ALKALIZATION PRETREATMENT OF THIN JUICE SUPPLIED TO WEAK ACID CATION (WAC) THIN JUICE SOFTENING

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Introduction

Operation of Weak Acid Cation (WAC) thin juice softening requires that the concentration of limesalts in hard thin juice supplied to the WAC ion exchange cells be less than 0.100g CaO/100dsS. During beet storage the organic acid concentration in beets increases resulting in limesalts concentration in hard thin juice often exceeding 0.100g CaO/100ds. When limesalts concentration in filtered hard thin juice increases above this operational limit, the juice alkalinity must be increased by addition of an alkalizing agent to correct the alkalinity deficiency thus lowering the limesalts concentration in hard thin juice supplied to the WAC ion exchange cells.

Soda ash is generally used for the purpose of juice alkalization. Proper operation of 2nd carbonation and correct soda ash addition reduces the limesalts concentration in hard juice to below 0.100g CaO/100ds usually approximating that in thin juice produced from fresh beet. Good control of the re-alkalization process results in thin juice having sufficiently low limesalts concentration for supply to the WAC softening cells while also having stable pH and color behavior during juice concentration and sugar end operations. When so supplied, the WAC softening system routinely produces soft thin juice having a suitably low limesalts concentration.

Relatively precise control of 2nd carbonation alkalinity and the subsequent addition of alkalizing agent, especially during periods of fluctuating beet quality, is essential to avoiding both under and over alkalization of the juice. Under alkalization results in limesalts concentration exceeding the operational limit to the WAC cells. Sustained and/or repeated excess limesalts concentration in hard thin juice supplied to the WAC cells leads to resin overloading and a reduction in effective resin capacity along with difficulty in fully restoring resin capacity during routine regeneration cycles. Over-alkalization, in spite of yielding sufficiently low limesalts concentration to the WAC cells, leads to high juice pH and increased color rise during juice concentration.

Quality Requirements for Hard Thin Juice Supply to WAC Softening Cells

The juice supplied to the WAC softening cells must meet certain quality requirements in order to insure optimal performance of the softening system. Certain characteristics of the hard juice supply influence both the capacity and life of the resin as well as the hydraulic efficiency of the resin beds in the WAC cells. The following hard juice characteristics are essential to optimal operation of a WAC softening system.

 <u>The MAXIMUM juice hardness supplied to the WAC cells should not exceed 0.100g CaO/100ds</u>. Variation in limesalts concentration in hard thin juice is expected in normal factory operation. Due to such "normal" variation, the targeted average limesalts concentration in hard juice produced must be such that the limesalts concentration limit is never exceeded. For example, if the standard deviation of limesalts in the hard juice produced is +/- 0.020g CaO/100DS, the average limesalts concentration in hard juice supplied to the WAC cells should not exceed about 0.064g CaO/100ds.

- 2. Post precipitation of calcium carbonate in filtered hard juice must be avoided or at least minimized to the extent possible. Juice exiting the 2nd carbonation gassing vessel is normally supersaturated with calcium carbonate. The 2nd carbonation post reaction tank serves to desupersaturate the juice by fully precipitating the excess CaCO₃ in solution. Sufficient retention time and adequate agitation (or recirculation) are required to fully de-supersaturate the 2nd carbonation effluent. The post reaction tank is also an effective place for the final addition of soda ash for limesalts and juice pH control.
- 3. <u>The hard juice supplied to the WAC cells must be essentially sediment free</u>. It is essential that precipitated calcium carbonate is never allowed to pass to the WAC cells. If precipitated calcium carbonate accumulates in the WAC resin bed, the hydraulic capacity and softening efficiency of the resin bed is compromised due to channeling. The resin itself is also affected even to the point of irreversible fouling leading to premature resin replacement. It is essential that 2nd carbonation filtration be operated in such manner as to avoid even the occasional passage of calcium carbonate sediments. Double filtration is often practiced to avoid such excursions altogether.

Operation of Alkalization Pretreatment Process

When limesalts concentration in hard thin juice supplied to the WAC cells approaches the operational limit of 0.100g CaO/100ds, the alkali content of the juice must be increased to lower the concentration of soluble limesalts in the juice. While an alkalizing agent may be added anywhere in the beet end upstream of 2nd carbonation, it is most commonly added as soda ash (Na₂CO₃) either directly to 2nd carbonation or to the 2nd carb post reaction tank. The most precise control of juice alkalization may be achieved through a 2-step operation. The first step is the close control of the 2nd carbonation gassing at the "optimum" juice alkalinity in the absence of alkali addition. The second step is the relatively precise addition of soda ash to the 2nd carbonation post reaction tank to achieve an optimal alkali balance in the juice.

2nd Carbonation Operation

Second carbonation should be operated at or near the optimum alkalinity of the under-alkalized juice in process. This is essential to insure the lowest limesalts concentration in juice exiting the 2nd carbonation gassing vessel. It is quite important, relative to both lime salts control and proper overall juice alkalization, to maintain 2nd carbonation alkalinity near the "optimum" to enable minimum addition of alkali (soda ash) to achieve acceptable lime salts to the WAC units WITHOUT over-alkalizing the juice. Juice over-alkalization leads to pH increase during juice concentration, higher thick juice color and high pH values (>9.0 pH) in the sugar end.

It is relatively common in some areas of the USA for factories to process beets having relatively low natural alkalinity. Many of these factories do not normally employ any form of juice alkalization in the absence of juice softening. In such factories, 2nd carbonation is often operated at an alkalinity (pH) significantly higher than the optimum so as to provide a relatively higher thin juice pH to help support

evaporator and sugar end pH profiles. When WAC juice softening is installed in a factory where soda addition has not been used to control juice pH stability, it is often a bit uncomfortable for operators to accept the operation of 2nd carbonation at the lower optimum alkalinity until it is demonstrated that the appropriate addition of an alkalizing agent to the juice satisfactorily compensates for the lower target alkalinity (pH) in 2nd carbonation.

Soda Ash Addition and Control

Soda ash may be introduced to the process as an alkalizing agent in a number of different locations in the beet end. As noted earlier, an optimal point of addition is to the 2nd carbonation post reaction tank for, at least, a final "trimming" addition of soda ash to that vessel. This approach may assure minimum soda ash usage and the opportunity for relatively precise control of the overall alkalization process.

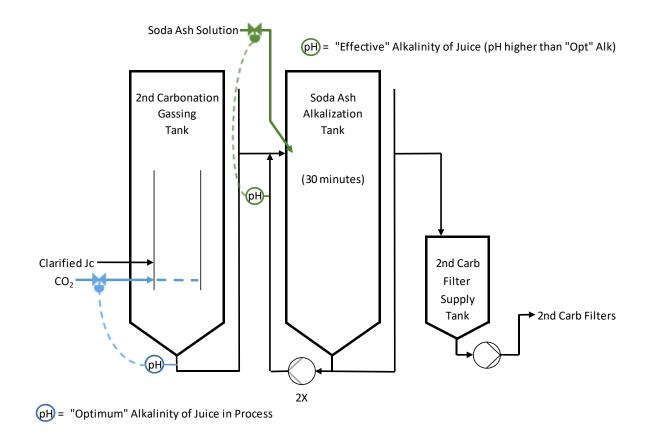
Soda ash may be introduced directly to the juice stream as a solid in granular form or as a solution of soda ash at a standardized concentration. Improved control of juice alkalization is usually achieved by dissolving granular soda ash in condensate or soft thin juice to yield a 10-20% solution of soda ash by weight. The stock soda ash solution is then metered to the process in a controlled manner via a simple control valve or VFD equipped treatment pump. As the limesalts concentration in the 2nd carbonation effluent varies, the concentration of the soda ash stock solution may be adjusted relative to the demand for soda ash solution flow in order to remain within the control range of the soda ash dosing system.

The most common alkalization control approach is to manage the flow of stock soda ash solution to the process based on the flow of juice through the system. Normally, the flow of juice through 2nd carbonation filtration is selected for the flow control input. The flow of soda ash stock solution to the process is managed through a juice flow/ratio controller. As process juice flow varies, the flow of soda ash solution varies relative to process juice flow as a function of a "manually selected" flow ratio. When there is a change in juice quality relative to limesalts concentration, this flow ratio must be manually adjusted to maintain the correct limesalts concentration/juice alkalization targets for the process. Such a control arrangement depends on the observation of a change in 2nd carbonation or hard juice effluent limesalts concentration followed by an adjustment of the soda ash flow ratio to correct any deviation in the targeted hard juice limesalts.

This system of control is satisfactory as long as the juice quality is relatively stable and only occasional adjustment of the flow ratio is necessary to maintain the targeted hard juice limesalts concentration. However, when large routine variations in juice quality occur, such as are encountered with longer beet storage or when beets are received from different areas, the "manual" flow ratio control of juice alkalization often results in a series of over and under alkalized conditions. This is primarily due to the lag in adjustment of the "manual" flow ratio input relative to the juice in process as a result of the inherent 1) lag in laboratory data output relative to process condition, 2) process retention time relative to process control point and 3) any delay and/or error in corrective process adjustment.

This control lag may be largely eliminated with the "two-step" alkalization process described below. The first step is the control of 2^{nd} carbonation at optimum alkalinity <u>without</u> soda ash addition FOLLOWED by the second step of soda ash addition to the juice exiting the 2^{nd} carbonation gassing tank. The process may be automated to control the addition of soda ash to the 2^{nd} carbonation post reaction tank.

This automatic control approach is relatively simple and depends upon operating the 2nd carbonation gassing at the optimum alkalinity without soda addition. In juice having higher organic acid content, the operation of 2nd carbonation at the optimum alkalinity in the absence of added soda ash will produce a juice having a relatively lower pH and the lowest limesalts concentration in the un-alkalized juice. Soda ash addition may then be controlled at a higher pH relative to the end point for optimally alkalized juice. The pH control set point generally approximates that of juice produced from freshly harvested beet. For such operating conditions and depending on the beet in process, the optimum pH of 2nd carbonation without soda ash addition is often in the range of 8.7-9.0 whereas the pH after correct soda addition is usually in the range of 9.2 to 9.5 pH. An example of this system of control is shown in Figure 1.



Double Alkalization Control of Total Juice Alkalization Figure 1

The following points describe the important aspects in the control of the alkalization process using the "Double Alkalization" approach.

 Double Alkalization gives acceptably consistent hard juice limesalts to the WAC cells while also providing optimal pH balance during juice concentration. Ideally, there should be no pH rise (due to juice overalkalization) in the juice evaporator. A slight drop in pH of about 0.5 pH during juice concentration is quite common and usually acceptable. When over-alkalization occurs due to excess soda ash addition, pH and color rise across the evaporator increase relative to the degree of excess alkali in the juice.

- 2. As the alkalinity of juice in process declines due to beet deterioration, the difference between the "optimum" alkalinity (pH) of 2nd Carbonation and the "effective" alkalinity (pH) of optimally alkalized juice generally increases relative to an increase in the organic acid content of the juice in process.
- 3. To be most effective, 2nd Carbonation gassing must be operated as close to the optimum alkalinity as possible without entering the bicarbonate range and in the absence of added or excess alkalizing agent allowing a subsequent final alkali addition to the juice to be managed at minimum total soda ash usage. The operator need only make occasional small trimming adjustments to the pH set point of the "Soda Ash Alkalization" tank to manage the WAC supply hardness and optimal thick juice pH.

Benefits of Optimal Juice Alkalization

While the control of limesalts concentration to the WAC softening cells is the primary goal of juice alkalization pretreatment, it is only one aspect of the result of correct juice alkalization. As beet quality deteriorates due to storage or any other adverse condition the organic acid content of the beet increases. The addition of an alkalizing agent to the juice produced must vary with respect to the resulting change in alkalinity of the juice related to the degree of beet deterioration.

When limesalts concentration in the hard juice produced from 2nd carbonation increases above 0.100g CaO/100ds or begins to vary significantly, one might conclude that a simple solution is to simply increase soda ash addition to a point where the hard thin juice never exceeds this limit. While such a practice may largely avoid the problem of hard juice limesalts exceeding the operational limit to WAC softening, it largely ignores the potential consequences to thick juice quality.

When thin juice is over-alkalized, there will be an associated pH and color increase across juice concentration relative to the degree of over-alkalization. The color increase in the juice is directly proportional to the increase in pH. The greater the degree of over-alkalization the larger the pH and color increase. Figure 2 shows the relationship between thick juice pH and color rise across juice concentration in one North American factory. Similar curves have been observed for a number of different factories indicating minimum color rise during juice concentration in the range of 8.5 +/-0.3 pH in thick juice.

The example in Figure 2 is during relatively "normal" variation in beet quality in a factory operating a 5effect Roberts evaporator. This factory must routinely vary the addition of soda ash to control lime salts to the WAC units. When juice alkalization is on target, hard thin juice limesalts concentration is well within operating limits to the WAC cells with thick juice pH in the range of 8.0-8.5 accompanied by the lowest color rise during juice concentration. During the period of lowest color rise thick juice averaged 8.1 pH with color rise of 620 IU. The hard juice limesalts during the same period averaged 0.075g CaO/100ds.

It must be added that, due to variation in beet quality during this same operating period, hard juice was under-alkalized 18% of the time. This resulted in WAC supply limesalts exceeding the operational limit of 0.100g CaO/100ds and thick often below 8.0 pH during such events. These under-alkalization events were related to sudden changes in beet quality and soda ash addition adjustment lag relative to juice quality. Similar adjustment lag was also noted when beet quality again improved and soda ash was not simultaneously reduced relative to the juice in process resulting in over-alkalization of the juice. Over-alkalization is noted as lower limesalts concentration in hard thin juice along with higher thick juice pH and color rise across evaporation. Such events may be significantly reduced or even eliminated by pH control of the addition of alkalizing agent as noted in the "double alkalization" control described above.

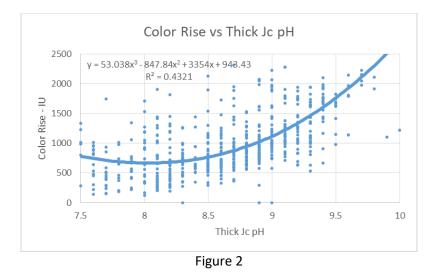


Figure 3 shows the difference in juice pH stability between a "flow based / manual ratio" control approach to soda ash addition and the "automatic pH control" of soda ash addition referred as double alkalization. During the period shown in Figure 3, hard juice limesalts control averaged 0.027g CaO/100ds for the entire period while the standard deviation in 2nd carbonation limesalts increased three-fold from the "manual control" period to the "pH auto control' period with virtually no change in soft juice limesalts. It is quite easy to see the improvement in the pH control stability in spite of increased variation in process juice quality.

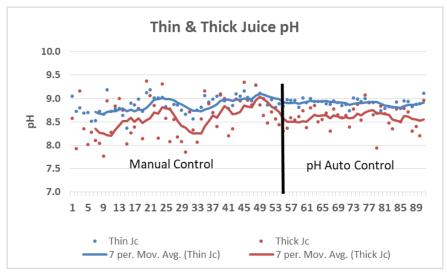




Figure 4 shows the 2nd carbonation, hard juice and soft juice limesalts concentrations for the same period shown in Figure 3. With pH control of soda ash addition, even with a much larger variation in 2nd carbonation limesalts concentration, the hard juice limesalts concentration did not vary significantly. This is due to 2nd carbonation being operated at optimum alkalinity and the soda ash addition to the juice in process automatically adjusted relative to the variation in the pH of the 2nd carbonation effluent. As a result, process adjustment lag is eliminated improving the overall control of the alkalization process.

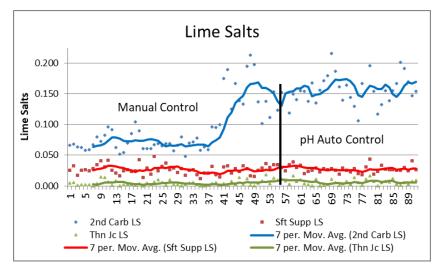


Figure 4

In short, optimal juice alkalization usually always results in sufficiently low limesalts concentration in hard juice for supply to the WAC softening cells and also in thick juice having ideal pH in roughly the range of 8.5+/- along with minimum color rise across juice concentration. The pH profile across the sugar end is also relatively stable exhibiting minimal pH drop from standard liquor to molasses relative to the beet in process.

WAC Softening Performance and Capacity Observations

Assuming that WAC service cycles and resin regeneration parameters are set within proscribed operating guidelines, certain operational performance characteristics may be observed. These characteristics are influenced by the operational environment to which the WAC system is subjected. However, observations of a number of installations in different operating environments have indicated that certain characteristics appear to manifest regardless of the operating environment.

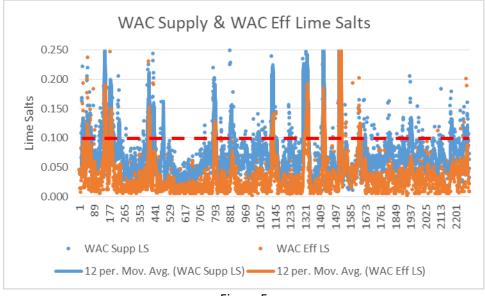
The first of these observations is related to the expected reduction in limesalts across the WAC cells. In general, the reduction is a function of the hard juice supply limesalts concentration. In theory, since a soft juice limesalts concentration of zero is the minimum, the reduction across the WAC cells is directly proportional to the hard juice supply limesalts concentration. Thus, a lower hard juice supply limesalts results in less reduction across the WAC cells and, potentially, a somewhat lower soft juice limesalts concentration. The actual reduction is influenced by a number of factors including:

- The degree to which hard juice limesalts is controlled within operating guidelines.
- The amount of Ca⁺⁺ "overloading" to which the resin is subjected.
- The degree to which the resin bed is subjected to fouling of the void volume causing channeling.
- The actual "working" capacity of the resin.

Each of these factors have an individual effect and are generally additive in their influence on WAC performance. All of these factors, with the possible exception of resin capacity which is influenced significantly by the other three factors, are controllable and significant with respect to system performance.

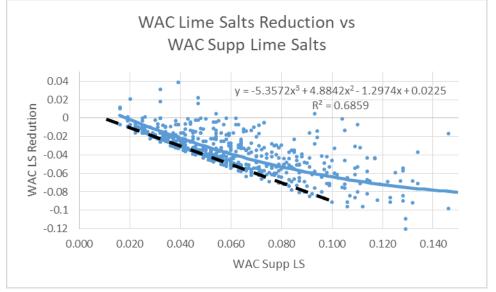
Figure 5 shows an example of the influence of hard juice limesalts on soft juice produced from the WAC cells. Virtually every change in hard juice limesalts concentration to the WAC cells, over the 72 operating days noted in the trend plot, resulted in a corresponding change in soft thin juice limesalts.

This is especially the case whenever the hard juice supply to the WAC units approached or exceeded the operational limesalts limit of 0.100g CaO/100ds.





The average degree of reduction in limesalts across the WAC cells can be determined by comparing the limesalts reduction to the hard juice limesalts supplied to the WAC cells. The relationship will vary from one installation to another depending on the degree to which the hard juice supplied is consistently within acceptable operational limits. Figure 6 shows this relationship for the same installation as that in Figure 5. The scatter in the data points is largely the result of resin overloading due to frequent and/or sustained excursions in hard juice supply limesalts above 0.100g CaO/100ds.



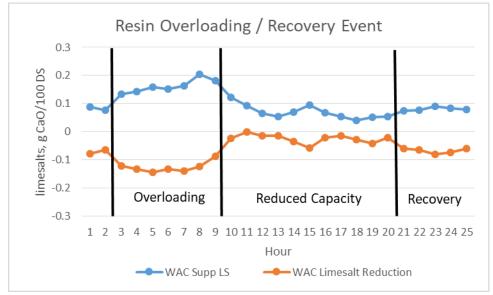


This relationship is quite important in setting a suitable target hard juice limesalts concentration to achieve the desired concentration of limesalts in soft thin juice. For example, from the illustration in Figure 6, WAC juice supply limesalts at 0.060g CaO/100ds gives an average reduction (blue trend line) of

approximately 0.038 limesalts across the WAC units yielding soft juice having 0.022 limesalts: the approximate targeted soft juice limesalts concentration for this installation.

It is worth noting the "theoretical" operation that is shown by the minimum data points (black dashed line) on the scatter diagram in Figure 6. This is the best possible performance that may be expected from the system operating with little deviation from optimal performance. If such optimal performance could be routinely achieved, then any hard juice limesalts concentration below 0.100g CaO/100ds would produce a soft thin juice having approximately 0.010g CaO/100ds. Of course, it is not likely that such optimal performance always occurs in actual practice. It is also important to note that the concentration of data points on the "optimal trend" increases as the concentration of hard juice limesalts decreases. The degree of "scatter" in the operation depends on the degree of "scatter" in the WAC supply hardness along with the degree to which hard juice supply limesalts exceeds the operational limit of 0.100g CaO/100ds.

Whenever the WAC supply limesalts concentration exceeds the operational limit, the capacity of the resin is at least temporarily affected. Figure 7 shows what generally occurs during and after a typical excursion in WAC supply limesalts.





During the "Overloading" phase, the resin removes an increased amount of Ca⁺⁺ (reduction across WAC cells exceeding 0.100g CaO/100ds) until it is effectively fully loaded. At that point the resin is essentially unable to remove any more Ca⁺⁺ from the juice and enters a "Reduced Capacity" phase. During this phase the reduction in limesalts across the WAC cells is reduced to near zero (may actually give up Ca⁺⁺ to the WAC effluent juice). This condition is largely responsible for the significant "scatter" in the data points shown in Figure 6. When the hard juice limesalts is once again managed below the operational limit, the resin then enters a "recovery" phase and eventually regains its operating capacity over the course of multiple regeneration cycles. In this example, the WAC system required approximately 12 hours to return to acceptable operational capacity after the excursion above the operational limit of 0.100g CaO/100ds hard juice limesalts. This is a bit longer than the length of time required to complete one regeneration of <u>all</u> WAC cells in this WAC installation.

To achieve design performance of the WAC softening system, overloading of the resin due to hard juice supply limesalts exceeding the operational limit must be avoided. Such operational control integrity is

necessary to insure that the resin capacity can achieve stable and maximum limesalts reduction across the WAC cells.

Resin capacity analysis more or less confirms the influence of frequent resin overloading. Table 1 shows the resin capacity analysis for several WAC systems currently in operation. Factory E sampled the resin just after a series of relatively significant overloading events.

	Resin Capacity				
	New	Half Camp	Full Camp	Fctry Regen	Design
Factory A	4.3	4.1	3.9		1.5
Factory B	4.3	3.8	3.7		1.5
Factory C	4.3	3.7	3.6		1.5
Factory D	4.3	3.7	3.2		1.5
Factory E	4.3	3.2 (30 days)		2.6 (30 days)	1.5

Table 1

The analysis of the Factory E "Fctry Regen" sample is only 40% of the capacity of new resin and also only about 81% of the capacity of the HCl pretreated portion of the same resin sample. The "Fctry Regen" sample was not subjected to the usual "acid stripping" before capacity measurement. Even so, the acid stripped resin sample is also lower than the Factory A-D samples which were taken after approximately 2 months of operation whereas the Factory E sample was taken after only one month of operation during which Factory E frequently operated with hard juice supply to the WAC cells exceeding the operational limit of 0.100g CaO/100ds. The comparatively lower capacity for the Factory E resin may suggest increased fouling of the resin due to the operating conditions to which it was subjected.

Operating Targets for Juice Alkalization

The primary goal of juice alkalization, with respect to the operation of WAC juice softening, is the control of the limesalts concentration in the hard juice supply to the WAC cells. As noted, the normal variation in 2nd carbonation effluent limesalts and the resulting variation in hard juice limesalts related to the control of alkalizing agent dosage must be taken into consideration in setting the average hard juice limesalts target. Depending on the normal variation of processing variables, the targeted average limesalts concentration must be adjusted so there are NO excursions in limesaltss above 0.100g CaO/100ds. Such operating performance may be difficult to attain if there are large fluctuations in 2nd carbonation limesalts concentration in the absence of fully automatic control of soda ash addition. If limesalts excursions exceeding the design concentration limit occur, the capacity of the WAC resin will be affected as well as the reliability of the limesalts concentration of the WAC effluent soft juice.

The degree to which the process is directly managed with online process measurement has a critical influence on the stability of the process. One possible method has been described herein and has shown positive results in its limited deployment where large variation in beet quality and 2nd carbonation effluent limesalts were experienced. One extremely important aspect of the performance of the WAC softening system is to minimize the variation in hard juice limesalts concentration and to avoid any excursion in limesalts concentration above the operational limit of hard juice supply to the WAC cells.

Many system operators seem to arrive at a hard juice limesalts target that is significantly below the operational limit. The hard juice limesalts target often depends on the variation in hard juice limesalts experienced in routine operation and the soft juice limesalts targeted by that operation. Table 2 gives approximate general guidelines followed by many operations in the USA.

Limesalts Targets: g CaO/100ds				
Hard Jc	Soft Jc			
0.030 - 0.040	0.000 - 0.005			
0.045 - 0.055	0.008 - 0.015			
0.060 - 0.070	0.020 - 0.025			

Table 2

A secondary, but no less important consideration in establishing juice alkalization targets are the pH and color of thick juice and pH stability in the sugar end. Ideally, as shown in Figure 2, a thick juice pH should be targeted relative to a minimum color rise in juice concentration. The actual color rise noted at optimal thick juice pH will be influenced by the type of evaporator employed and the quality of the beets in process. Under most conditions a final thick juice pH in the range of 8.5+/-0.03 accompanied by a slight pH drop across the evaporator in the range of 0.02-0.05 pH is usually associated with the lowest color rise.

If the juice is alkalized with soda ash to yield the optimum thick juice pH and there is no additional juice alkalization (caustic addition) downstream of the WAC softening cell, the average limesalts in hard thin juice will likely be in the range of 0.035 to 0.060g CaO/100ds and fully suitable for supply to the WAC cells. WAC effluent soft juice will commonly be in the range of 0.010+/- 0.002g CaO/100ds or less if resin overloading is avoided. In cases where a higher soft juice limesalts concentration is desired, a small amount of hard thin juice may be bypassed to achieve the desired limesalts concentration in soft thin juice.

Unless the juice is under-alkalized with soda ash prior to entering the WAC cells, it should not be necessary to add additional alkali after the WAC units to achieve ideal conditions for juice concentration and sugar end pH control. However, under certain conditions where there is very low natural alkalinity in beets processed or where a higher limesalts concentration in hard thin juice is desired, there may be a post WAC alkali adjustment requirement. This may especially be the case if the control of soda ash addition is less than ideal or not automatically controlled as described earlier and the juice is being deliberately under alkalized in the 2nd carbonation operation.

In Summary

To summarize the influence of thin juice alkalization on WAC thin juice softening performance and juice quality, the following points are emphasized:

- 1. Juice alkalization must be managed to insure that the hard juice supply to WAC cells does not exceed 0.100g CaO/100ds. Both over and under alkalization of juice is to be avoided.
- A 2-step pH based control of the alkalization process provides stable on-line control of hard juice limesalts and thick juice pH. Such control is especially important where large excursions in juice quality (2nd Carb limesalts) are experienced.
- 3. If hard juice exceeds the operational limit of 0.100g CaO/100ds, resin capacity is temporarily reduced resulting in increased soft juice limesalts and reduced resin capacity. Resin capacity is generally not restored until regeneration of all cells in the system is completed.
- 4. Juice alkalization management targeting optimal thick juice pH and color behavior during juice concentration generally results in limesalts concentration within acceptable limits in the hard juice supplied to WAC softening cells.