DOWEX Ion Exchange Resins

Procedure for Cross-Regeneration of Anion Resin Used in Sweeteners

Cross-Regeneration of Resins to Maximize Resin Life

Both strong acid cation (SAC) and weak base anion (WBA) resins accumulate organic foulants in service. A portion of these foulants remain on resins even after the use of normal regeneration chemicals. The practice of cross-regeneration with both SAC and WBA resins in sweetener processing is an effective method to extend the useful life of a resin by reducing the build-up of organic foulants on the resins. CAUTION: Dow does not recommend cross-regenerating resins which have had greater than six months service life without prior cross-regeneration. Cross-regeneration on overly fouled resins can causes foulants to slough off into the product syrup stream for a considerable time.

Weak Base Anion Resin Cross-Regeneration Benefits

In service, weak base anion resins remove mineral acids that are generated by the cation bed and organic acids that are generated in the starch conversion process. In addition to removing acids, weak base anion resin can also remove other organic material such as protein that passes through the cation bed, fatty acids, and color bodies. Routine regenerations with sodium hydroxide (NaOH), soda ash (Na_2CO_3) or ammonium hydroxide (NH_4OH) are incapable of fully removing these organic materials. With time, the anion resin becomes organically fouled with these materials that inhibit the kinetics which reduces operating capacity of the resin bed. It is more difficult for acids to diffuse into the resin bead during service and out of the bead during regeneration.

Cross-regenerating anion resin is beneficial because the resin swells when exhausted with acid and shrinks when restored with base to the free-base form. This swell-shrink cycle helps to evacuate the macroporous resin of some of the organic material. An analogy is to squeezing a sponge to remove soap that is trapped in the pores. In addition to the swell-shrink cycle, exposing the foulants to low pH can also affect their affinity to the resin and promote removal.

Weak Base Anion Resin Cross-Regeneration Guidelines

1) **Regenerate the anion resin with 4% NaOH.** If a normal regeneration involves either soda ash or ammonium hydroxide, it is preferable to use 4% NaOH for the cross-regeneration steps so that materials that are soluble at higher pH can be removed. The use of 4% caustic in this step will convert the resin into the free-base form; this causes the resin to shrink. The recommended caustic loading is the same as the caustic loading used in a standard regeneration of the WBA bed.

2) **Displace with 2-3 bed volumes water (15-20 gal/ft^3) over 30-60 minutes.** The caustic that is introduced should be rinsed from the anion bed before acid is added to avoid an acid-base reaction, it should not be necessary to rinse the bed to the normal targets of very low conductivity.

3) **Cross-regenerate anion resin with 2 bed volumes of 7% hydrochloric acid (HCl) over 30-60 minutes.** To swell the resin again, the resin should be cross-regenerated with 7% HCl. This will exhaust the resin with acid, cause the resin to swell, and also drastically change the pH environment, which will change the ionic attraction of some foulants so they are easier to remove. As a rule of thumb, the HCl loading into the anion bed for cross-regeneration should be the same as the volume of NaOH for anion resin regeneration.

4) **Displace with 2-3 bed volumes water (15-20 gal/ft^3) over 30-60 minutes.** The acid that is introduced should be rinsed from the bed before the final base regeneration, again to avoid an acid-base reaction, it should not be necessary to rinse the bed to the normal targets of very low conductivity since other regeneration steps will follow.

5) **Regenerate the anion resin with base, using 1.2X normal regeneration.** The final base regeneration will restore the anion resin to the free-base form so it is ready for service. This step can be accomplished with caustic, soda ash, or ammonium hydroxide (use the chemical that is normally used to regenerate the resin). Since the anion bed is almost fully exhausted with a mineral acid (HCl) an increased regeneration quantity is recommended to remove the acid. This can be

fine tuned with experience. If premature pH break is observed in the first cycle after cross-regeneration, then it may be necessary to increase the base loading in to ensure more complete regeneration of the anion bed.

6) **Rinse anion bed to plant target conductivity.** The final base regeneration should be followed with a complete rinse to low conductivity.

Note: A cost saving measure when cleaning the WBA resin is to skip the initial caustic regeneration (Step 1) and begin the cross-regeneration with 7% HCl. By skipping the initial caustic step, one of the two shrink-swell cycles will be lost; potentially leaving more foulants on the resin.

Suggested cross-regenerating conditions for DOWEX deashing resins

	DOWEX 88 SAC	DOWEX MONOSPHERE 88 SAC	DOWEX 66 WBA	DOWEX MONOSPHERE 77 WBA or DOWEX MONOSPHERE 66 WBA resin
Regenerant Concentration	7% HCl	7% HCl	4% NaOH	4% NaOH
Regenerant Level (100% basis)	6-7 lb/ft^3 96-112 kg/m^3	5-6 lb/ft^3 80-96 kg/m^3	5-6 lb/ft^3 80-96 kg/m^3	4-5 lb/ft^3 64-80 kg/m^3
Regenerant Temperature (max.)	200°F 93°C	200°F 93°C	140°F 60°C	140°F 60°C
Substitute Regenerants			5% Na_2CO_3 @ 7-8 lb/ft^3 (112-128 kg/m^3)	5% Na_2CO_3 @ 6-7 lb/ft^3 (96-112 kg/m^3)
			5% NH_4OH @ 5-6 lb/ft^3 (80-96 kg/m^3)	5% NH_4OH @ 4-5 lb/ft^3 (64-80 kg/m^3)
Cross-regenerant Chemical	4% NaOH	4% NaOH	7% HCl	7% HCl
Cross-regenerant Level	5-6 lb/ft^3 (80-96 kg/m^3)	4-5 lb/ft^3 (64-80 kg/m^3)	5-6 lb/ft^3 (80-96 kg/m^3)	4-5 lb/ft^3 (64-80 kg/m^3)

Other Considerations

ISEP and CSEP carousel units are typically not plumbed with a port for cross-regeneration. However, these systems can be plumbed and programmed to include a cross-regeneration step and in some cases this has proven to be very beneficial.

Warning: Oxidizing agents such as nitric acid attack organic ion exchange resins under certain conditions. This could lead to anything from slight resin degradation to a violent reaction (explosion). Before using strong oxidizing agents, consult sources knowledgeable in handling such materials.

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