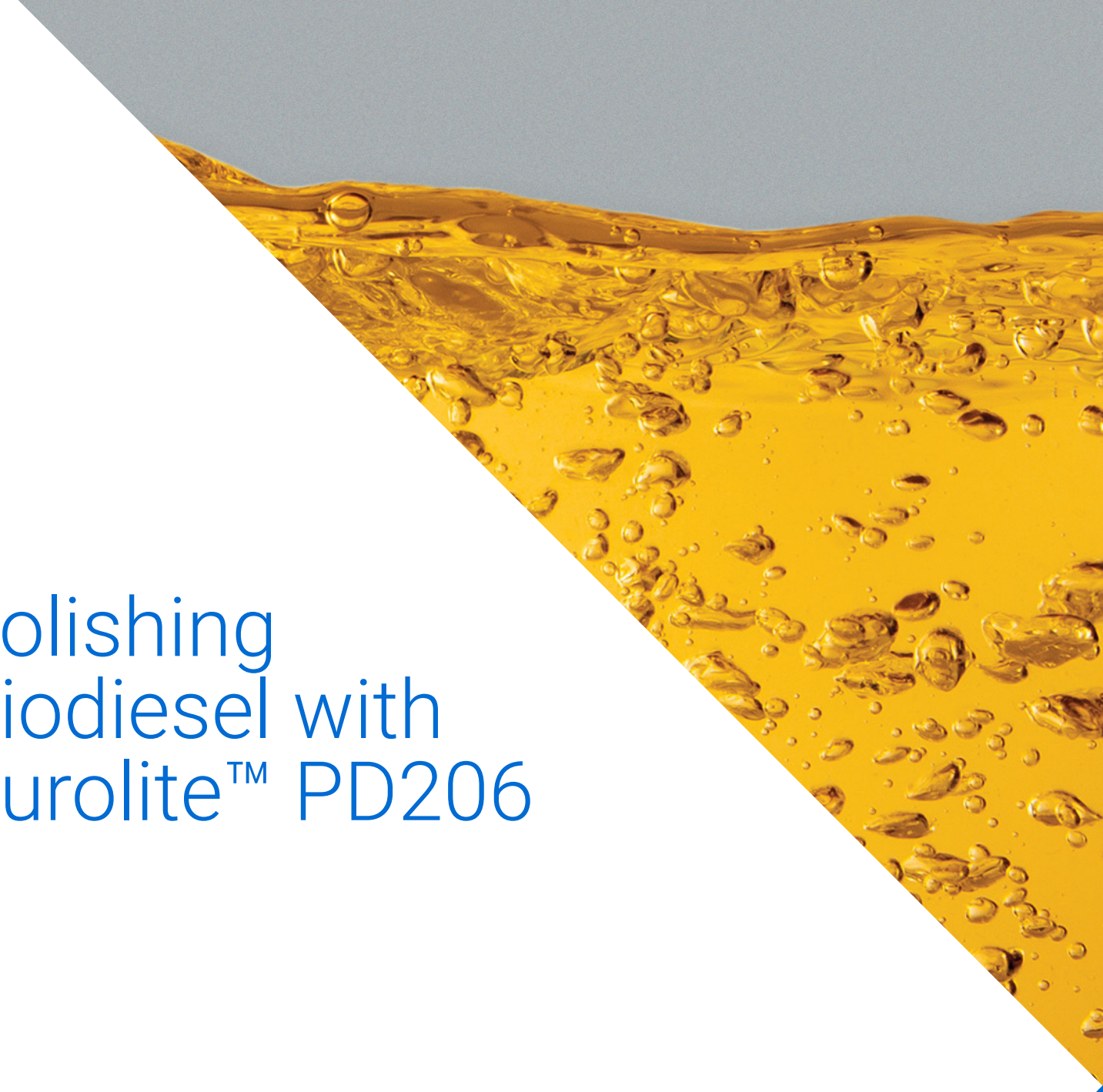

Polishing Biodiesel with Purolite™ PD206

Purolite PD206 exhausts in one of two ways — by dehydrating biodiesel or by ion exchange of catalyst, salts and soaps. The latter method neutralizes the methylate catalyst reverting it back to methanol.

ECOLAB®

Polishing Biodiesel with Purolite PD206

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Polishing Biodiesel with Purolite PD206

Production of Biodiesel or methyl ester can be relatively simple such that some even make this environmentally friendly fuel in the back of trucks. Although simple to make, producing biodiesel that meets ASTM and EN standards can be considered an art. Several mechanical and chemical technologies are required to meet the demand of this industry, one technology is the use of ion exchange.

The ion exchange resin, Purolite PD206, is a synthetic copolymer formed from components of crude oil. This copolymer bead is functionalized with an ion exchange group that will remove soluble ions such as the catalyst sodium methylate remaining from the transesterification process as well as adsorb trace reaction by-products of glycerin and water that remain in biodiesel.

Purolite PD206 is best used in a final polishing step because of:

A. Ease of use

B. Capacity to adsorb glycerin and water

C. Removal of dissolved salts

D. Ability to be reused; the glycerin that loads on this resin can easily be removed by a methanol wash process

Methanol Washing of Purolite PD206 When Used to Remove Glycerin and Water in a Dry Biodiesel Manufacturing Facility

Purolite PD206 is a dry whole bead media that performs two functions:

1. Adsorb polar compounds such as trace glycerin and water from methyl ester. As polar compounds are removed, Purolite PD206 media will swell to twice its original volume before exhausting. Polar compounds adsorbed by the media are displaced with methanol allowing the media to be reused.

2. Salts associated with catalyst, specifically sodium (Na^+) or potassium (K^+), are removed from biodiesel by ion exchange and form methanol or water. Once the resin exchange sites are fully exhausted with these salts, the media will continue to remove glycerin but will not remove additional catalyst.

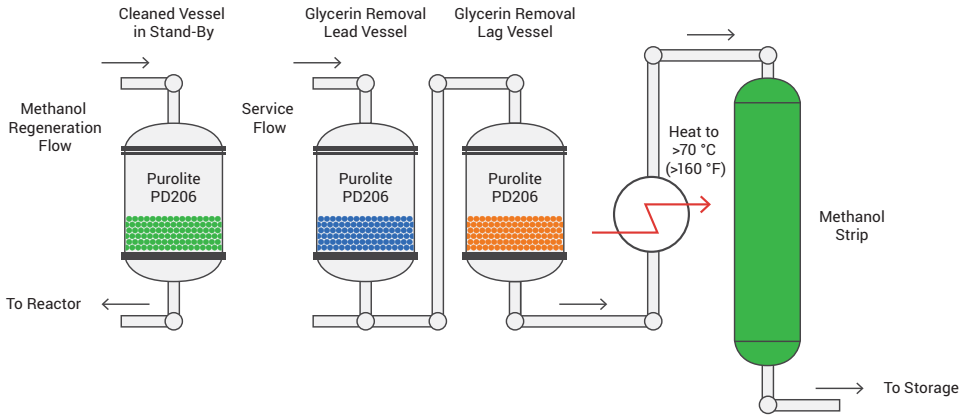
Purolite PD206 loads with glycerin and water well before exhausting the ion exchange capacity. Recovering the ability for resin to absorb glycerin and water will not recover ionic capacity. If Purolite PD206 exhausts on ionic capacity, replacement is normally recommended. If glycerin and water absorption is the limiting factor, removal of these contaminants from the resin is practical using the methanol washing process detailed below.

Purolite PD206 polishing systems consist of 2–3 vessels with at least 2 vessels online in series. Vessels are best located before demethylation. The lead vessel receives the greatest glycerin loading with the lag column polishing trace glycerin that passes the lead column. 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 offline for methanol washing. After washing or replacement of the resin, the cleaned column is put into the lag position or standby.

FIGURE 1

Methanol Wash to Remove Glycerin

Methanol Wash to Remove Glycerin



Vessel Operation

- Lead Vessel regenerated with methanol to remove glycerin at breakthrough
- After regeneration lag vessel moves to lead position
- Methanol and glycerin are returned and used in the transesterification process
- Removal of catalyst by the resin will reduce the holding capacity for glycerin. Once ionic capacity has been exhausted, as indicated by elevated sodium or higher pH, the resin should be removed from the vessel and replaced with fresh Purolite PD206.

Washing of Glycerin From Purolite PD206

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.

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 replaced with Purolite PD206.

Raw Biodiesel (Left)

Polished Biodiesel with PD206 (Right)

Addendum

1. Purolite 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 Purolite PD206 equals 34% of the swollen volume of resin (i.e., 100 lbs of Purolite PD206 will require 12 gal of methanol to equal 1BV of Purolite 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, Purolite PD206 will convert this to free fatty acids and will increase total acid number (TAN) in the polished biodiesel. If this occurs, you may need to use a sodium form resin (Puropack™ PPC150) in place 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 may contain soap, the MeOH used for the washing will need to be distilled before reuse in the transesterification step.

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