Comments on Use of Ion-Exchange Resins in Demineralizing Sugar Beet Juices[1]

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THE PARTICULAR points to which your attention will be called are 1. the effect of the concentration of minerals in the beets on the cost of ion-exchange operation and 2. the effect of the composition of the sugar beet minerals on the amount of the additional sugar recovered.

We have made extensive studies of the final molasses from nearly all of the non-Steffen factories of the domestic industry. Taking into account the molasses produced, calculated as percentage on weight of beets, it is evident that the composite beet processed at some factories contains double the concentration of mineral ions that occur in the beets at other locations. At a particular factory the differences between beets from various fields serving the factory may show equal or even greater variation in mineral content. This latter fact is not so important from the viewpoint of the use of ion exchange for demineralization where we are only concerned with the average mineral content of the total crop. In addition to the contrasting total amounts of mineral ions in the beets, there is also a very considerable variation in the proportional amounts of the individual mineral constituents. These conditions have a decided effect on the economic balance obtainable in the use of ion exchangers.

From the known chemistry of ion exchangers it is an accepted fact that a given amount of resin will remove only a definite number of either basic or acidic ions according to the type of resin. Further, for regeneration of the resins, a chemically equivalent amount of regenerant chemical must be used plus a certain excess. It is, therefore, evident that for beets with a high content of mineral ions that the length of cycle that a resin can be used before exhaustion will be relatively shorter and the amount of regenerants required per ton of beets processed will be much greater than when the beets are low in minerals.

The range of diffusable sulphated ash content in beets is roughly from 0.4 percent to 0.8 percent on weight of beets. This means that for factories with the lowest ash beets the minerals to be removed from diffusion juice are of the order of 8 pounds per ton. However, for those in the high-ash bracket, the removal must approximate 16 pounds of minerals per ton of beets. For evaluation purposes the determination of sulphated ash is the best of the simple methods; it has the advantage over carbonate ash in that at least the acid radical part of the ash is uniform. Even fully sulphated ash does not represent an entirely accurate measure of ionizable minerals since the composition of the basic portion is still variable. Sufficient accu-

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racy for preliminary predictions is obtainable by assuming that the average combining weight of the metallic base is 31 in raw diffusion juice and 35 in clarified or thin juice. This assumption of a lower combining weight in the raw juice is due to the presence therein of fair quantities of magnesium. This metal is practically eliminated in the carbonation process, and lime is also brought to a low concentration, with the result that in thin juice the ratio of potassium to sodium governs the mean combining weight.

To estimate the work that the ion exchangers must do and the amount of regenerants required, we can proceed on the basis of chemical calculations. For example, a pound of sulphated ash with a combining weight of 31 for the metallic bases is equivalent to 5.74 normal weights of both basic and of acidic ions. With 35 as the basic combining weight the equivalent is slightly less, 5.47 normal weights. It is recognized that in the case of the raw juice the basic ions (which determine the amount of sulphated ash obtained) are very slightly less than the acid radicals to be removed. For the thin juice the opposite prevails, that is, the basic portion is greater so that the cation exchanger has more work to do than the anion exchanger, and the sulphated ash value agrees best with the work done by the cation exchanger. For the extremes of diffusable sulphated ash already mentioned (0.4 to 0.8 percent on weight of beets) the corresponding normal weights of minerals to be exchanged per ton of beets would be approximately 44 and 88.

Now what do these facts mean in terms of capacity of exchanger needed per ton of beets and in terms of regeneration chemicals per ton of beets?

We will assume an exchange capacity of 17.5 normal weights per cubic foot of resin in the column. The actual capacity may be higher or lower than this depending not only on the quality of the resin but also on its tendency to compact in the column. For beets that contain about 0.4 percent diffusable sulphated ash or 44 normal weights per ton, this means that approximately 2.5 cubic feet of exchanger will be needed per ton. At the other extreme of high-ash beets (0.8 percent) the capacity needed would be 5.0 cubic feet per ton of beets. For most factories the requirement would be intermediate and will be dependent on the diffusable ash concentration of the average beet for that particular factory and season.

It may also be stated that if the maximum removal of ionized nitrogen compounds is desired, the exchanger capacity required per ton of beets will exceed the amount indicated by the ash content. This is due to the ease with which such compounds are displaced from the exchangers. Hence, at some locations where relatively high concentrations of nitrogen occur in the beets, the exchanger capacity needed will correspond to a high-ash content, but the regenerant chemicals required would still be governed by the actual ash content of the beets.

For the alkali regeneration of the anion exchanger it will be assumed that 1.1 times the chemical equivalent will suffice and that for the cation exchanger 1.3 times the theoretical quantity of the acid will be needed. (In practice the efficiency in the use of regenerant chemicals is usually much less than indicated by this example.) For the anion regeneration per ton of low-ash beets (0.4 percent), the amount of alkali required will be 44x1.1 or 48.4 normal weights. In the case of high-ash beets (0.8 percent) the amount of alkali required would be doubled or 96.8 normal weights. For the cation regeneration the amount of acid required per ton of low-ash beets (0.4 percent) would be 44x1.3 or 57.2 normal weights, while in the case of high-ash beets (0.8 percent) the amount would be 114.4 normal weights. What this means in terms of weights of chemicals is shown in table 1.

Table 1.—Weights of regenerant chemicals per ton of beets.

Reagent	Chemically pure reagent		Commercial-grade reagent		
	Low-ash beets (pounds)	High-ash beets (pounds)	Quality grade (percent)	Low-ash beets (pounds)	High-ash beets (pounds)
Caustic soda ($NaOH$)	4.26	8.52	76	4.35	8.69
Soda ash (Na_2CO_3)	5.65	11.30	58	5.70	11.40
Ammonia (NH_3)	1.81	3.62	liquid	1.81	3.62
Sulphuric acid (H_2SO_4)	6.23	12.46	66° Be′	6.69	13.38
Hydrochloric acid (HCl)	4.60	9.20	22° Be′	12.78	25.56

If ionizable minerals were the only melassigenic non-sugars in beet juice and if the total concentration of minerals were directly related to the amount of sugar normally left in molasses, then the exchange capacity and the weight of regenerants required would be directly correlated with the additional sugar made recoverable. In other words, the cost per unit of additional sugar recovered would be uniform irrespective of the ash content. But this is not the actual situation. There are colloidal and organic non-sugars which are not removed by the ion exchangers, and, further, the amount of sugar normally lost in molasses is not directly correlated with total minerals. That is, a factory with a high-ash beet may obtain better sugar extraction than another factory with a low-ash beet. This may be partly due to non-ash melassigenic constituents and in part to operating care, but from studies of molasses composition in relation to sucrose lost in molasses calculated as percentage on weight of beets, it is also evident that the relative amounts of the different mineral constituents are also a factor. Metallo-organic salts form a variable but usually large proportion of what we are here calling mineral content. These organic salts appear to be much more melassigenic than any of the strictly mineral salts such as sulphates, sulphites, or chlorides. The only exception is when chlorides are very high and sulphates low. Hence, for a given total concentration of ions when the proportion of mineral acid radicals is high, a relatively small amount of sugar is lost to molasses as compared with the situation when the proportion of organic acid radicals is high. As a matter of record, six factories with beets of essentially the same total ash content show a variation from 2.1 percent to 2.6 percent in sugar loss in molasses calculated as percentage on weight of beets. Hence, while the cost of using ion-exchange demineralization would be equal at all six factories, the additional sugar recovered would not be the same.

Therefore, the general conclusion is that when the sugar loss is high in relation to the ash concentration in the beets then ion exchange has a better chance to be profitable. When, however, the mineral content is high mainly due to strictly inorganic salts, then with a smaller amount of sugar to be made recoverable by the process, the cost will be much harder to justify. Further, under this condition of higher proportions of strictly inorganic salts, it is likely that losses through inversion during demineralization will be greater than when organic salts predominate. This would be due to the greater inverting power of sulfuric and hydrochloric acids over that of the weaker organic acids, such as the amino and sugar acids. It can also be mentioned that the application of ion exchange to raw diffusion juice should produce a greater tendency to inversion because the liberated phosphoric and oxalic acids are stronger than the sugar acids which appear to replace them in thin juice.

By way of summary the following points will be restated:

1.—Independent of the unit capacity of the exchange resins the total ionic capacity required per ton of beets will be directly related to the total mineral ions per ton of beets. Between different factories this mineral content is widely variable and some factories may need double the exchange capacity per ton of beets that is needed at another location.

2.—The weights of regenerant chemicals needed will be variable to exactly the same degree as the exchanger capacity.

3.—While, for a particular mineral content in beets the exchange capacity and quantity of regenerant chemicals are fixed, the additional amounts of sugar made recoverable by the process will be variable between different factories. This is due to the differing compositions of the mineral constituents which affect the amounts of sugar normally lost to molasses.

4.—The fully sulphated ash determined on diffusion juice or thin juice calculated as percentage on weight of beets gives a reasonably accurate basis for estimating exchange capacity and weight of regenerant chemicals required per ton of beets.