

What is Purolite[®] PD 206?

Purolite[®] **PD206** is a dry ion exchange resin that functions as a combined desiccant and ion exchange media it is specially formulated to enable maximum removal of free glycerin as well as catalyst, soaps and salts from crude biodiesel. **Purolite**[®] **PD206** is designed for use in “purification” vessels primarily installed after phase separation and before or after demethylation.

What are the benefits of using Purolite[®] PD 206?

Purolite[®] **PD206** is a cost effective method of purifying biodiesel removing glycerin and ionic contaminants. This technology provides a simple, low energy process to eliminate water wash steps and other inorganic labor intensive processes. Higher biodiesel production can be obtained by reducing processing time compared to alternate purification methods. **Purolite**[®] **PD206** will reduce waste disposal of both liquid and solids. Since **Purolite**[®] **PD206** may be regenerated, returning it to original performance; this product can be reused for many years before being disposed.

Where is Purolite[®] PD 206 used?

Polishing columns loaded with **Purolite**[®] **PD206** can be located in one of two positions within a biodiesel production facility. These positions are as a final polishing after demethylation or for glycerin removal, before demethylation.

Final polishing should be used only if biodiesel after demethylation contains <0.1% glycerin and methanol meets ASTM specification consistently. Resin used in this position exhausts on glycerin and must be replaced in the lead column with new or regenerated material when free glycerin leaving the lag column is 0.01%.

If glycerin levels after demethylation are above 0.1% it is strongly recommended to position **Purolite**[®] **PD206** before demethylation. This allows glycerin to be reduced to <.01% consistently along with the removal of residual catalyst minimizing back reactions in the demethylation column. Resin in this position is easily flushed with methanol to remove glycerin. This is done within the biodiesel plant and glycerin loaded methanol can be recycled without issue back to the transesterification process. **Purolite**[®] **PD206** used in this method is limited by ionic loading but can be flushed many times before the resin exhausts ionically, at which time the resin is replaced with new or regenerated resin.

How is Purolite[®] PD 206 used?

Dry **Purolite**[®] **PD206** should be loaded into two or three tall narrow purification columns (“lead”, “lag” and/or “standby”) operated in series. The lead vessel does the bulk of the glycerin removal and the second or lag vessel polishes trace glycerin leaking from the lead vessel. Recommended vessel

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configuration is for the height to be at least 3 times the diameter and that resin be filled to 50% of the vessel height as the resin will swell to nearly twice its original volume.

Crude biodiesel flows through the lead and lag vessels to maximize media loading on the lead column optimizing operating cost and labor. When free glycerin leaving the lag vessel exceeds 0.01%, the standby vessel is put on-line in the lag position, the original lag is now in the lead position and the original lead bed is now removed from service. Media in this off-line vessel is either replaced if operated after demethylation or flushed with methanol to remove glycerin if positioned before demethylation then placed in standby (or in the lag position if only two vessels are used).

How much Purolite® PD 206 do I need?

A Purolite® PD206 polishing system is sized based on design flow for processing biodiesel. The minimum size recommended is a system that will handle 3 bed volumes per hour (BV/h). Since glycerin is absorbed slowly, flows of 1.5-2 BV/h may be advised. As an example, a 5 million gallon per year (19 thousand m³) plant operating 5 day per week 16 hrs per day will require a minimum flow of 20 gpm. (77 litres/min) A 20 gpm system will consist of 3 vessels each loaded with 2600 lb (1.2 metric ton) of Purolite® PD206. Consult your Purolite representative for complete details.

How long will Purolite® PD 206 last

A Purolite® PD206 system located before demethylation is limited by ionic loading. Glycerin absorbed is flushed with 2-3 bed volumes of methanol 12-24 time before the resin is ionically exhausted. Ionic loading is primarily due to catalyst remaining in the glycerin and biodiesel and to a lesser degree neutral salts from the oil and additives. Exchange sites on the resin change from H⁺ to Na⁺ or K⁺. As H⁺ is converted the ability of the resin to hold glycerin is reduced. When the lead column allows catalyst to pass, this column should be replaced. Estimated column life is 3-6 months. This resin can be returned to Purolite for regeneration.

If the system is in the final polishing position after demethylation glycerin becomes the limiting factor. When the lead column becomes loaded resin is replaced. Resin replacement will be 4-8 weeks depending on the level of glycerin in the biodiesel. If methanol and or water pass demethylation resin life will be significantly reduced. A well operated demethylation column is essential as resin is not efficient at holding volatile materials like methanol and water.

How does % methanol in B100 affect flash point?

Flash point is directly related to percent methanol in the biodiesel. Methanol must be below .08% by weight to achieve the current ASTM standard of 130 °C flash points. This is best achieved by vacuum demethylation at temperatures of 160°F (71°C). A small amount of nitrogen bubbled to purged through the biodiesel significantly aids in methanol removal. Purolite® PD206 capacity for methanol in a dynamic operation is limited therefore demethylation is the recommended method to remove methanol and control flash point.

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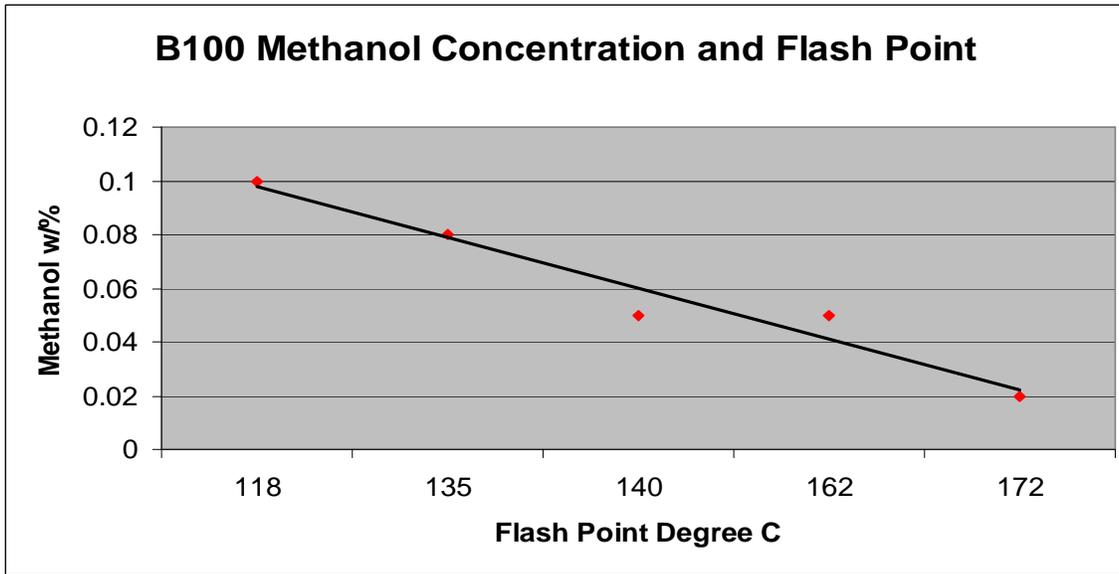
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What impacts performance of Purolite[®] PD 206

Purolite[®] PD206 in the hydrogen form will load high levels of glycerin. If methanol and or water are present this will prematurely exhaust the resin when operated in a final polishing position. When operated before demethylation methanol and water are allowed to pass through the resin where they will be removed by demethylation. Water should not be present in a dry process. If water is present check moisture of raw oil, methanol and methylate.

Some feed stocks will have higher free fatty acids (FFA) present. If esterification is done with acid to convert FFA to methyl esters trace water that is produced should be removed before transesterification. Waters in the Transesterification process will create additional FFA and may impact acid number after Purolite[®] PD206.

Maintaining efficiency of mechanical separation equipment and the completion of reaction is critical. **Purolite[®] PD206** has virtually no impact on removing mono, di, or triglyceride.

If higher levels of catalyst or salts from the raw oil are present ionic capacity of the **Purolite[®] PD206** will be reduced especially when operated before demethylation. As the hydrogen capacity is reduced the glycerin loading capacity will drop increasing frequency of methanol flushing.

Acid form resin significantly reduces viscosity of glycerin improving adsorption into the resin. Sodium form glycerin's can be very viscous which can limit their adsorption into the resin bead.

How do I know when Purolite[®] PD 206 is exhausted?

Purolite[®] PD206 will adsorb methanol, water and glycerin until the adsorption capacity is reached. At this point methanol will be displaced, then water and finally glycerin. If methanol and water are

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properly removed, the lead column should exhaust when glycerin begins to break through the lag column or when turbidity increases in the effluent.

There are some simple field tests if analytical testing is not available that may assist in determining breakthrough if water and methanol are limiting factors;

Effluent clarity - which is a simple visual inspection through a glass container? If you can read news print through 4 inches of B100 clarity is good.

Water wash conductivity – is simply taking 20 ml of B100 and 80 ml of deionized water. Shaking vigorously in a closed container or separatory funnel then allowing the water to separate before measuring conductivity. Catalyst and glycerin will contribute to an elevated conductivity.

Methylene Purple test – this color indicator will change from a lemon yellow to purple if catalyst increases in the polished biodiesel

Can Purolite® PD 206 be regenerated?

The term regeneration is often used for two separate processes when resins are used for polishing biodiesel. One method is when **Purolite® PD206** has glycerin flushed with 2-3 BV of methanol. Columns operated before demethylation will displace residual methanol from the resin with the biodiesel. Demethylation then removes methanol from the biodiesel

The second method is when resin loses ionic capacity it can be converted back to the original acid content at Purolite. Resins returned to Purolite can be reconditioned to their original condition converting exchange sites back to the acid form and then dried to original low moisture. This resin is returned to the customer, reducing overall operation costs and solid waste volumes. Contact your Purolite representative for more information on this service.

What design Parameters for Purolite® PD 206 vessels need to be considered?

Purolite® PD206 will perform at ambient temperature, saving on energy costs. The maximum operating temperature should not exceed 300°F or 150°C. (Deterioration of the resin increases with increasing temperatures)

Vessels should be designed with a man-way at the top and base of the vessel to facilitate loading and unloading of **Purolite® PD206**. Providing a man-way located above the support bed or screen is recommended to facilitate inspection of the vessel and repair of internal piping and distributors/nozzles when necessary.

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What screen size is needed to retain Purolite® PD 206?

A support screen size no greater than 150 micron is recommended.

How do I load the vessels and start up with DRY Purolite® PD 206?

Add initial amount of **Purolite® PD206** to the vessels slowly (avoiding mechanical breakage of **Purolite® PD206** or support dish or screen when the product hits the bottom of vessel) into the bed to cover the bottom screen area. A small amount of biodiesel in the vessel or a sleeve guided loading is recommended. After this, loading can be accelerated. Once the loading is completed, back fill the bed with purified biodiesel (if possible) to remove any air. When biodiesel completely covers the resin, allow the bed to sit for several hours in order to purge any entrained air. Close up the vessels and place online.

How do I remove and recharge Purolite® PD 206?

An access port at the top and at the base of the vessels allows for easy installation and removal of **Purolite® PD206**. This media has excellent flow characteristics both dry and when exhausted. New resin should be charged through the top port of the vessel and is transferred dry. Load **Purolite® PD206** to occupy 50% of the vessel capacity. This will allow for swelling of the media as it adsorbs glycerin.

Spent **Purolite® PD206** should be removed through the bottom port into a shipping container if being returned for regeneration or trash container if being disposed of. Please contact yoy Purolite representative to find out more about our regeneration services.

How do I dispose Purolite® PD 206?

Purolite® PD206 can be disposed of as a non-hazardous material, as long as methanol and biodiesel is removed is removed. Consult with your local waste disposal supplier for specific requirements.

For more information visit our WEBSITE: <http://www.purolite.com>

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