



AMBERLITE™ BD10DRY™
Simple Process. Cost Effective Solution.

USERS GUIDE



**THE
AMBER
ADVANTAGE:**

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AMBERLITE™ BD10DRY™

THE LEADER IN BIODIESEL PURIFICATION

TO LAB TEST A SAMPLE OF AMBERLITE BD10DRY PURIFICATION MEDIA

To test Amberlite BD10DRY purification media we encourage you to use the "contaminated biodiesel" that has been well-separated from the glycerol phase after your transesterification process. This biodiesel should not be dried and preferably contains methanol traces. From experience, this phase usually contains less than 400 ppm of impurity cations from soap and catalyst. You can also prepare test solutions using pure biodiesel spiked with catalyst.

a) Column Purification Mode

Amberlite BD10DRY is a specialty polymer media that is most effective at removing impurities and glycerol traces when used in a "column mode". For lab testing, a glass column with roughly a 10:1 height:width ratio is recommended. A suitable column can be obtained from Ace Glass (Vineland, NJ; part number 5820-30; 25mm (1 inch) diameter by 300 mm (12 inch) length). A bed support must be used to keep the polymer beads in the column while allowing the biodiesel to easily flow through. Generally, glass frits or a stainless steel screen can be used for this purpose where the openings in the screen are no greater than about 0.18mm (80 mesh).

The column should be loaded about ¼ full with biodiesel and the Amberlite BD10DRY media is then poured into the column. The presence of a liquid "heel" in the column helps to ensure uniform packing of the beads as they are loaded which minimizes possible channeling or flow distribution irregularities.

The column should be filled no more than 1/3 full with Amberlite BD10DRY media in order to allow for expansion of the resin as it removes impurities. Avoid contacting the media with pure methanol or water as this will cause it to swell up to 3 times in volume and the glass column can shatter if insufficient void space is left at the top of the column.

A liquid head of at least 25 mm (1 inch) should be maintained above the resin to prevent air infiltration into the bed. Gravity feed can then be used to pass contaminated biodiesel through the bed, although a pump will allow more precise control of flowrate.

The flowrate of the biodiesel in the column should be around 3 L/hour of biodiesel per kg of Amberlite BD10DRY in the column (0.36 gallon/hour per lb). With gravity flow, the flowrate can be easily controlled by using a pinchcock on a rubber hose connected to the column outlet fitting. The biodiesel recovered at the bottom of the column can then be analyzed.

b) Batch Purification Mode

Amberlite BD10DRY technology will yield the best results (lowest concentration of catalyst, soap, and glycerol in the treated biodiesel) when used in a column purification mode (see above). Use in a batch purification mode is therefore not recommended. It is, however, a quick and easy way to demonstrate the performance of Amberlite BD10DRY technology.

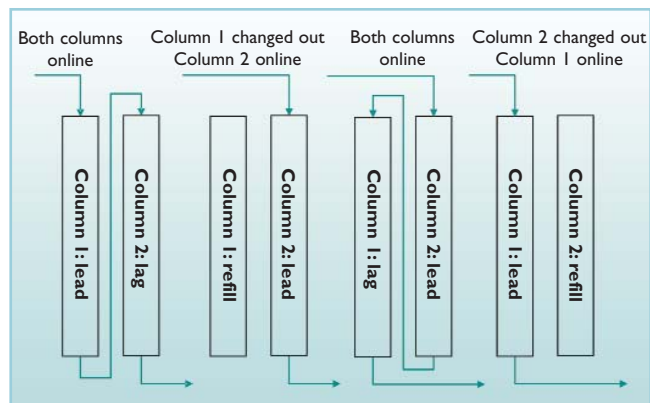
- Pour 10 g of Amberlite BD10DRY media into a glass vessel (e.g. a beaker).
- Add 100 mL of contaminated biodiesel and stir gently for one hour.
- Decant the biodiesel and analyze.



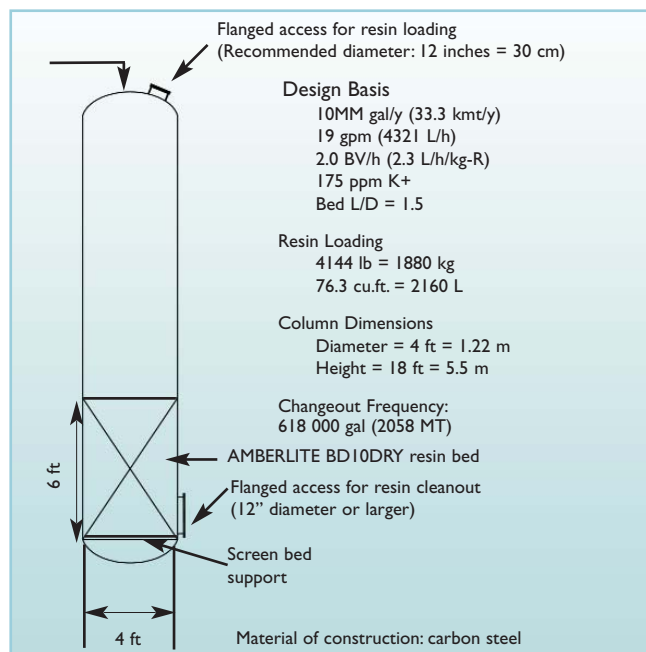
If you have any questions regarding these procedures, please call your local Rohm and Haas sales office. You will find the contact details on the back cover of this brochure.

INDUSTRIAL USAGE

For a continuous process, it is recommended to install two columns to eliminate downtime when replacing spent resin. Please note that introducing a second column will not impact your consumption of Amberlite BD10DRY. The two columns are switched by valves to alternate between "lead" and "lag" treatment positions, as shown in the following figure.



A Rohm and Haas Technical representative will work with you to select a column design best suited to your process. A column design sketch based on 10 MM gallons (37.8 MM liters) per year throughput is enclosed.

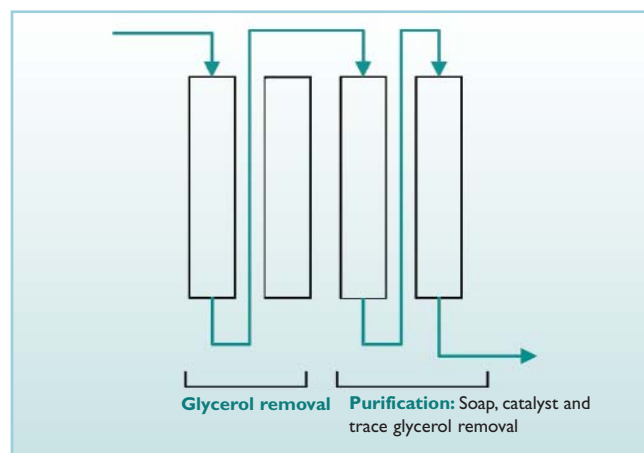


CAUTION! Amberlite BD10DRY technology is designed to be used in a water-free process. The polymer beads will swell to up to 3 x their original volume as they absorb water. The beads will also swell over their life cycle as they remove impurities, including methanol and glycerol. It is therefore strongly recommended to leave sufficient void space in the columns.



Extra glycerol removing capacity

Even higher capacity for glycerol can be achieved by adding one or two extra columns of Amberlite BD10DRY media dedicated exclusively to glycerol removal, as shown in the following figure. These additional glycerol removal columns are inserted just after the phase separation step. Note that the Amberlite BD10DRY media purchased for these columns will not impact your overall resin consumption. Why? Because when the glycerol removal columns are initially installed they will remove not only glycerol but also catalyst and soap. Therefore consumption of resin in the downstream purification columns will be saved.



When the media in a glycerol removal column is saturated with glycerol, the column can be regenerated by rinsing with methanol. The methanol solution coming off the column can then be sent back to the transesterification unit where the methanol is recycled. The interest in having two columns dedicated to glycerol removal is that the plant can continue to operate while the first column is being regenerated. These columns can thus be regenerated several times to reestablish their glycerol removing capacity. However their capacity to remove soap and catalyst is finite; in the steady state of operation they will only serve to remove glycerol. Under industrial conditions, the Amberlite BD10DRY media in the glycerol columns is usually replaced from time to time by the spent resin in the downstream purification columns.

FREQUENTLY ASKED QUESTIONS

Do I still need to do a water wash?

No. After transesterification, the biodiesel phase is separated by using settlers or centrifuges and then passed directly through Amberlite BD10DRY.

Should I remove the methanol from the biodiesel before or after purifying with Amberlite BD10DRY?

We recommend drying the biodiesel after it has been purified by Amberlite BD10DRY. Methanol traces in the biodiesel will enhance the effectiveness of Amberlite BD10DRY to remove soap and catalyst.

What filtering accessories are required?

None. The column towers contain a permanent sieving Johnson screen plate. One can think of the columns of Amberlite BD10DRY resin as self-contained filtering units.

How often must the resin be changed?

Each weight unit of Amberlite BD10DRY has a given capacity for purifying biodiesel. In a well-run process with good phase separation, 1 lb (kg) of Amberlite BD10DRY resin will treat 900 to 1600 lbs (kgs) of biodiesel. The lifetime of the resin thus depends on three main factors: (1) the impurity levels in the incoming contaminated biodiesel stream, (2) the flow rate, and (3) the amount of Amberlite BD10DRY in the column. Note that changing the loading of the columns (while maintaining at least 200% freeboard) will affect the replacement rate but will not change your overall consumption of Amberlite BD10DRY.

How do I know when the Amberlite BD10DRY media needs to be changed?

The purification capacity of Amberlite BD10DRY is exhausted when impurities are no longer captured by the resin. At this "breakthrough" point, one will start to detect low levels of cations in the biodiesel that has passed through the column. Although we recommend that the Industry Standard test methods be used, it is often practical to use a common indicator to detect when the breakthrough point has been reached.

What size column do I need?

The column size will depend on the production rate you want to achieve in your plant. It should be noted that the size of the column will not impact your overall resin consumption rate or the operating cost. Our customers typically find it convenient to change the resin every 4 to 8 weeks. Rohm and Haas can assist in determining the appropriate column size to achieve this changeout interval.

How do I remove the spent Amberlite BD10DRY from the column?

The columns should have a port at the bottom for unloading spent resin. Most of the resin will flow out on its own. To facilitate unloading, it may be convenient to incline the screen plate at the bottom of the column. Residual resin can be vacuumed, blown, or transferred out in a slurry.

How much Amberlite BD10DRY should I load in the column?

The column should only be loaded about 25 to 30% full to allow for swelling as the resin absorbs water and methanol traces. Be sure to maintain sufficient headspace, at least 50 cm, above the resin in its fully swollen state (we recommend to budget a swelling of 3 times the original volume for safety reasons) to ensure proper mixing.

How do I load fresh Amberlite BD10DRY into column?

Safety Note: If the relative humidity is less than 60%, an explosion risk does exist due to a potential static discharge when loading fresh Amberlite BD10DRY resin into a vessel that previously contained biodiesel. To minimize any explosion risk it is recommended to rinse the vessel with biodiesel containing less than 2.5 weight % methanol prior to loading fresh Amberlite BD10DRY resin.

If the methanol concentration in the biodiesel is unknown, another possible approach is to use a portable flammable gas detector to determine that a flammable environment is not present in the vessel prior to loading the fresh Amberlite BD10DRY resin.

1. Open the top of the vessel. Examine the vessel to make sure that the old resin and biodiesel have been removed.
2. Lift the drums to the top of the vessel and load the resin by dumping from the drums into the empty vessel. Make sure that the vessel is not loaded more than 1/3 full with Amberlite BD10DRY resin.
3. Close the top of the vessel. Introduce biodiesel (use purified biodiesel if available) from the bottom of the vessel until the resin is covered. Allow a minimum of one hour for the resin to swell in the biodiesel prior to starting the flow.
4. Start the flow of biodiesel from the top of the vessel at the specified flow rate (approximately 2 BV/h).

How do I unload Amberlite BD10DRY?

1. Open the valve at the bottom of the vessel and allow the biodiesel to drain out of the bed of Amberlite BD10DRY resin.
2. If available, purge the resin bed with nitrogen or compressed air for one hour to remove additional biodiesel that may be in the resin bed.
3. Open the manhole and empty the used Amberlite BD10DRY resin into containers. If the resin will be disposed of these containers should be suitable for transporting.

What flow rate should be used?

The flow rate should be chosen by the biodiesel producer as a function of the target production capacity. Rohm and Haas will then work with the biodiesel producer to design the columns appropriately. As a general rule, the flowrate should be around 3 Liters per hour per kilogram of installed Amberlite BD10DRY (or 0.36 gallons/hour per lb). Thus, if the column contains 1000 kg (2205 lb) of BD10DRY, the biodiesel flowrate should be around 3000 Liters per hour (794 gal/hour).

Do I need a pump or is gravitational force sufficient for the biodiesel to flow through the columns?

For small scale (e.g. laboratory) or batch treatment operations, it may be possible to use gravity flow although careful thought should be given to how flowrate is controlled. It is highly recommended, however, that large scale commercial installations should use a pump to control the flow through the column.

Are there any temperature restrictions?

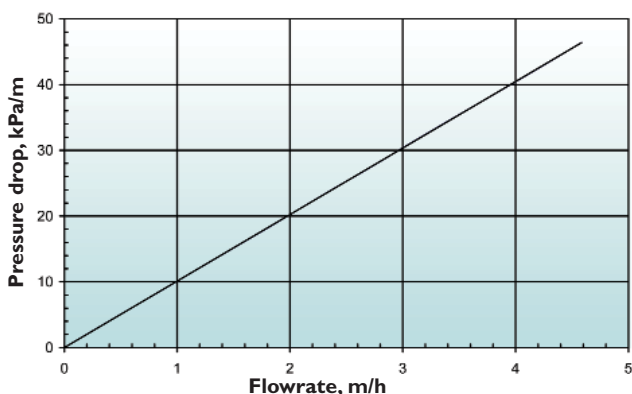
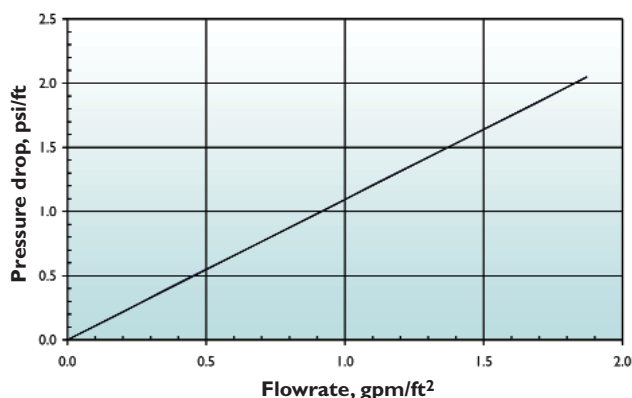
Amberlite BD10DRY has a performance range from ambient temperature up to temperatures around 120°C (248°F).

How do I know when I need to regenerate the glycerol purification columns and what is the process?

Glycerol breakthrough can be measured using the ASTM test method D-6584. For information on the methanol regeneration procedure, please see the document "Methanol regeneration procedure for Amberlite BD10DRY". The Amberlite BD10DRY purchased for these columns will not impact your overall resin consumption because when the glycerol removal columns are initially installed they will remove not only glycerol but also catalyst and soap. The resin in the glycerol columns can be replaced from time to time by the spent resin in the downstream purification columns. In practice the glycerol purification resin is replaced every 7 to 10 desorption cycles.

What is the pressure drop across the resin bed and how will this change as the resin swells?

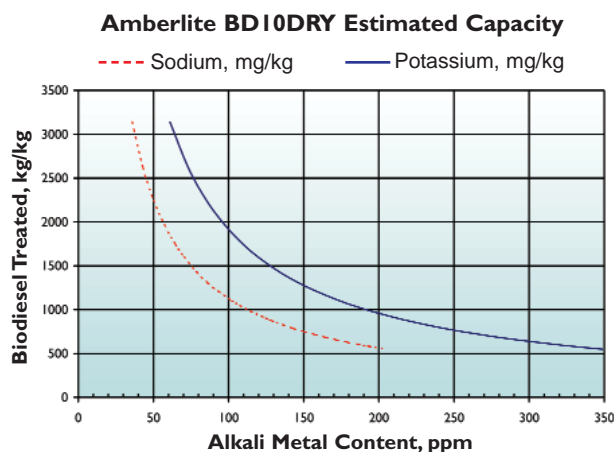
The pressure drop per unit of bed length will depend on the biodiesel linear flow velocity and can be estimated from the graphs below. The pressure drop will not significantly change as the resin swells during operation.



How much biodiesel will Amberlite BD10DRY treat?

The usage rate for Amberlite BD10DRY will depend on the system design including column configuration and operating conditions. In order to maximize the amount of biodiesel treated, a multiple column configuration is strongly recommended where the lead column is overrun to complete ionic exhaustion by soap. Please see the suggested system layout elsewhere in this document.

The removal of free glycerol and soap by Amberlite BD10DRY occurs via two different mechanisms. For glycerol removal, the resin can be regenerated when it is exhausted and can therefore be used for many glycerol adsorption cycles. The removal of soap therefore effectively determines the operating life of a charge of Amberlite BD10DRY and this will depend on the concentration of soap in the crude biodiesel. As a rough guideline, 1 kg of Amberlite BD10DRY will treat between 900 kg and 1600 kg of crude biodiesel for soap removal. The estimated usage rate can be inferred from the following figure. The alkali metal (sodium or potassium) concentration can be calculated from the soap concentration. For example, a soap concentration of 1500 ppm as potassium oleate corresponds to a potassium concentration of 183 ppm. Similarly, a soap concentration of 1425 ppm as sodium oleate corresponds to a sodium concentration of 108 ppm.



Can the biodiesel purified with Amberlite BD10DRY meet the specifications for mono- and diglycerides (total glycerine)?

Yes. Amberlite BD10DRY users consistently produce biodiesel that exceeds all local specifications. The presence of mono- and diglycerides indicates that the transesterification reaction is incomplete. These species often signal a dangerous process inefficiency and must be dealt with at their source where they are easily eliminated by refining process parameters including reaction time, temperature, and catalyst loading.

Are there any restrictions related to resin disposal?

Amberlite BD10DRY is a non-hazardous material. Please check local regulations regarding disposal. Incineration and land filling are two options that are often adopted.

For other general information, please visit the FAQ page at www.amberlyst.com

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CAUTION

Ion exchange resins and polymeric adsorbents, as produced, contain by-products resulting from the manufacturing process. The user must determine the extent to which organic by-products must be removed for any particular use and establish techniques to assure that the appropriate level of purity is achieved for that use. The user must ensure compliance with all prudent safety standards and regulatory requirements governing the application. Except where specifically otherwise stated, Rohm and Haas does not recommend its ion exchange resins or polymeric adsorbents, as supplied, as being suitable or appropriately pure for any particular use. Consult your Rohm and Haas technical representative for further information. Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact. Nitric acid and other strong oxidising agents can cause explosive type reactions when mixed with Ion Exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidising agent such as nitric acid is contemplated. Before using strong oxidising agents in contact with Ion Exchange Resins, consult sources knowledgeable in the handling of these materials.

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