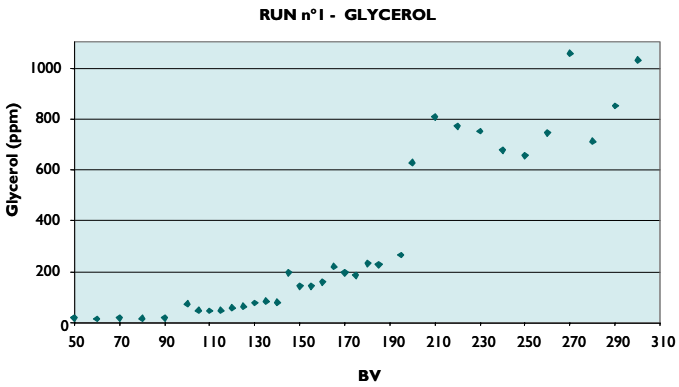
PRACTICAL GUIDE

Methanol regeneration procedure for AMBERLITE™ BD10DRY™ Resin

Amberlite BD10DRY will remove glycerol from biodiesel by an adsorption process. The very polar glycerol molecule prefers the polar environment of Amberlite BD10DRY rather than the biodiesel phase that consists of fatty acid methyl esters which are quite non-polar. The adsorption capacity for glycerol depends heavily on the methanol content of the raw biodiesel: the higher the methanol content the lower is the capacity. Since it is an adsorption process, the capacity also depends on the concentration of free glycerol in the raw biodiesel. We observed that at a methanol level of 2,7 % and a glycerol content of 1 000 ppm, Amberlite BD10DRY adsorbs an equilibrium capacity of 566 g of glycerol per kg. The operating capacity achievable under dynamic conditions is a little lower – in the region of 350 – 450 g per kg.

The glycerol breakthrough point can be measured by regularly determining the glycerol content in the treated biodiesel using either the ASTM test method D-6584 or EN test method EN 14105 (see Figure 1 below).

Fig. 1: Breakthrough curve for glycerol



In addition to the glycerol breakthrough, there also will be an ionic breakthrough point where Amberlite BD10DRY is removing Na or K from the crude biodiesel via an ion-exchange mechanism. These two mechanisms – ion exchange and adsorption – and therefore their breakthrough points are independent of each other. Which breakthrough occurs first depends on the level of the impurities in the raw biodiesel. In any case, ionically exhausted Amberlite BD10DRY can still adsorb glycerol as efficiently as fresh material. At customers where the glycerol breakthrough occurs prior to the ionic breakthrough, a regeneration procedure is recommended to recover the glycerol adsorption ability of Amberlite BD10DRY.

The regeneration procedure is carried out with methanol. The high polarity of the methanol enables the glycerol to desorb from Amberlite BD10DRY. After regeneration, Amberlite BD10DRY will again be able to remove glycerol and a similar capacity compared to the original one is achievable. The regeneration procedure can be carried out multiple times. However, the capacity will decrease with time. Practically, regeneration is carried out 5 – 10 times.

There are several ways of applying the regeneration procedure. The easiest procedure is as follows:

1. Drain the biodiesel from the column
2. Introduce 1 – 1,5 bed volumes (BVs) of methanol from the bottom such that all Amberlite BD10DRY is covered with methanol
3. Re-circulate the methanol downflow for a minimum of 4 hours at 2 BV/h
4. Drain methanol which can be used for transesterification
5. Introduce biodiesel from the bottom and allow a minimum 1 hour stand before starting the service cycle.

The advantage of this procedure is that there will be virtually no liquid waste since the methanol can be re-used in the transesterification reactor. However, a more efficient procedure can be applied by passing approximately 5 BVs of methanol downflow through the column. Such a procedure may fully recover the original glycerol capacity of Amberlite BD10DRY but this may not be applicable from a practical point of view.

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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 Company 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.