Effect of Solids Recirculation On Purification of Raw Juices

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Introduction

Purification of raw juice extracted from beet cossettes is universally performed by means of lime and carbonic acid treatment in various steps. A large variety of processes are employed, all using the same chemicals for eliminating as many impurities as possible.

These notes deal with a particular aspect of the juice purification process, namely, with the recycle of calcium carbonate particles and the various effects obtained. It is beyond the limits of this paper to examine solids recycle from a strictly chemical point of view, although a thorough study in this direction is highly recommended. Existing trends in Europe, as far as solids recycle is concerned, are briefly reviewed with special regard to those with which the writer has had direct experience or has been able to obtain firsthand information. Recycle of clean solids, as successfully practiced in some Italian factories, is briefly described and some qualitative results are given.

Historical Background

The Dorr System of continuous first and second carbonation includes, in the broad use of the term, the steps of liming, gassing, mud thickening and filtering prior to evaporation. This system is the heart of all modern juice purification processes, all of which include some recirculation of carbonated juice within the saturation step. This recirculation is necessary in order to facilitate filtering of the carbonated juice and sweetening off of the cake on continuous rotary filters. Batch carbonation, it is well known, produces saturated juices that are very difficult to thicken and/or filter with any type of equipment. A typical flowsheet of the Dorr Continuous Carbonation System is shown in Figure 1.

The basic Dorr Carbonation System, first practiced commercially about 1928, has now undergone numerous modifications. One such modification is shown in Figure 2. In this system, in order to adapt it to a particular purification need, continuous preliming and separate main liming were included. This system is often practiced in some European countries. Other modifications of the basic system are being used as will be covered later.

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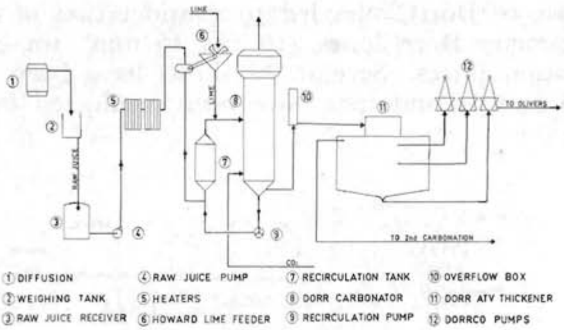

Figure 1.—A typical flowsheet of the Dorr Continuous Carbonation System.

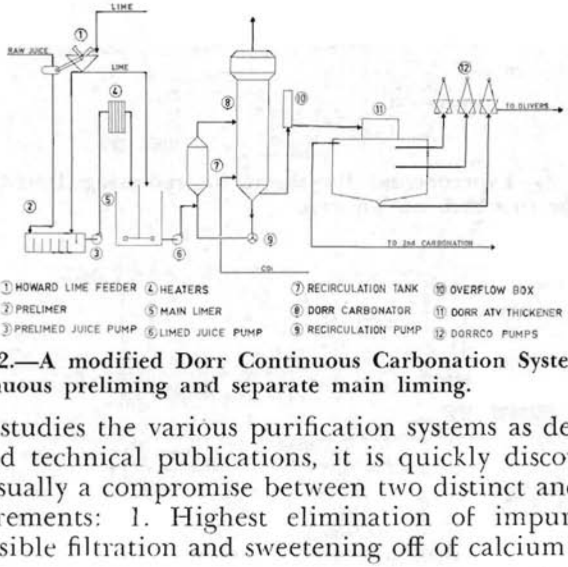

Figure 2.—A modified Dorr Continuous Carbonation System incorporating continuous preliming and separate main liming.

If one studies the various purification systems as described in patents and technical publications, it is quickly discovered that they are usually a compromise between two distinct and contrasting requirements: 1. Highest elimination of impurities and; 2. best possible filtration and sweetening off of calcium carbonate muds.

It is interesting to note that in order to cope with both requirements, some solids recirculation is considered necessary in all of the flowsheets. This very simple consideration led the writer to the investigation of a modification of the basic system utilizing recycle of clean calcium carbonate particles.

Early DorrClone Tests in Europe On First and Second Carbonation Juices

Porcelain DorrClones[2] of 50- and 100- mm diameter are widely used in European factories for degritting milk of lime. Because they make separations on grit in the range 20 to 30 microns, they are much more effective than conventional machines such as rotary and vibrating screens.

[2] Trademark for hydrocyclone manufactured by Dorr-Oliver Companies.

This use of DorrClones led to consideration of the use of smaller diameter DorrClones (10 and 15 mm) for clarification of carbonation juices. Several flowsheets have been considered (Figures 3 and 4) and tests have been conducted in Germany and Italy.

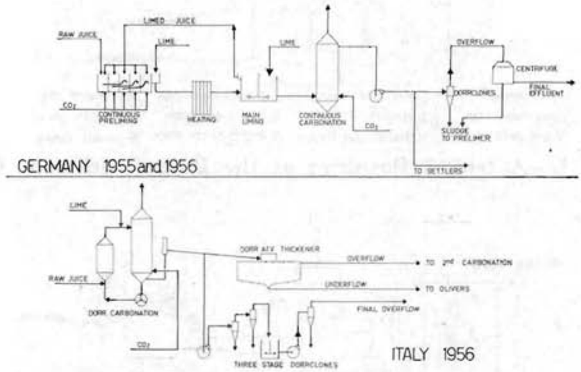

Figure 3.—Experimental flowsheets incorporating Dorr-Clone clarification in the first carbonation step.

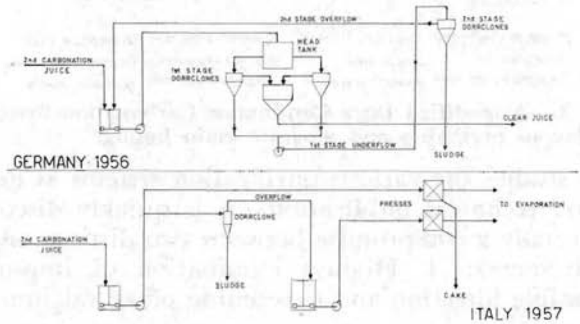

Figure 4.—Experimental flowsheets incorporating Dorr-Clone clarification in second carbonation step.

On first carbonation juice it was found relatively easy to obtain high solids removals at high concentrations but overflow clarities comparable to thickener overflows were never achieved. The cloudiness of the DorrClone overflows was mainly due to colloidal particles. Attempts to polish DorrClone overflow with disc centrifuges were not successful because of the inability of the centrifuge to make adequate separations at reasonable capacities. With polishing filters, low filtration rates and cloth blinding were encountered.

Better results were obtained when DorrClones were tested on second carbonation juices. Although the clarification was not

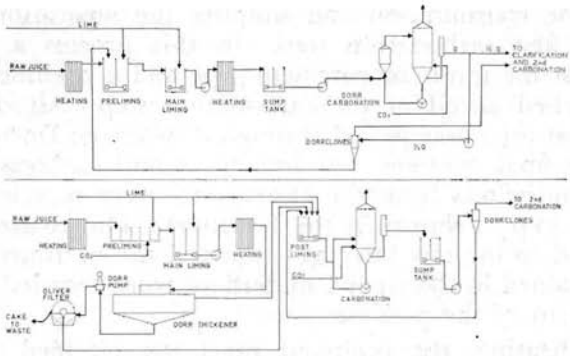

Figure 5.—First carbonation flowsheets currently in use in Europe incorporating DorrClones to recycle selective fractions of the carbonate mud.

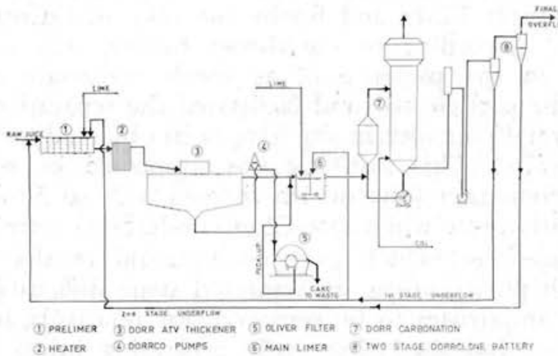

Figure 6.—Experimental flowsheet incorporating two-stage DorrClone separation of first carbonation juice and recirculation of solids to raw juice and prelimer.

complete, the length of the cycles on the second carbonation pressure filters was increased. Results from the Italian experiences indicate that the capacity of the plate-and-frame filters increased from two to five times when they were fed with the Dorr-Clone overflow. DorrClone underflow is sent back to the raw juice tank.

Although DorrClones were not satisfactory for clarification, they do perform a useful function when used in various ways within the carbonation process. They enable selective fractions of the carbonate mud to be recycled so as to improve mud settling and filtering characteristics. For example, Figure 5 shows some flowsheets which are being used in Europe and other flowsheets to be shown later also embody this use of DorrClones.

Research Work In Belgium On Recirculation of Solids

An interesting flowsheet (Figure 6) has been tested in Belgium, at the suggestion of A. Schaus[3]. The objectives are to

[3] Chief Chemist Dorr-Oliver S.A., Brussels.

reduce lime consumption and simplify the separation of solids from the first carbonation step. In this process a horizontal prelimer of the multicompartment type and a prelime thickener were installed ahead of the carbonation step. All of the first carbonation juice was passed through a two-stage DorrClone battery. The final overflow was sent to second carbonation while the two underflows from the DorrClones were recycled into the preliming step as shown in the flowsheet. The coarser particles were added to the raw juice as it entered the prelimer while the fines, contained in the second underflow, were recycled to the last compartment of the prelimer.

After heating, the prelimed juice was clarified in a Dorr Thickener. This overflow was then heated, limed, and carbonated. The underflow of the prelime thickener was sweetened off on an Oliver Filter and finally the cake was discarded from the system. According to the theory behind this, carbonation conducted in the presence of as much carbonate as possible increased the particle size and facilitated the separation of solids. Particles over 40 microns in size have been observed and measured microscopically. This thinking was supported by several tests on first carbonation juice which showed a 3- to 5-fold increase of the filtration rate when DorrClone underflows were recycled.

This flowsheet, which gave encouraging results when processing high purity juices, encountered some difficulty when the amount of impurities to be removed was relatively high. Tests in Italy have indicated that while processing juices of, say, 82 to 84 purity the overflow from the DorrClone was still cloudy, even when a 3-stage arrangement was used. It was found that about 80% of this turbidity was due to organic impurities in colloidal form. These organics could not be removed by any simple polishing filtration and would prove detrimental if conveyed to the second carbonation vessel.

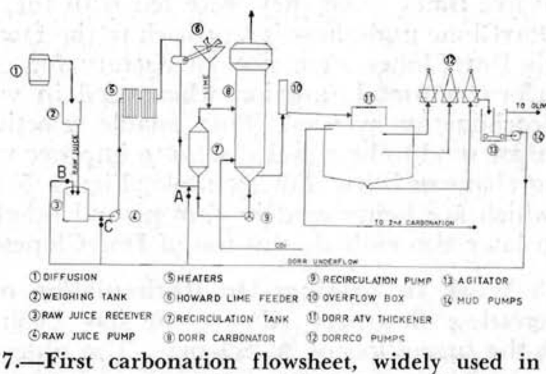

Figure 7.—First carbonation flowsheet, widely used in England and Italy, incorporating recycle of Dorr Thickener underflow.

Recirculation of Thickener Underflow Ahead of Purification Station as Practiced in Several European Factories

Recycle of Dorr Thickener underflow has been used for years in Europe, principally in England and Italy where it has become a standard practice in most plants. Although results obtained in various factories are not strictly comparable, it has been established that recycling of Dorr Thickener underflow is effective in reducing the color formation within the thickener and in improving settling and filtration.

A typical flowsheet utilizing this recycle is shown in Figure 7. Tests have been made in which the Dorr Thickener underflow was returned to three different points ahead of the carbonator. These were:

Point A—into raw juice pipe just before it entered the recirculation tube.

Point B—into the raw juice tank where the diffusion juice flowed from the weighing tanks.

Point C—into the suction line of the supply pump.

Recycling to Point A did not show very good results except for color reduction, whereas other positive advantages were obtained when mud was recycled in Points B and C, although no significant differences were noted between these two.

The advantages obtained may be summarized as follows: 1. Color reduction of thickener overflow ranging from 25% to 30%; 2. improved settling with higher settling rates and smaller mud volumes thus reducing the actual unit area requirement to 3.9 sq ft/short ton solids/day which is equivalent to 0.24 sq ft/short ton beets/day under the conditions of the plant where the tests were made in Italy and; 3. improved filtration due to porous filter cake. The solids handling capacity of the Oliver Filters was increased by 1/3 as shown in the following figures developed at the Italian installation.

Capacity	Without Mud Recycling	With Mud Recycling
Sq ft/short ton beets/day	0.171	0.113
Sq ft/short ton solids/day	4.40	1.88

Although the overflow turbidity increased with mud recycling from about 50 to 150 ppm the polishing filtration ahead of second carbonation was greatly enhanced. Filtration cycles of 75 to 80 hours were experienced with porous ceramic candle filters while, without mud recycling, the cycles were not more than 10 to 20 hours.

Another minor but consistent advantage was the reduction of foam in the raw juice tank where the Dorr underflow was continuously added.

Certainly, when filtration on rotary drum filters is a serious problem the recycle of Dorr underflow is a simple and inexpensive solution without increasing the total lime consumption. In some cases an increase in lime salts has been experienced compared to the conventional Dorr-Oliver scheme, especially when the first carbonation (phenolpthalein) alkalinity is relatively high (over 0.10 grams Ca0/100 cubic centimeters).

A curious phenomenon which has been experienced is the poisoning of the calcium carbonate being recycled after a time ranging from 1 to 3 weeks. The color reduction decreases progressively and the slurry becomes darker and darker. The recirculation of Dorr underflow must then be interrupted for two or three shifts in order to purge the system completely.

At this point a question might be raised as to how much Dorr underflow should be recycled in order to obtain the best results. Any figure concerning the volumetric percentage of the recycled underflow would be misleading if not supported by other data, such as sludge and juice density. As it is rather difficult to measure and control continuously the volume or weight of Dorr underflow in commercial installations, it seems advisable to express the recycle in terms of equivalent grams of Ca0 recycle into raw juice before any further treatment. Our experience in Europe indicates that the optimum is in the range of 0.7 to 1.0 grams of Ca0 per 100 cubic centimeters of juice, depending on local conditions. Higher alkalinities in first carbonation, thicker raw juices, and lower purity juices demand a higher percentage. A maximum value of 1.2% Ca0 has been found necessary in a plant where extremely rich beets are processed (percent sugar in the cossettes over 22%).

All the above considerations apply also when disc type pressure filters are used as intermittent thickeners.

Pilot Plant Work in the United States With Preliming and Solids Recirculation

A few years ago, extensive test work was carried out in a pilot plant erected by Dorr-Oliver at Betteravia, California, with the cooperation of the Union Sugar Division.

The beet juice purification process tested in California is shown in Figure 8. The following processing steps were used: 1. Stabilization of the slightly acid raw juices by massive recirculation of carbonation thickener underflow; 2. progressive preliming with milk of lime and partial recirculation of prelime juice; 3. separation of coagulated impurities (nonsugars) by sedimentation; 4. addition of carbnate solids to the clear juice; 5. mainliming, carbonation, and thickening and; 6. sweetening off of carbonate cake.

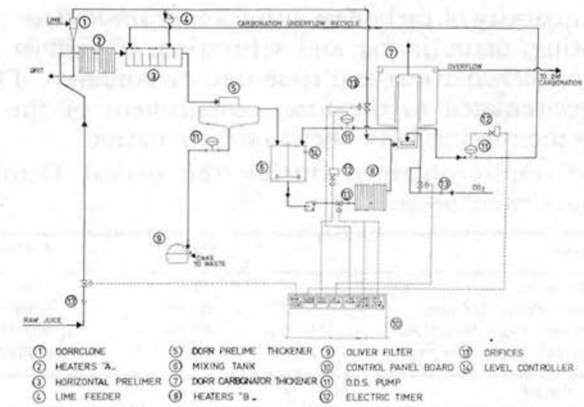

Figure 8.—Beet juice purification pilot plant used in cooperative test program at the Union Sugar Company's factory at Betteravia, California.

According to several authors (Dedek, Vasatko, Teatini, Brieghel-Müller, Salani and others) an improved purification can be accomplished with progressive preliming because the impurities (nonsugars) present in the raw juice will coagulate, flocculate, and segregate at different pH values. It would seem desirable to remove these segregated impurities as soon as they precipitate and before any further treatment. In this experimental process, therefore, a prelime thickener was used for separating the coagulated impurities. A massive recirculation of carbonation sludge was needed for this purpose. Otherwise these colloidal solids would not settle satisfactorily. The prelime thickener overflow was then treated according to standard practice,, i.e., it was limed and saturated with CO_2. In order to obtain good settling rates in the carbonation thickener, carbonation sludge was added before main-liming and carbonation to maintain a controlled concentration. This recirculation promoted growth of large crystals.

In normal operation, Heater A (Figure 8) was not used. Juice coming from the diffuser was fed into the first compartment of the Brieghel-Müller prelimer at about 55-60°C. Milk of lime was added in the next-to-last compartment at about 0.25-0.35% Ca0 on juice. A large paddle agitator and the surface baffles provided a certain backward recirculation of prelimed juice from each compartment to the preceding one, thus achieving progressive preliming. Practically all of the carbonation thickener underflow was added into the first compartment of the prelimer.

The prelimed juice was then fed to the prelime thickener. The underflow from the thickener was sweetened off on an Oliver filter, while the overflow was sent to a mixing tank where a controlled quantity of carbonation thickener underflow was added before heating, main-liming and saturation. Overflow from the carbonation thickener was sent to second carbonation. The underflow was recirculated to the first compartment of the prelimer and also to the mixing tank ahead of carbonation.

Average results obtained during the period October 1-24, 1958, are tabulated below:

Juice	Purity[1]	Color[2]
Diffusion	82.15	—
Pilot Plant Effluent	88.07	0.2030
Factory Dorr Overflow	87.78	0.2642
Factory Kelly Filtrate	87.79	0.2327

[1] Apparent Purity
[2] Optical Density at 420 Angstrom. — These values times 1000 equal Spekker degrees-the color classification used in British factories.

Operating conditions were as follows: Flow Rate—75 U.S. gallon/minute; Preliming-0.25% CaO on beets, 55-60°C; Carbonation—1.75% CaO on beets, 70°C.

During the period October 29 to November 11, 1958, the temperature after the preliming was raised to 70°C, obtaining the following results:

Juice	Purity[1]	Color[2]
Diffusion	82-15	—
Pilot Plant Effluent	87.01	0.2350
Factory Dorr Overflow	86.36	0.2540
Factory Kelly Filtrate	86.28	0.2210

[1] Apparent Purity
[2] Optical Density at 420 Angstrom. — These values times 1000 equal Spekker degrees—the color classification used in British factories.

Under both operating conditions mentioned, unit areas in the thickeners were as follows: Prelime Thickener—0.309 sq Ft/short ton beets/day; Carbonation Thickener—0.215 sq Ft/short ton beets/day.

Statistical analysts of the date tabulated above indicate a high level of confidence.

In summary, it can be said that this pilot plant achieved a 0.3 point purity increase over the Factory Dorr overflow, with a color reduction of 30% and a lime saving of 20%. By heating prelimed juice, the lime saving was maintained and a purity rise of 0.7 point was achieved but the color did not improve. The tests therefore demonstrate the improvements which can be obtained with preliming and removal of prelime solids. Although a thickener was used for removal of prelime solids, other removal devices, such as a Webtrol belt filter might also be used.

Present Recirculation Practice With Dorrclones in Italy

From all of the experiences briefly summarized up to this point, as well as from a survey of the patents and of the various processes which have been described from time to time in the technical press, the basic requirements for improving the filterability of carbonation juices can be said to consist of:

1. The presence of calcium carbonate particles in the raw juice before it is submitted to treatment by heating or liming. Organics that are coagulated only by the action of heat and lime do not form aggregates with the calcium carbonate which is precipitated later in the gassing step. The failure to form such aggregates renders the subsequent clarification operations more difficult regardless of whether this clarification step consists of settling followed by vacuum filtration or by direct filtration with either pressure or vacuum filters.

2. The calcium carbonate particles added to the raw juice should be as clean, as hard, and as large as possible. The addition to the raw juice of calcium carbonate, from which the coagulated organics and fine particles have not been separated, does not produce optimum results.

Other considerations that contributed to the process described in this section are:

1. Only a small part of the total lime usually added to the juice is necessary for reacting with those nonsugars which can be precipitated by liming and carbonating. The balance is needed only for creating solid nuclei within the liquor to be clarified.

For instance, it can be said that 1% Ca0 on beets, from a strictly chemical point of view, would be just as effective as 2%, all other conditions remaining the same.

2. First carbonation cake as discharged from Oliver drum filters is an excellent source of calcium carbonate which has been formed in the liquid being treated—the sugar solution.

Being inexpensive, and a waste product unless employed for soil conditioning purposes, it is logical to make use of it.

The addition of finely ground limestone to raw juice has been tried but without satisfactory results, to the best of our knowledge, for reasons that are beyond the scope of these notes.

It has also been suggested that calcium carbonate for use in raw juice might be obtained by washing and classifying carbonation cake in a hydroseparator, but this has not been tested[3].

3. Porcelain DorrClones were extremely effective in thickening first carbonation juice but their overflow was always slightly turbid on account of the suspended fine solids.

[3] Suggested by R. C. Campbell, retired sugar technologist of Dorr-Oliver Inc., Stamford, Conn.

Since 80% of these fines were colloids, it was reasonable to take advantage of this apparently negative result for removing coagulated impurities from a slurry of carbonation cake. DorrClones were expected in this use to overflow the finest solid particles so that the larger particle sizes could be recovered in the underflow. The intense shearing action which occurs within the cylindriconical DorrClone body was expected to separate some organics not fully amalgamated with the particles but adhering to their surfaces.

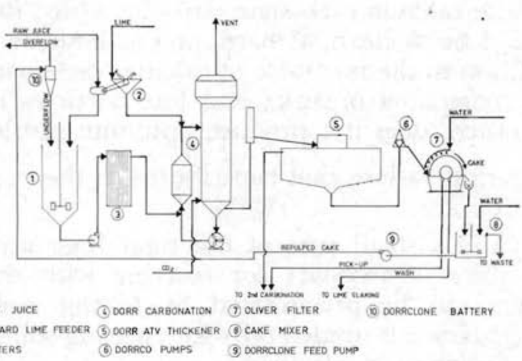

Figure 9.—First carbonation flowsheet incorporating recycle of filter cake after washing and classifying in DorrClones.

As a result of all the above considerations, the simple flowsheet shown on Figure 9 was developed[4]. It includes repulping first carbonation filter cake with clean water, pumping the resulting slurry through a battery of DorrClones which classifies out and discards the organic impurities and the finest calcium carbonate particles, and yields a clean calcium carbonate in the underflow. This product is returned to the raw juice prior to any heating or liming treatment. Among the several purification processes using DorrClones for facilitating the handling of first carbonation juice, this one has two unique features: 1. Washing of the first carbonation cake by repulping in water and; 2. classifying the suspended solids and discarding the fines which are the most detrimental, chemically and physically.

The addition of a slurry containing clean and classified calcium carbonate particles to the raw juice produces results which, according to the available information, include:

1. Stabilization of the raw juice. Its pH is increased from a value below 7 to 8 - 8.5 by the alkaline slurry which still contains a little active lime. This stabilization enables the

[4] Patents applied for.

juice to be heated ahead of liming without fear of inversion or of coagulation of nonsugars, such as occurs when raw juice, free of lime, is heated.

This is particularly important with raw juice from certain types of continuous diffusers which tend to increase the extraction of organic impurities that are usually very difficult to remove without a large excess of lime.

2. Foam in the raw juice tank is easily controlled by adding the recycled carbonate slurry as a spray. The addition of conventional defoaming chemicals is unnecessary except in emergencies.

3. The lime consumption is consistently reduced because it is possible to build up a stock of calcium carbonate within the system of any desired concentration. The solids handling capacity of the equipment is the limiting factor. A maximum reduction of 40% in the lime consumption was obtained in a factory operating in accordance with the flowsheet shown on Figure 10A.

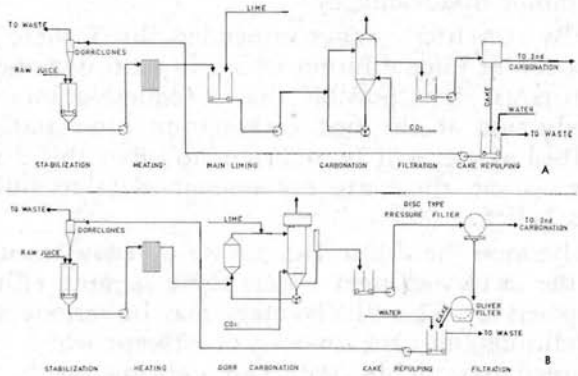

Figure 10.—Flowsheet A resulted in recovery of 50 tons of sugar in a 60-day campaign with a 40% reduction in total lime consumption. Same factory later adopted flowsheet B, with comparable results.

It can be said that the governing factor for the clarification of carbonation juice is the weight ratio of the calcium carbonate solids to the nonsugars, all other conditions, such as temperature concentration, viscosity, alkalinity, and detention times, being held constant. The recirculation of clean and classified calcium carbonate permits a reduction in the fresh lime addition while maintaining, or even increasing, this weight ratio.

4. Sweetening off of the carbonate cake is greatly improved as it contains mostly large particles. The fines are restricted to those originating from the carbonation of the reduced addition of fresh lime.

The carbonate particles are continuously washed, classified, and recirculated within the system, allowing the nascent calcium carbonate to grow on large size nuclei of 15 to 35 microns. The filter cake is thus unusually porous and requires less water for sweetening off.

Wash water dilution of the juice amounting to 6% on beets was sufficient to reduce sugar losses on press cake to about 0.6 to 0.7% whereas before the installation of the process, the dilution was 12% on beets and still left about 1% sugar on cake at 50% moisture. Press operators agree that the cake is extremely porous and not sticky, so that it drops easily, on opening the presses, without any manual help.

On Oliver drum filters the dilution by wash water required to reduce the sugar loss to 0.4% on wet cake is about 4% or less on beets.

Like everything born of the human mind, this system also has some minor disadvantages:

1. By recycling a water-suspended slurry there is a small increase in juice dilution which in turn increases evaporation costs. It is possible that a reduction in sweet water production at the first carbonation filter station, as described above, will be sufficient to offset this disadvantage but, as yet, there are not enough data to fully support this belief.

2. Because the dilute DorrClone overflow must be added to the factory effluent waters there is more effluent to be disposed of. This disadvantage may be serious where local conditions limit the amount of effluent which can be discharged into public waters or existing ponds. It should be remembered, however, that the total amount of waste solids has been reduced, as compared to conventional processes, because of the reduction in the fresh lime consumption.

No significant change for better or worse has been experienced to date in juice purity or color.

Although it does have a limited amount of chemical activity, the carbonate recycled is not essentially a chemical reagent. Consequently, this recirculation scheme, although conceived as a logical improvement to the standard Dorr Carbonation Process, can be adapted to any juice purification process without losing the unique characteristics of that process.

The recirculation of cleaned and classified calcium carbonate from the DorrClones improves settling rates as well as filtration rates on thickened sludge. Bulk settling rates ranging from 18-23 feet/hour, or double the rate found without recirculation, were obtained in most of the many tests made. After 30 minutes detention, the sludge volumes never exceeded 20% of the unsettled juice volumes.

The settling and filtration rates obtained in commercial installations were in agreement with the laboratory tests and checked the values obtained with other systems of solids recirculation described earlier. Consequently in new plants equipment sizes can be reduced and, in existing plants, a higher factor of safety is obtained or plant capacity may be increased. The reduction in fresh lime consumption extends these advantages to the lime kiln, slaking, and degritting stations and the gas pumps.

No detailed figures are given with regard to economics since the unit costs of lime, fuel, beets, and sugar vary from one country to another and, even in the same country, from one factory to another. A factory slicing 1200 tons beets per day operating according to the flowsheet shown on Figure 10A recovered about 50 tons of sugar in a 60-day campaign, because of reduced filter cake losses and in addition, other savings resulting from a 40% reduction in lime consumption.

After two campaigns, this same factory adopted the flowsheet shown on Figure 10B, using leaf-type pressure filters from which the cake is sweetened off on Olivers, and maintained the same advantages.

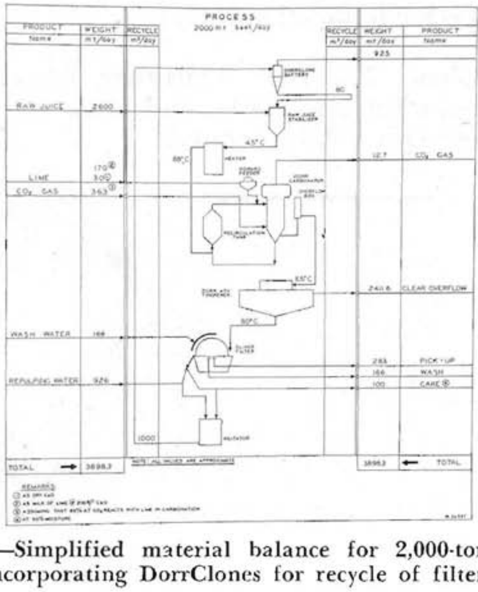

Figure 11.—Simplified material balance for 2,000-ton per day beet sugar factory incorporating DorrClones for recycle of filter cake.

A simplified material balance for a factory with a capacity of 2,000 tons of beets per day is detailed on Figure 11 from which economic calculations can be made using existing unit costs for any particular case along with the following factors: 1. Reduced lime consumption, with a maximum of 40%; 2. reduced cake losses to about 0.4% on cake at 50% moisture; 3. reduced wash water dilution to about 4% on beets; 4. increased load on the evaporators of about 2% and; 5. increased amount of waste effluent depending on present practice.

Installation and operating costs must, of course, be calculated for each individual case.

Summary

To summarize, a number of modifications of the basic Dorr Carbonation System have been reviewed. All include a scheme of solids recirculation. Several systems using preliming in Brieghel-Müller type prelimers together with solids recirculation showed possible advantages in improved color, settling, and filter rates and a reduction in lime consumption.

Through the use of DorrClones in a novel flowsheet developed in Italy, cleaned and sized calcium carbonate particles are produced from filter cake and recycled to raw juice prior to heating or liming. Results from installations show substantial advantages.

A material balance is presented which permits economic calculations for specific installations.

NOTE: DorrClone, The Dorr Thickener, Oliver, and Dorrco are registered trademarks, and Webtrol is a trademark of Dorr Oliver Incorporated.