CONTROLLED PORE SIZE FLEXIBLE POLYURETHANE FOAM

Basic Methodology of Controlling Nucleation and Pore Size

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Basic Methodology of Controlled Pore Size Flexible Slab Stock Polyurethane Foam Manufacture

Abstract: Flexible polyurethane foams for technical applications with tight performance specifications manufactured for further processing (e.g.: chemical or thermal reticulation) require uniformly controlled pore size. Basic elements of pore size control are addressed. These elements are largely the relationship of processing parameters such as pressure, temperature, dissolved gas and mixing shear to resultant pore size. The effect of adjusting each of these control parameters is examined. An emphasis is placed on controlling dissolved gas level in the isocyanate stream and polyol stream. Examples of equipment configurations required to obtain good pore size control are discussed and illustrated with piping schematics and equipment diagrams.

Relationships of dissolved gas concentration in Toluene Di-isocyanate in storage and process facilities

Temperature affects the solubility of Nitrogen in TDI. Temperature and solubility are inversely related. i.e.: as temperature increases, solubility decreases and dissolved nitrogen is released from solution. As temperature decreases, solubility increases and Nitrogen will be drawn into the liquid at all liquid/gas interfaces.

Pressure affects the solubility of Nitrogen in TDI. Pressure and solubility are directly related, i.e.; as pressure increases, solubility increases and free Nitrogen is dissolved in the liquid at all liquid/gas interfaces. As pressure is reduced, dissolved Nitrogen is released at all locations in the liquid.

Shear affects the solubility of Nitrogen in TDI. Shear and solubility are inversely related. i.e.: As shear increases, the solubility of Nitrogen decreases. As shear is decreased the solubility of Nitrogen increases. It is understood that this phenomenon is related to 1.2 above. That is; shear creates localized pressure drop or pressure reduction.

What can be accomplished with this information?

Within the polyurethane foam reaction it is the CO_2 gas generation caused by the reaction between Isocyanate and water which needs uniformly distributed and sized nucleation sites to create undifferentiated cell size in polyurethane foam. It has been found that the control of N_2 gas dissolved in the isocyanate stream will control the nucleating sites in number and size via the control and manipulation of temperature, pressure and shear (the relationships in 1.1, 1.2 and 1.3 above).

There are three main elements within an isocyanate system which are used to adjust foam pore diameter or bubble size.

Maximizing, minimizing and control of dissolved gas concentration in the isocyanate stream

Control of mixing pressure at the mixing chamber

Control of isocyanate injection pressure

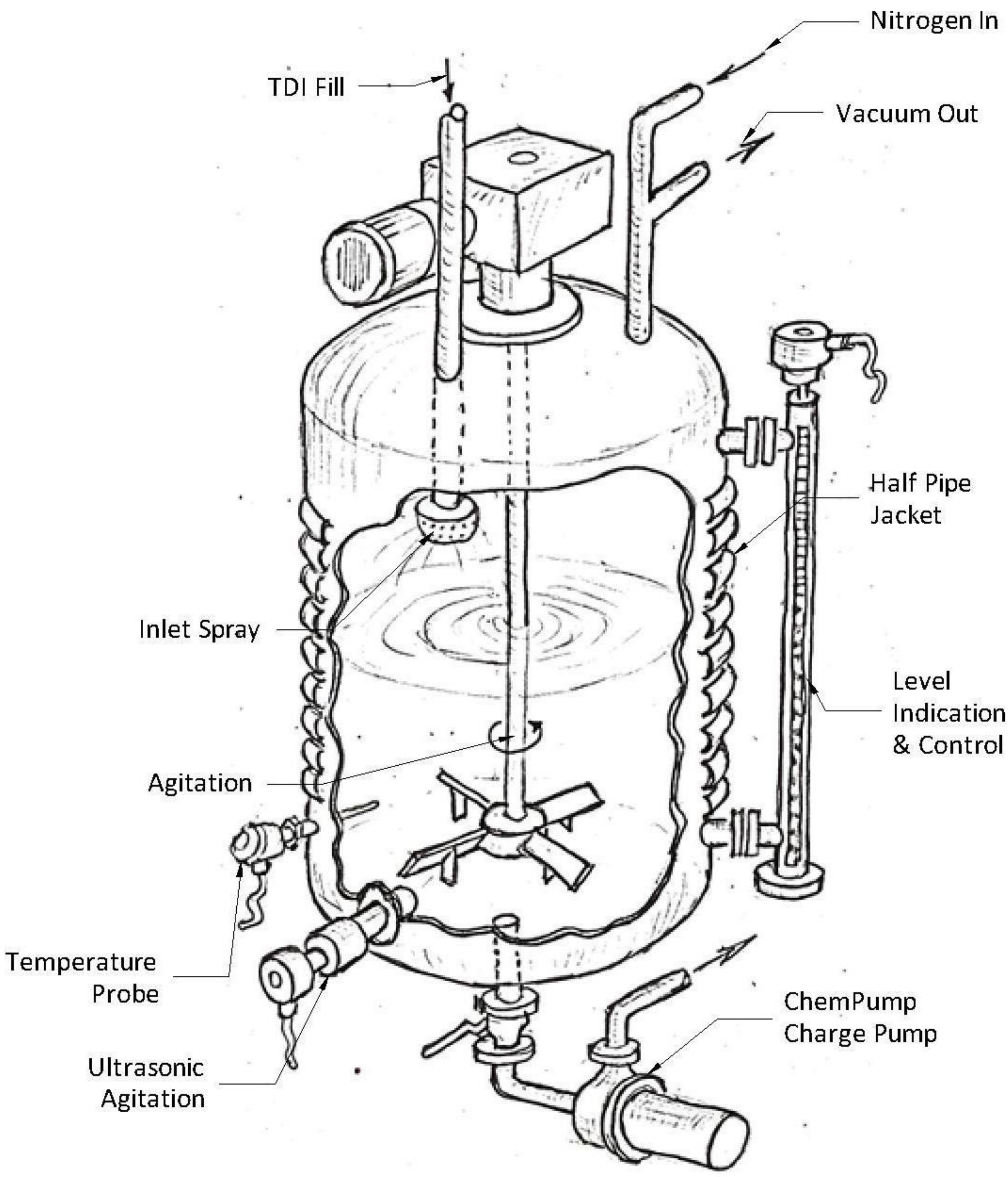


Figure 1: Dissolved Gas Conditioner Vessel

Control of dissolved gas content with the Gas Conditioner Vessel

Dissolved gas content can be maximized or minimized by batch process

The batch process consists of a code pressure (gas conditioner) vessel of suitable size. Pressure capabilities of the vessel should be 50 $^{\#}/in^2$ to full vacuum.

This gas conditioner should be equipped with agitation, level indication and control (i.e., level limits), pressure control, ASME pressure relief among other instrumentation.

Pressure control consists of both vacuum and pressure systems to extract dissolved Nitrogen under vaccum or dissolve additional Nitrogen under pressure.

Removing dissolved Nitrogen in this vessel is primarily accomplished by evacuating the headspace with a vacuum pump, with agitation at a controlled temperature. The Nitrogen removal can be facilitated by the addition of an ultrasonic transducer to create high shear.

Enriching the isocyanate with Nitrogen is accomplished by pressurizing the vessel with Nitrogen above atmospheric pressure, with agitation and temperature control.

Dissolved gas content can be maximized or minimized via a hybrid batch/continuous process.

This batch/continuous method also consists of a code pressure (gas conditioner) vessel of suitable size. Pressure capabilities of the vessel should be 50 $^{\#}/in^2$ to full vacuum.

This gas conditioner should be equipped with agitation, level indication; constant level is critical, pressure control, ASME pressure relief, among other instrumentation.

The batch/continuous process can be inserted into an isocyanate metering system on the suction side of the metering pump.

Removing dissolved Nitrogen in this vessel is primarily accomplished by evacuating the headspace with a vacuum pump, with agitation at a controlled temperature. The Nitrogen removal can be facilitated by the addition of an ultrasonic transducer to create a shearing environment.

Enriching the isocyanate with Nitrogen is accomplished by pressurizing the vessel with Nitrogen above atmospheric pressure, with agitation and temperature control.

The importance of agitation cannot be overstated. It is adamant to maintain uniform temperature and dissolved Nitrogen concentrations throughout the volume of liquid in the gas conditioner vessels.

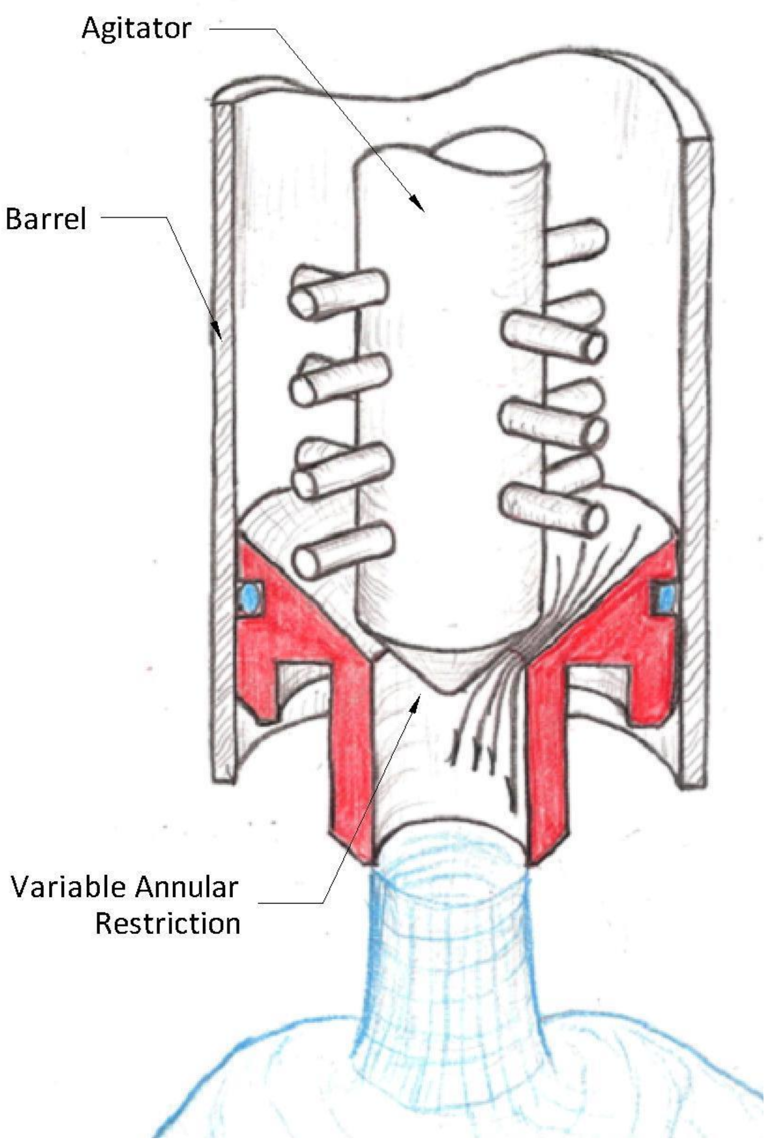
Temperature control is important throughout the conditioning process. Isocyanate must be fed into the conditioner at a correct and constant temperature. The conditioner vessel should be jacketed to maintain the proper temperature of the process fluid while it is in the vessel. If the conditioning vessel is not jacketed a second heat exchanger should be installed between the conditioner and the metering pump. This is done to remove any heat energy or "shaft work" imparted via circulation pumps, agitation, environment, etc. Temperature control methodology is critical but is a separate discussion. Thermal insulation should be applied to all temperature controlled surfaces.

Pressure control in the conditioning vessel is through vacuum system or pressure regulator. Constant pressure or vacuum is critical.

Control of dissolved gas concentration is the first level of pore size adjustment.

With the dissolved gas concentration minimized or maximized, the level of dissolved gas can be varied in the metering stream by blending the conditioned stream with a non-conditioned stream either on the suction side of a metering pump or with parallel streams through two separate metering pumps.

As the ratio of these two streams is adjusted: an increase in dissolved gas will increase the number or cells per linear unit and conversely a decrease in dissolved gas will decrease the number of cells per linear unit.



<u>Figure 2: Mixhead Pressure Control</u>

Control of mixing pressure is the second level of controlling pore size.

As mixing pressure is increased, the number of cells per linear unit decreases.

As mixing pressure is decreased, the number of cells per linear unit increases.

There are practical limits on this pressure adjustment as there is an undesired result from too much or not enough mixing pressure. Properly conditioned isocyanate will allow reasonable and workable mixing pressures to accomplish the required pore size adjustment.

Limitations of mixing pressure control: too much pressure causes streaking of irregular pore size and undo load on the agitator spindle shaft seal.

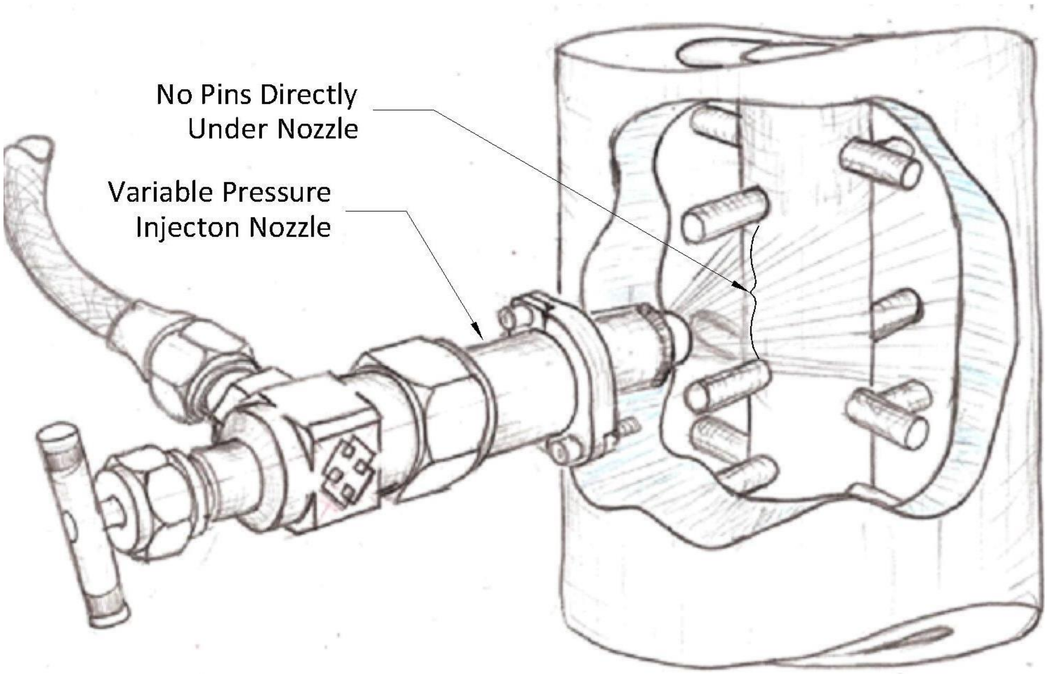


Figure 3: Isocyanate Injection via Variable pressure Nozzle

Control of isocyanate injection pressure is the final level of controlling pore size.

To properly release dissolved gas in the isocyanate stream the metered isocyanate must be forced through an impingement device such as an adjustable injection nozzle.

The combination of the abrupt pressure drop and high shear of the injection nozzle initiates the release of dissolved Nitrogen.

As injection pressure increases the number of cells per linear unit increases (finer).

As injection pressure decreases the number of cells per linear unit decreases (coarser).

There is no advantage using multiple injection nozzles vs. a single injection nozzle. Multiplication of injection nozzles does not change the cell count per linear unit. Nozzle multiplication does not change the amount of energy being imparted into the mix chamber via the isocyanate stream. It is impossible to balance multiple injection nozzles which are piped in parallel circuit; i.e., simultaneous opening and equally divided flow.

The position of injection must not have any agitator pins. Pins passing through the spray pattern create defects in uniformity.

The Formulation Record (Run Sheet) should include

1.1. Percentage blend of isocyanate streams; e.g., % non-conditioned vs. % conditioned.
1.2. Mixing pressure
1.3. Isocyanate injection pressure.

Summary

Entrained gas in the isocyanate stream or in any other stream is not the same as dissolved gas in the isocyanate stream. That is; Nitrogen injected is not the same as nitrogen dissolved. The isocyanate liquid has affinity for Nitrogen gas. The amount of Nitrogen that can be dissolved is directly proportional to the pressure of the solution and inversely proportional to the temperature of the solution. Dissolved nitrogen in a properly conditioned MDI or TDI stream, under pressure and uniformly distributed and released as the stream is metered into the mix chamber through an injection nozzle that creates a large pressure drop is far superior for controlling pore size and permeability than entrained, injected nitrogen. Controlling dissolved nitrogen in a "high pressure" isocyanate stream is a tool that is very valuable and should be understood and considered. This is a tool that will facilitate more even distribution of openness and cell size.