Technifax

CELLULOSE

DVT POLYPHASE REACTOR AND THE CELLULOSE STORY

Littleford Day, Inc. of Florence, Kentucky takes great pleasure in presenting its Carboxy Methyl Cellulose (CMC) Reactor Technology to the process industry.

Littleford's DVT Polyphase® Reactor. Polyphase® signifies in short: a single apparatus that is designed to handle materials of varying composition which may pass through phases of liquid, dough, granular, powder and has the combined operational features of pressure (250 PSIG at 100°F), vacuum (29.7" Hg) and effective heat transfer (heating or cooling).

Littleford has drawn upon its process technology and advanced Ploughshare® action to develop a system for the drying of these products. The Littleford system vacuum dries and cools the product all in the same vessel. The Littleford system allows the processor to accurately control the product temperature during drying to maintain the quality of the product.

The Reactor operates according to the proven fluidized bed mixing principle whereby the materials being reacted are maintained in a mechanically fluidized suspended state, thus permitting the reacting media, gas-solid or liquid-solid to achieve intimate contact with each other and the heat transfer surfaces. This will result in:

1. Controlled reactions through effective heat transfer.
2. Improved reaction rates.
3. Increased efficiencies of reaction.
4. Ability to complete an entire process in a single unit including vacuum drying.

These are distinct advantages over autoclaves or slow moving ribbon agitated vessels wherein the contact between the medium and the material is more by diffusion and is therefore slow, uncontrolled, and takes place at different times throughout the material.

The basic design of the Polyphase® Reactor is a stationary, jacketed horizontal cylinder built for internal pressure. It is top loaded and bottom discharged. The basic mixing impellers radiate from a horizontal shaft that is supported by outboard bearings. The basic mix action is supplemented by high speed blending choppers that are positioned between adjacent plows. The shaft seals can either be stuffing boxes or double faced, mechanical seals depending on the application.

An application that has proven itself to be ideally suited for the DVT Polyphase® Reactor is the preparation of cellulose derivatives and in particular Carboxy Methyl Cellulose (CMC). CMC is one of the most widely used synthetic water-soluble polymers. When dissolved in water at a 2% concentration it forms a clear viscous solution. This viscous characteristic has produced many applications for CMC as a thickening agent in the food, pharmaceutical, chemical and petroleum industries. You will find common household items such as syrups, chocolate powder drink mixes, jellies, detergents, soaps, toothpaste, shampoos and a host of others listing CMC as one of the ingredients. Even the gas and oil for automobiles depend on CMC. It is used in a special lubricating mud when drilling new oil wells. New applications are constantly being developed for this unique polymer.

In order to better understand what takes place in the manufacture of CMC, let's first take a look at what cellulose is.

Cellulose is the principle material in the walls of plants. As an example, cotton fiber is over 90% cellulose and wood fiber about 50% cellulose. In this form, cellulose is insoluble in most solvents and only partially soluble in a fairly strong alkaline solution. The insoluble portion is called alpha cellulose and is used in the preparation of cellulose derivatives.

DVT POLYPHASE® REACTOR

The chemical structure of cellulose is that of a glucose molecule minus a water molecule and is appropriately named anhydro (minus water) glucose. This gives the imperical formula of $(C_6H_{10}O_5)_n$. As with all polymers, the number of times that this anhydroglucose unit is repeated is quite large. For cotton, or linters as it is sometimes referred to, n = 3500 or a molecular wt. of 600,000. Wood pulp has n = 600—1000 or a molecular wt. of 100,000 to 175,000.

Now that we know what cellulose is, let's look at what's necessary to make it a water-soluble polymer. For this let's study more in detail the anhydroglucose molecule. As you will notice there are 3 (OH) groups on a molecule. These are the groups that must be changed to achieve solubility. This is commonly referred to as the degree of substitution or the average number of substitutions per anhydroglucose molecule. There are a maximum of 3 substitutions possible, but the most commonly used figure is 0.5—1.0.

The first step in the process is to uniformly add a solution of sodium hydroxide to the cellulose fibers. This will cause the fibers to swell and a sodium atom to randomly replace some of the hydrogen atoms of the OH groups on the molecule. This is one of the most critical parts of the whole reaction and each fiber must be coated to achieve the desired reaction and subsequent solubility. Also, the reaction must be cooled and kept oxygen free because cellulose degrades rapidly when alkalized.

The next step in the reaction is to add, and uniformly coat each fiber with a solution of monochloracetic acid. This will cause a random addition of carboxy methyl groups to attach themselves to spots where we previously attached sodium atoms. This reaction temperature is then raised to 50-70°C and allowed to remain there for a prescribed amount of time. The product CMC is then either dried or further processed depending on the end usage.

Now that we know what cellulose is and the general process necessary to make it a water-soluble polymer, let's look at the advantages for manufacturing it in a Littleford Polyphase® Reactor.

1. Since the Reactor is equipped with high-speed choppers, diced cellulose can be used to facilitate ease of material handling to the Reactor. The choppers quickly reduce the chips, when mixed with alcohol, to a fine fibrous mass.
2. Due to the effective fluidized mix action, which is supplemented by high speed blending choppers, a uniform dispersion of the reagents is achieved with the cellulose.
3. Since the reactor is tightly sealed and can be evacuated, the reaction can proceed in an inert atmosphere.
4. Reaction temperatures can be easily maintained due to the very effective heat transfer characteristics of the vessel.
5. Vacuum drying or recovery of reacting solvents can be accomplished in the same reactor vessel.
6. High efficiencies of reaction—standard processes are usually 60% efficiency; the improved DVT process yields 75-90% efficiency.
7. Fast reaction rates—typical cycles, including alcohol recovery, are approximately 6 hours.

This CMC reaction has been conducted several times in our Laboratory DVT Reactor. Diced cellulose and alcohol are charged to the Reactor. The Reactor is evacuated and cooling water is circulated on the jacket. Plows and chopper are started and allowed to mix for 20 minutes. At this time the sodium hydroxide solution is introduced via a spray nozzle mounted in the top cover. The addition time is adjusted to take approximately 30 minutes. After all of the solution is added the mix is permitted to mix for an additional 60 minutes (plows and chopper). At this point, while still continuing to cool the reaction, a solution of monochloro acetic acid is added via the spray nozzle. Again the addition time is adjusted to take approximately 30 minutes. This step of reaction is called the etherfication. At this point, sufficient heat is applied to the jacket to raise the reaction temperature to 50°C and the reaction is allowed to remain there for an additional 60 minutes. Due to the alcohol present in the reactor, a vapor pressure of approximately 20 PSIG develops. At the end of the etherification the pressure is released, vacuum applied and the alcohol recuperated. The reacted CMC is then discharged. The total reaction time is approximately 4 hours.

For a free brochure or a detailed discussion, contact us at:

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