

Methanol washing of Purolite[®] PD206 when used to remove glycerin in a dry biodiesel manufacturing facility.

Purolite[®] PD206 dry will exhaust in two capacities:

- 1.) By dehydrating biodiesel, removing glycerin through absorption which results in the media swelling.
- 2.) The other is by ion exchange of catalyst, salts and soaps exchanging primarily sodium (Na⁺) or potassium (K⁺) for hydrogen (H⁺) on the resin. This exchange also neutralizes the methylate catalyst reverting it back to methanol. Soaps will be converted back to fatty acids.

Purolite[®] PD206 loads first with glycerin well before exhausting on ion exchange. Recovering the ability for resin to absorb glycerin will not recover ionic capacity. If **Purolite**[®] PD206 exhausts on ionic capacity it will continue to absorb glycerin but to a lesser capacity. If glycerin absorption is the limiting factor, removal of glycerin from the resin within the biodiesel plant may be practical. This process is best described as **METHANOL WASHING**.

If resin exhausts ionically, after several methanol washes, the resin will need to be returned to Purolite for a traditional **IONIC REGENERATION** (minimum amount that can economically be returned is 2500lb original dry or 100cf wet, which is approximately 3 super sacks or totes). This regeneration consists of converting the resin to the H⁺ form, drying then returning to the customer. Ionic regeneration of this resin can be done many times before the resin needs replacement.

Polishing systems must consist of 2-3 vessels with at least 2 vessels online in series. This system is best located before demethylation. The lead vessel receives the greatest glycerin loading with the lag column polishing trace glycerin. Both columns should be monitored for glycerin and when free glycerin reaches 0.01% at the effluent of the second (lag) column, it should be moved to the lead position. The original lead column is taken off line for methanol washing. After washing or replacement of the resin, the cleaned column is put into the lag position or standby.

Washing of glycerin from Purolite[®] PD206 is done with the following procedure:

If the polishing system is located before demethylation, drying or removing minor methanol from the resin is not required before placing the column into service. Methanol will move from this column with the biodiesel and be removed in the demethylation system.

- A. Drain all biodiesel from the polishing column.
- B. Back- fill resin column with methanol to above resin bed. Allow this to stand 30 minutes minimum.
- C. From the top add 1 bed volumes (BV) of methanol through the bed at half design flow of the column. Let this second BV of methanol stand in the vessel for 30- 40 minutes.
- D. Pass a third BV of methanol through the resin at the same flow rate.
- E. Drain methanol from the bed to a separate dirty methanol storage vessel. This methanol loaded with glycerin can be used in the transesterification process. Be sure to adjust methanol levels in the reactor to insure sufficient methanol to complete reaction.
- F. Back fill bed with clean biodiesel to above the resin and allow it to settle, until air in the bed is completely removed.
- G. From the top, finish filling the tank with biodiesel and begin processing. The first few bed volumes of biodiesel will be high in methanol and may overwhelm the demethylation column. Be sure to circulate biodiesel to bring this methanol level down.

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Glycerin can be washed from the **Purolite**[®] **PD206** multiple times. After each wash, throughput will drop as the resin becomes more exhausted with catalyst, until runs are approximately half that of new resin. **Purolite**[®] **PD206** will continue to remove glycerin after it is ionically exhausted, although not as efficiently. Biodiesel can be monitored for elevated pH or sodium. When resin no longer removes sodium or potassium (ionically exhausted) the resin should be removed from the column and returned to Purolite for ionic regeneration. **Purolite**[®] **PD206** can be regenerated many times with a service life of 5 or more years. Please contact Purolite for availability of offsite regeneration in your region and for procedures to prepare and return resin to Purolite.

Addendum

1. **PD206** will swell approximately 2.3 times from a dry volume to methanol wet.
2. The BV's of MeOH required to remove glycerin from PD206 equals 34% of the swollen volume of resin (i.e., 100lbs of PD206 will require 12gal of methanol to equal 1BV of **PD206**).
3. Glycerin and methanol are 100% miscible therefore the methanol wash is more of a dilution of glycerin. When MeOH is filled into the bed from the bottom up the resin will begin to swell and the glycerin will begin to disperse into the methanol.
4. Glycerin is heavier than methanol and will generally settle so once the resin bed is filled with MeOH and the resin is completely swollen (requiring a minimum of 15 minutes) the resin should soak an estimated 30 minutes allowing glycerin to migrate from the bead. This diluted glycerin may also begin to settle in a downward direction.
5. After the soak MeOH should be introduced from the top and the initial BV of MeOH sent to a dirty MeOH storage. This dirty MeOH can be used in the transesterification process if water is not present.
6. Once the initial BV is out the second BV can soak or be circulated before being moved to holding tank. The third BV of MeOH should be flushed through and not re-circulated. This will move residual glycerin out and not back to the top of the polishing bed. The last BV can be used in the next MeOH wash as the first BV.
7. Once the wash is complete biodiesel will be used to move MeOH from the bed. Biodiesel can be circulated through the bed then across a demethylation column to remove the MeOH from the resin. As MeOH is removed the resin will shrink to a volume approximately 1.2 times the original dry volume. It will be difficult to remove all MeOH so demethylation afterwards will be necessary.
8. If soap is present in the biodiesel PD206 will convert this to FFA and will increase total acid number (TAN) in the polished biodiesel. If this occurs you may need to use a sodium form resin (**Purolite**[®] **D9947**) ahead of the **Purolite**[®] **PD206** to reduce soap conversion. The sodium form resin will need to be MeOH washed more frequently as the glycerin and soap will be more viscous and will collect on the surface of the resin reducing throughput and increasing pressure drop across the bed. Also, since this glycerin will be loaded with soap the MeOH used for the washing will need to be distilled before reuse in the transesterification step.

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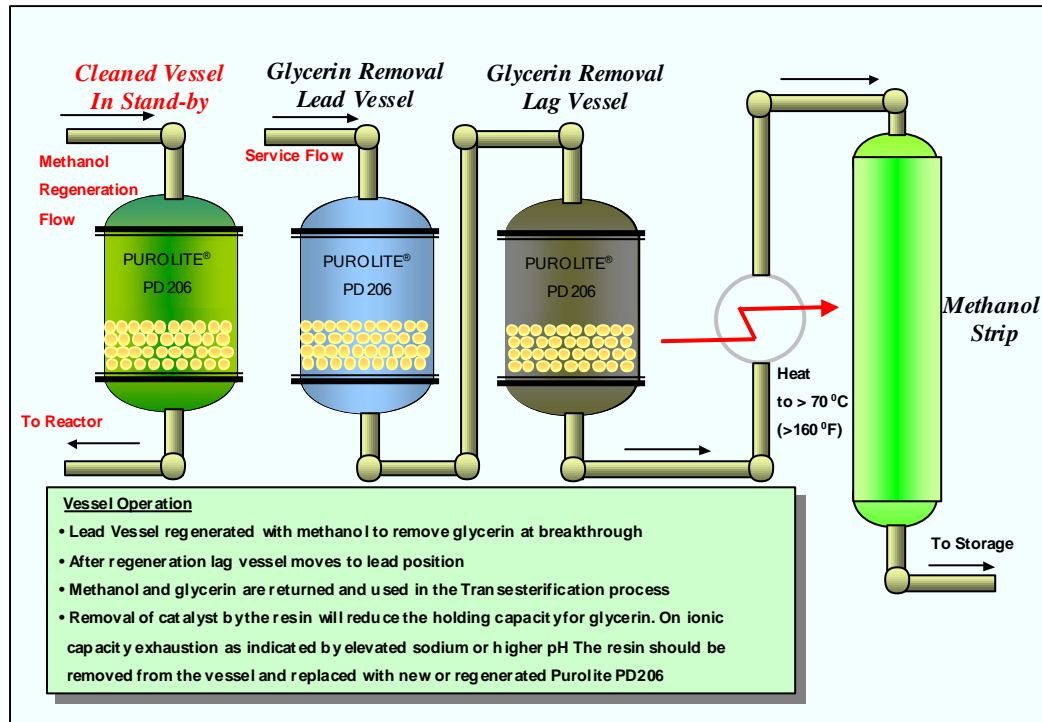
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