

PURIFICATION OF POLYOLS

MAGNESOL® is the registered trademark for a special hydrated, synthetic, amorphous magnesium silicate possessing a porous structure and active surface. This unique structure imparts high performance selective and sorptive properties.

Purification by **MAGNESOL®** is widely recognized as the most effective way to produce high quality polyols. With high adsorptivity and excellent filtration characteristics, **MAGNESOL®** magnesium silicates are economical and easy to use.

The highly active surface of **MAGNESOL®** attracts sodium, potassium, and other metal ions as well as other polar compounds by chemisorption and holds them for removal from the process by filtration. The result is a high purity, odor-free, crystal clear, sparkling polyol. No other method or adsorbent achieves lower residual sodium and potassium levels than **MAGNESOL®**.

Differences in processing equipment and polyol chemistry require that each process be evaluated and optimized beginning with the following recommendations.

1. For polyols susceptible to oxidative degradation, maintain a nitrogen blanket throughout the entire process.
2. For typical 1000-3000 molecular weight polyols use a **MAGNESOL®** to catalyst ratio of 4-6:1 by weight. The higher the catalyst to polyol concentration, the lower the required ratio of **MAGNESOL®** to catalyst (see graph A).
3. Charge the **MAGNESOL®** to the treatment tank before any excess water is added. Limit exposure of polyol to air. If

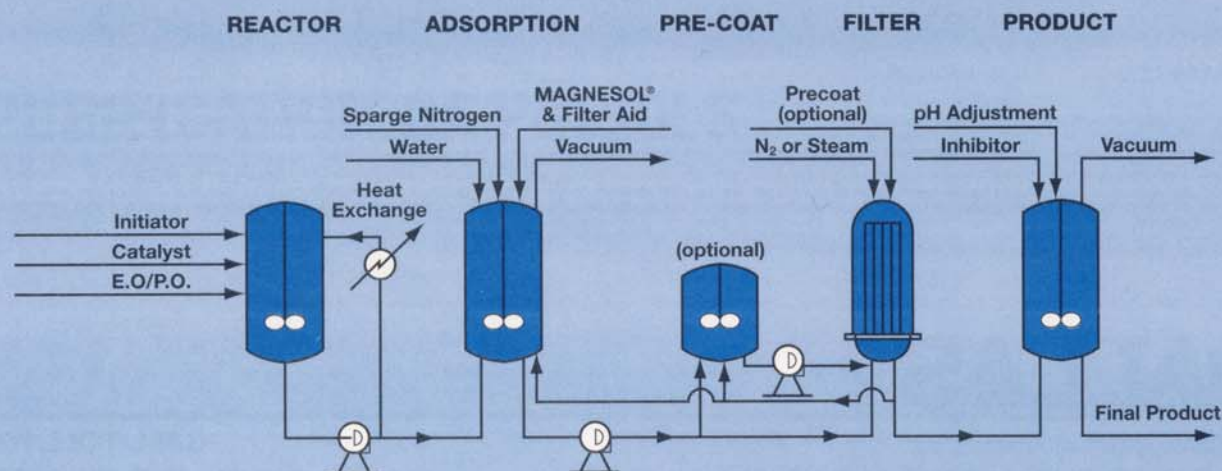
necessary, add filter aid along with the **MAGNESOL®** to maintain filter cake permeability.

The quantity and grade of filter aid used varies with the grade of **MAGNESOL®**, filter media, and viscosity of the polyol. Although some trial and error is involved in finding the best grade and proportion of filter aid, typically, the filter aid to **MAGNESOL®** ratio is 0.3:1 by weight for the high viscosity polyols down to 0.1:1 for low viscosity polyols.

4. Remove unreacted oxides from the polyol with a low boiler strip. Unreacted oxides may interfere with catalyst adsorption or react with free water to produce low molecular weight glycols.

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TYPICAL POLYOL PROCESS



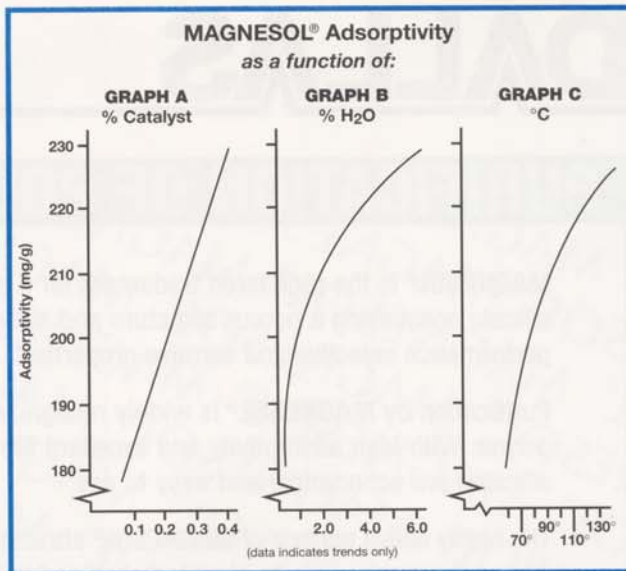
- Following stripping, add the proper amount of water to the treatment reactor prior to increasing the temperature. The **WET Method (Water Enhanced Treatment)** is effective for optimizing the use of **MAGNESOL®** in some polyols.

Water concentrations of 0.3% or greater in some polyols can significantly increase the performance of **MAGNESOL®**. The optimum water concentration can be determined in the laboratory or in pilot plant tests by construction of adsorption isotherms using methods simulating process conditions.

In general, the higher the water concentration, the lower the required ratio of **MAGNESOL®** to catalyst (see graph B). Water concentration is limited by the ability of the process to strip excess water from the polyol and the water specification of the final product. Once the optimum water concentration is determined, use the following method for calculating additional water to be added to the system:

- analyze the polyol for free water;
 - measure the free water in **MAGNESOL®** by drying for 2 hours at 105°C;
 - calculate additional required water by subtracting the sum of "a" and "b" above from the optimum water concentration.
- After water is added, gradually increase the temperature to 110°-130°C. The optimum treatment temperature will vary with each process. In general, the higher the treatment temperature, the greater the catalyst adsorption by **MAGNESOL®**. Consequently, this decreases the amount of **MAGNESOL®** needed (see graph C). Higher temperature is desirable as long as the polyol is not adversely affected and process safety criteria are not violated.
 - Treat the polyol for 2-6 hours at 110°-130°C with agitation sufficient to provide thorough mixing and suspension of **MAGNESOL®** in the polyol. Inclined blade turbine agitators operating at 60-80 rpm are well suited for the application.

Excess water may be stripped during the treatment cycle. However, if water stripping can be postponed until later in the process, **MAGNESOL®** will preferentially adsorb water over polyol and have the effect of improving yield by water displacement of polyol in the filter cake.



- Following treatment, transfer polyol to the filter feed tank. Horizontal or vertical pressure leaf filters with stainless steel mesh filter media are typically used. After filtration, blow the filter cake clear of polyol and store it for disposal in containers with an inert blanket or water cover to prevent autoignition.
- Adjust pH and add inhibitors to the final polyol as required. Perform a final water strip to bring product to water specification.

MAGNESOL® SYNTHETIC MAGNESIUM SILICATE

*The Dallas Group maintains fully equipped laboratory facilities in Jeffersonville, Indiana. Its staff of chemists is responsible not only for maintaining the highest standards of quality of **MAGNESOL®**, but also for conducting continuing research into the effects of adsorbent silicates on a variety of aqueous and non-aqueous systems. We welcome the opportunity to discuss your specific application and provide assistance in applying adsorbent technology to your process.*

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DALLAS

The Dallas Group of America, Inc.
374 Route 22 • P.O. Box 489 • Whitehouse, NJ 08888
(908) 534-7800 • Fax (908) 534-0084

**CALL TOLL FREE:
1-800-367-4188**