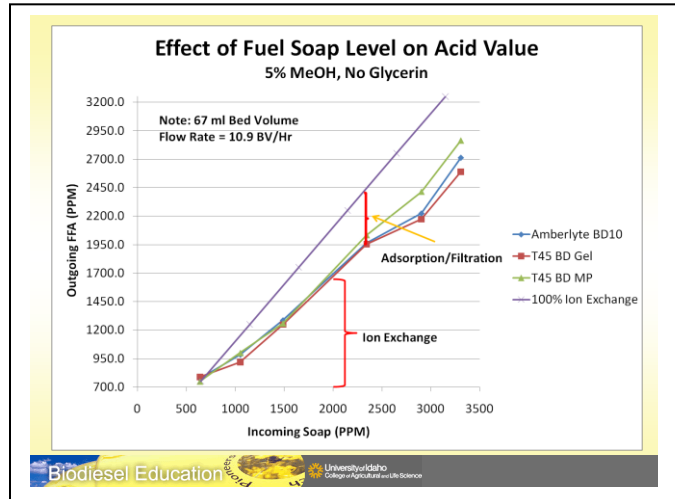


Methanol Regeneration Procedure

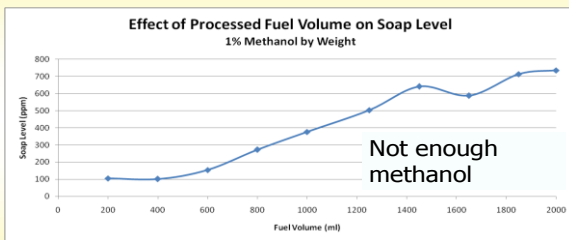
Tulsion T45 BD and T45 BD Macro Resins

Thermax T45 BD and T45 BD Macro will remove glycerol / glycerin from raw biodiesel fuel through the combined mechanisms of adsorption and ion exchange.

The very polar glycerin molecule prefers the polar environment of T45 BD Macro or T45 BD rather than the biodiesel phase that consists of fatty acid methyl esters which are non-polar. The adsorption capacity for glycerol depends heavily of the methanol content of the raw biodiesel. While a higher methanol level will result in lower operating capacity to adsorb glycerol, the adsorptive capacity of the resins is dependent on a low percentage of methanol in the raw biodiesel fuel to be purified. Studies conducted at University of Idaho clearly show the methanol content needs to be greater than 1%; 2-4% has worked well in many installations.

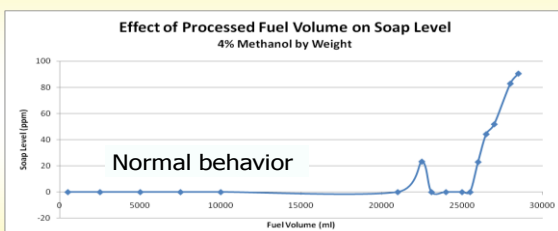


Effect of methanol (1%)



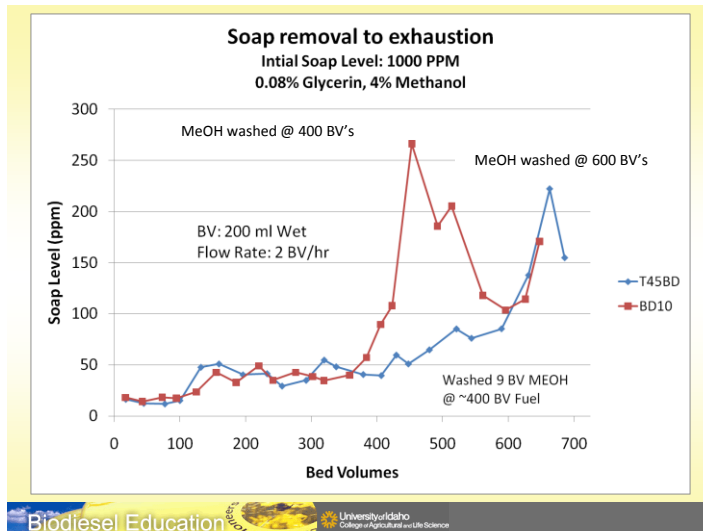
1500 ppm initial soap level

Effect of methanol (4%)



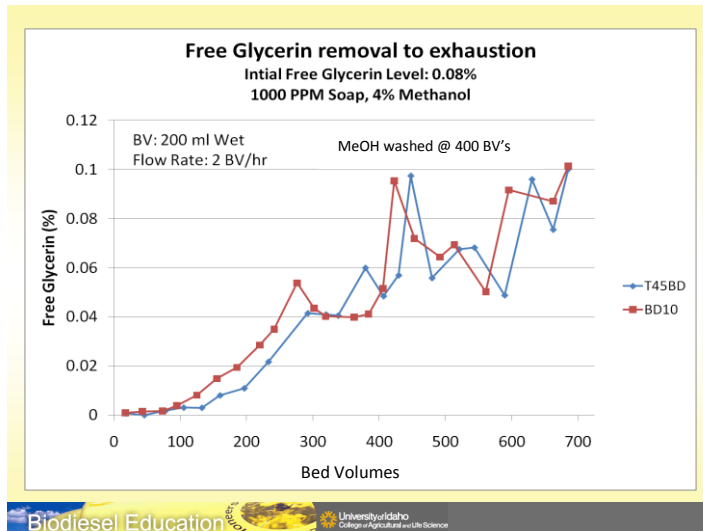
1000 ppm initial soap level

These two mechanisms, ion exchange and adsorption, are independent of each other as are their respective breakthrough points. Which breakthrough occurs first depends on the level of the impurities in the raw biodiesel. The glycerol breakthrough point can be measured by regularly determining the glycerol content in the treated biodiesel using either the ASTM D-6584 or the European EN14105 test methods. In addition to the glycerol breakthrough there will be an ionic breakthrough point where T45 BD or T45 BD Macro will be removing Na and K metal ions from the alkali catalyst used in the trans-esterification reaction, plus other metal ions such as S, P, Fe and Mg commonly found in the raw oils used as the starting feedstock. Regardless, ionically exhausted T45 BD or T45 BD Macro will still adsorb glycerol as effectively as fresh resin.



At customers where the glycerol breakthrough occurs prior to the ionic breakthrough, a regeneration procedure is recommended to recover the glycerol adsorption ability of Tulsion T45 BD or T45 BD Macro. It should be noted that with gellular resins a glycerin / soap “sheet” often forms on top of the resin bed and can be removed by conducting methanol regeneration.

This “sheet” will impair the flow dynamics of the raw biodiesel through the resin bed and may result in channeling and may also increase the pressure drop through the resin bed. The increased “delta P” will ultimately reduce effluent flow of the purified biodiesel and may facilitate early glycerol breakthrough. A number of installations have noted that the formation of the “glycerin-soap sheet” is either eliminated or significantly reduced in frequency when T45 BD Macro is used *in place of any gellular resin*. That said, a regeneration of the resin is possible with methanol.



The high polarity of the methanol enables the glycerol to desorb from Tulsion T45 BD or T45 BD Macro. After regeneration, T45 BD or T45 BD Macro will again be able to remove glycerol at a similar capacity of fresh resin. The regeneration procedure can be carried out multiple times however, the operating capacity will decrease with time. On a practical level, the T45 BD or T45 BD Macro resins can be regenerated 5-10 times before the product is completely ionically exhausted and must be replaced. In most installations, whether operating on virgin grain oils, reclaimed vegetable oils or animal fats the resins will need to be regenerated once 300 – 600 bed volumes of raw biodiesel fuel has been processed.

There are two (2) methods for applying methanol for regeneration of the bed:

Method 1:

- 1.1 Draining the biodiesel from the column.
- 1.2 Introduce 1 – 1.5 BV's methanol from the bottom such that all the T45 BD or T45 BD Macro is covered.
- 1.3 Re-circulate the methanol downflow for 4-6 hours.
- 1.4 Drain the methanol and send back to the methanol recovery unit to be used in subsequent trans-esterification reactions.
- 1.5 Introduce biodiesel from the bottom and allow 1-hour "conditioning" before restarting the service cycle.

Method 2:

- 2.1 Alternative to steps 1.2 through 1.4 above: Pass 4-6 BV's of methanol, down-flow through the column at one-half the service flow rate. This methanol can be captured once it exits the vessel and returned to the methanol distillation tower to be used in subsequent trans-esterification reactions.
- 2.2 Begin downflow introduction of raw biodiesel and begin next service cycle.

The advantage of these procedures is that there will be virtually no liquid waste since the methanol can be re-cycled and re-used.



CHEMICAL DIVISION
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TI/Methanol Regeneration Procedure /092009

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