ca^{++}]$ concentration is assumed to remain constant for the duration of the distillation/concentration operation.

The simulation is performed over the same distillate sampling intervals as the laboratory distillation of the juice. The equilibrium constant K_{H_2O} applicable to the distillation temperature of 100°C at atmospheric pressure is used in the equilibrium equations, and $[H^+]$ now becomes one of the unknowns. The equations are solved, yielding the concentrations of non-ionised HAc and NH_3 .

Bulk condensate. Having determined the concentrations of non-dissociated HAc and NH_3 in the juice, the corresponding components in the vapour are calculated from equations (5) and (6). At the same time the total acetate and total ammonia in the juice are reduced by those respective amounts for the next interval of the distillation. Once the vapour is condensed, HAc and NH_3 become the total acetate and the total ammonia in the condensate.

The same ionic equilibrium equations (1) to (4) apply to the condensate, except that there is no $[Ca^{++}]$, which remains in the juice. From these equations the pH of the bulk condensate can be calculated.

Initial condensate. In the limiting case, the initial condensate can be considered as the first few drops condensed from the vapour, and it will be in equilibrium with the vapour. By using the liquid-vapour equations (5) and (6) and the vapour composition, the total acetate and ammonia concentration of the initial condensate can be calculated. The ionic equilibrium and pH are calculated from equations (1) to (4).

Results of the model with artificial solutions

Figure 9 shows the distillation results from a solution consisting of 200 ppm HAC in a sugar solution of 20° brix. The predicted acetic acid content of the distillate is higher than measured. Figure 10 shows the distillation results from a solution made up of 200 ppm HAC and 60 ppm NH₃. The pH of the distillate is higher than for the previous case, in view of it now being a mixture of HAC and NH₃. It will therefore also contain ammonium acetate as a dissolved salt. Figure 11 shows the distillation of a solution of 200 ppm acetate and 30 ppm calcium hydroxide in 12° brix sucrose.

Results of the model with mill juices

Not knowing the effective equivalent amount of ammonia in the juices, various quantities of NH₃ had to be tried for the best fit to results. The amount of ammonia assumed in the initial juice will obviously affect the amount of calculated balancing Ca⁺⁺ ions per equation (4).

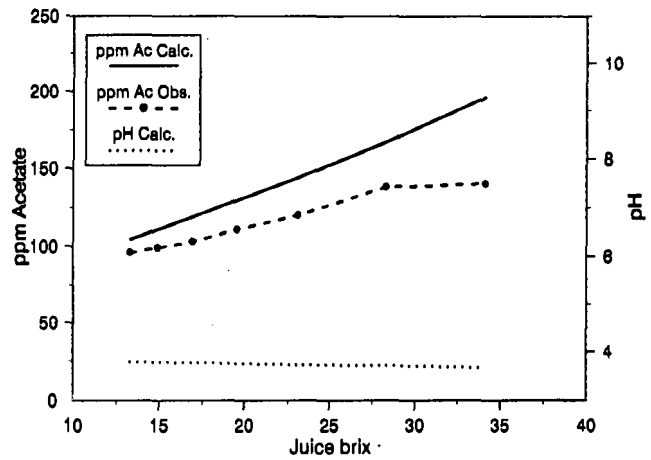


FIGURE 11 Artificial solution of 200 ppm HAC, 30 ppm Ca(OH)₂ and 12° brix.

Figure 12 shows the bulk condensate results for a 1st effect juice sample from Felixton mill. The steep increase in acetate concentration with increased brix is reasonably catered for by the assumption of 10 'non-available' molecules of water per molecule of sucrose. According to the model, the pH starts off at a high value and from there continues dropping, but the observed pH shows the previously mentioned strange 'hump'.

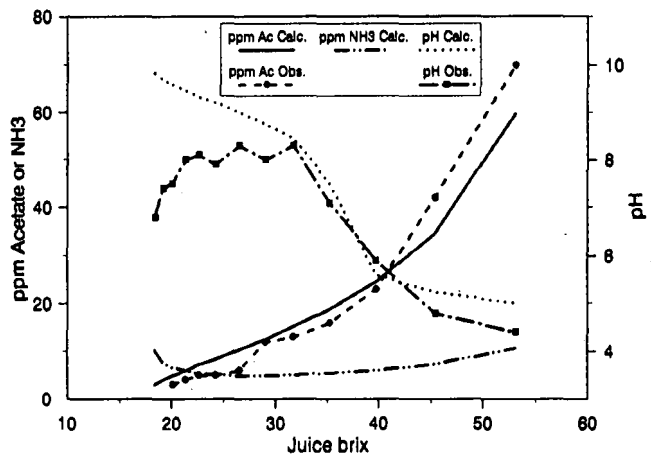


FIGURE 12 Bulk condensate for Felixton mill juice.

Figure 13 shows the analyses of the corresponding initial condensates. Both the predicted and observed acetate contents are much higher than for the bulk distillates.

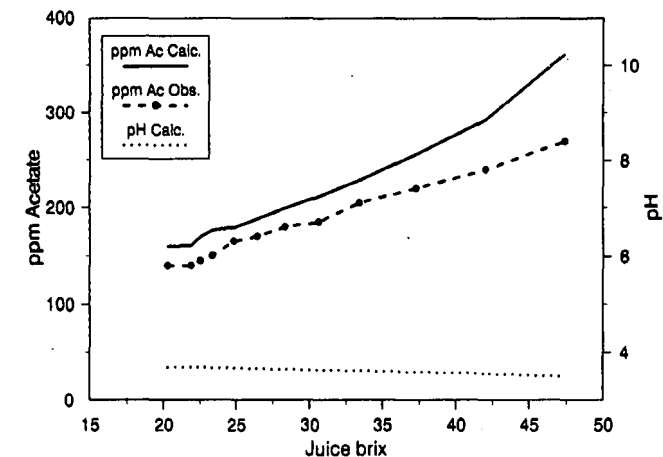


FIGURE 9 Artificial solution of 200 ppm HAC and 20° brix.

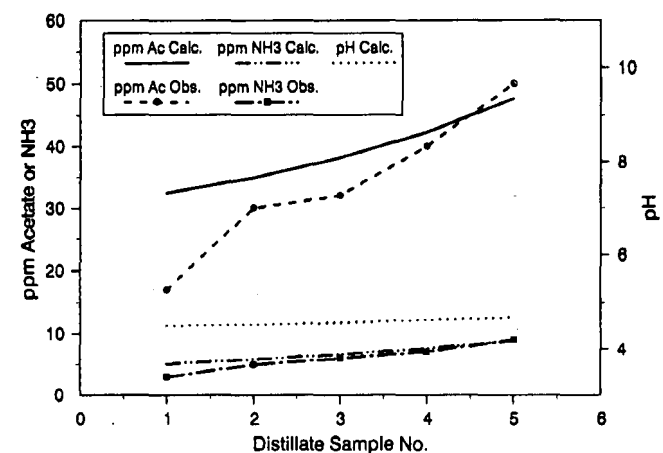


FIGURE 10 Artificial solution of 200 ppm HAC and 60 ppm NH₃

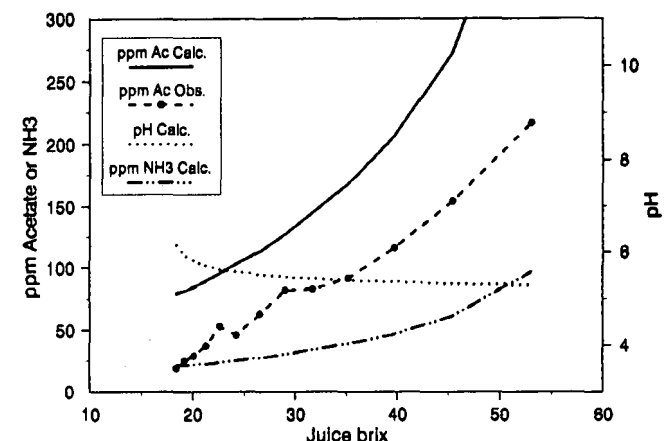


FIGURE 13 Initial condensate for Felixton mill juice.

In general, as the distillation progresses, the amount of NH_3 lost from the juice will be relatively greater than the loss of acetate, due to its higher relative volatility α_{NH_3} . The higher the NH_3 content of the juice, the higher will be its loss of NH_3 and therefore the greater its drop in pH, with consequent higher release of HAc during the later intervals in the distillation.

Conclusions

- The amount of acetic acid in the condensate increases rapidly as the distillation progresses which causes the pH of the condensate to drop. This cannot be explained merely by the increased acetate concentration in the solution and is thought to be as a result of the 'binding' of ± 10 water molecules to each sucrose molecule.
- Increasing the pressure and hence the temperature at which the evaporation is carried out increases the amount of acetic acid that is distilled from the juice. The reason for this has not been fully explained as the published data on the change of relative volatility with pressure cannot account for this effect.
- The acetate concentration in the initial condensate is several times higher than in the bulk condensate, leading to a much lower pH in the initial condensate. This effect is explained by the model, which also explains the localised severe corrosion of factory plant where the first condensing occurs.
- The amount of acetate in the juice has an effect on the quantity of acetic acid distilled into the condensate. Reducing the acetate in the juice is one way of reducing corrosion.
- The earlier concentrations of acetic acid distilling over are far lower than would be expected from the acetate concentration in the juice and this is due to the amount of acetate which is in dissociated form.
- There were two effects which are probably peculiar to the batch evaporations performed in the laboratory. These were:
 - (a) The drop in pH of the juice during evaporation was greater than that expected purely due to the release of ammonia and the concentration of acetate. This was

probably due to degradation of sugars and will have increased the quantity of non-dissociated acetic acid in the juice, thus increasing the quantity of acetic acid volatilised.

- (b) The pH curve of the bulk condensates collected in the laboratory equipment rises to a peak after which it drops in accordance with the model. This suggests a volatile acidic compound coming off during the early stages of the batch evaporation.

The work covered in this paper has answered some of the questions about the causes and reasons for the corrosive nature of the Felixton V2 condensates, yet a number of areas are still unclear. Further development of the model will take place, primarily as a continuous stirred tank model so as to approximate more closely the factory situation and this will require corresponding laboratory simulations. The use of additives to alter the pH of the juice and condensates will also receive attention both in the laboratory and the factory.

Acknowledgements

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