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Small Tank Tetraphenylborate Process Vessel

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Abstract

One of the alternatives to processing the highly radioactive salt solutions in the Savannah River Site's Waste Tanks, is to precipitate the highly radioactive cesium with sodium tetraphenylborate, concentrate, and wash the precipitate slurry. This process is called the Small Tank Tetraphenylborate Precipitation process (STTP). In the STTP process, soluble ions of cesium, potassium and ammonium are precipitated as insoluble tetraphenylborate (TPB) salts. The resulting slurry, which now contains most of the radionuclides as insoluble solids, is filtered to concentrate the solids. After washing the solids to reduce the concentration of soluble sodium salts in the slurry, the precipitate is processed and incorporated into a glass form in the Defense Waste Processing Facility (DWPF).

To simplify this process, Precipitation Tank #1 is fed continuously with the volume of process water necessary to carry out the precipitation. The precipitate slurry continuously overflows to a second identical Precipitation Tank #2, which serves to increase the residence time for the precipitation process to 16- 24 hours. The rate determining step is based on the absorption of the plutonium, uranium, and strontium from the solutions. The slurry exits Precipitation Tank #2 as insoluble solids and is concentrated through a crossflow filter in the Concentrate tank. The concentrated slurry is then washed with a dilute caustic using a crossflow filter to reduce chemical concentrations to achieve acceptable levels needed for glass production. During this process excessive foaming was observed in tests conducted using actual Savannah River Site (SRS) radioactive waste. Foaming was also observed during the precipitation, concentration, and washing steps using simulates during testing performed at the Savannah River Technology Center (SRTC). As a result of these observations an investigation into finding suitable antifoam/defoam agents that could eliminate or mitigate foam generation was conducted by SRTC. The studies were conducted to develop an antifoam strategy that ensured the maximum and minimum limits needed to prevent foaming during processing of the solutions.

Introduction

Recently I was faced with an interesting challenge. It was to make a 30-liter vessel, complete with baffles, draft tube, and a large lid containing seven- internal threads for stirring and various instruments readings. Two identical 30-liter vessels shown in

Figure (1) were needed to simulate the precipitation process. One vessel was used to prepare precipitate slurry with antifoam where the other was used to prepare precipitate slurry without antifoam. Both vessels were designed with a sidearm sealed at the 21.5-liter level, to allow slurry from the Continuous Stirred Tank Reactor (CSTR) to overflow into a collection tank. Good mixing was achieved by using both a four-blade 3.4-inch diameter axial propeller and four baffles (0.94" at 90 degree to each other) in the tank. The solutions were pumped into the reaction vessels using calibrated peristaltic pumps and flexible tubing. Nitrogen Gas was used to purge the vessel, preventing a flammable mixture from accumulating in the vapor space. This was positioned above the liquid or foam level, and was controlled by a calibrated gas flow meter. The antifoam solution was metered into the (CSTR) using an automatic titrator at a concentration of 100ppmV. The agitator was maintained at 750 rpm during the duration of the experiment except for a brief portion of the test, were the agitator speed was increased to1000 rpm, to determine if the slurry would foam. At the end of a twelve-hour shift the feed pumps, agitator, and nitrogen purge were stopped until the next day.

After the experiments were completed, all the concentrated slurries (with or without antifoam) produced by the experiment were stored and allowed to settle. All the insoluble solids in the concentrate slurries, containing the antifoam agent, settled to the bottom of the storage container. This observation demonstrated results of the wetting agent on the physical properties of the precipitate slurry.

Process Vessel

The Glass Development Lab, of the Savannah River Technology Center, was asked to fabricate four identical 30-liter vessels as in Figure 1 for testing of the CSTR Precipitation System. The vessel design provided an airtight seal permitting a continuous nitrogen gas purge during testing. The first step in manufacturing the vessel was to decide the most practical way of sealing the baffles and draft tube into place. The customer requested the four baffles rest no more than 1/8th inch from the sidewalls and bottom of the cylindrical jar and measure approximately 16 inches overall. The baffles or plate glass was purchased in 4-foot lengths and measured one-inch wide x 3/8 inch thick. These were later cut to 16 inch sections and ground to provide rounded ends, using a belt-grinder equipped with a 240 grit diamond belt. The 7mm rods, used to connect to baffles, were cut 3-inces and sealed onto the baffle plates, using a Bethlehem bench burner. Each baffle was placed into a hot oven, after sealing the top and bottom halves, to avoid unnecessary breakage. The draft tube was made from 130mm standard wall tubing measuring six-inches overall. This was used to represent the surface area consumed by the cooling coil in the simulate storage tanks.

To begin assembly of the inner unit, cut the 130mm tube six inches overall and mark 1-½ inches from the top and bottom of the draft tube, using a chalk marker. The draft tube was then secured onto a holder using Waletex High Temp Glass Tape. Next, seal all eight of the 7mm rods one at a time, using the marks as a guide and anneal. To obtain the proper height inside the vessel, a spacer was placed under the draft tube and held in place using a smaller inner tube. This was also extremely helpful when aligning the baffle and added stability to the unit. To provide a flat sealing surface, 7mm rods were attached to the top and bottom of the baffles. The top rods were bent 90 degrees and cut 1/8th inch from the outside edge of the baffles. The bottom rods was bent downward 180 degrees from the previous bend and was cut approximately 1/8th inch, measuring from the end of the baffle Figure 2. Once all of the baffles were sealed, the entire unit resembling a spaceship, was returned to the annealing oven for stress removal.

The next step is to secure the jar into the lathe and run the quartz-annealing oven to 850 degrees centigrade. This will allow the temperature of the oven to return to the desired 565 degrees after completing the top or bottom section and placing into the preheated oven. I chose this temperature at random and it worked great. I used a Litton EE lathe during the assembly of the jar equipped with a Universal Planetary three-jaw chuck with twelve-inch extension arms. To provide ample heating a 12 fire 7-jet swivel lathe burner was supplied with 15psi of oxygen & hydrogen. Kevlar gloves, sleeves, and a reflective face shield were used as protection equipment, during the assembly of the vessel.

Place the baffle unit into the jar, slowly and carefully secure the jar using the twelve-inch extensions. Preheat the bottom section for approximately fifteen minutes. Next, valve off the gas and air burner and begin heating using the lathe burner, with the oxygen and hydrogen mixture. Heat the bottom of the jar until it glows a pail pink color. Reduce the intensity of the fires while sealing the four rods into place, using a Victor hand torch. Once all of the rods are sealed, raise the oven hood by pressing the remote switch in the upward position. Once the oven is opened to the highest point, extinguish the lathe burners and release the vessel from the lathe chucks and place in the annealing oven. The temperature in the oven will continue to fall once the vessel is placed inside and the hood lowered. Watch the oven closely until it reaches 565 centigrade and allow the vessel to soak at this temperature for around 45 minutes. Switch the oven to the off position and allow the oven to ramp down slowly until room temperature is reached.

To assemble the top section, the vessel is secured into the headstock side of the lathe, use the twelve-inch extension arms as before. Place the 200mm Duran o-ring flat ground flange into the Universal Planetary three-jaw chuck on the tailstock side of the lathe, equipped with graphite spools. As before, ramp the quartz oven to 850 degrees centigrade and place on hold. Preheat both sections at once for a minimum of fifteen minutes before extinguishing the gas and air burner. Start by shaping the o-ring flange, using the lathe burners with the oxygen and hydrogen mix, to a 45-degree angle. Roll the top close to the vessel during this operation to permit the radiate heat from the burners to keep the top as hot as possible. Next, shape the vessel in the same manor. Position the o-ring flange so that is almost touching the vessel and heat until both edges have begun to sag. Lightly touch the two together allowing the lathe burner to continuously heat the seal as you slowly work them together. This allows the vessel and the flange to seal from the inside first, thus resulting in a complete seal. Once this is done, reduce the intensity of the burners and seal the rods into place. Alternate between sealing a rod and heating the vessel to assure the proper temperature is maintained. After sealing all four of the baffle rods, pull a hole in the vessel above the 21.5-liter mark. Seal the # 15 Ace thread at a 75 degree angle and reheat the vessel before placing in the annealing oven using the same procedure, allowing the completed vessel to soak for forty-five minutes at 565 centigrade.

Vessel Top

To begin assembly of the kettle top, collect all of the necessary fittings to be used in manufacturing the lid: 1) 6-each # 15 internal threads, 2) 3- each # 7 internal threads. Always plan for breakage during assembly by prepping one extra of each fitting. This may save you from scurrying for an extra fitting during assembly and possibly prevent a catastrophic break in the flange top. Pull a slight taper on each fitting and place on a heated ceramic hot plate. Next, take a chalk marker and draw the location of the fittings to be sealed on the kettle top. Secure the kettle top into the headstock, using the Universal Planetary three-jaw chuck, supplied with graphite spools. Begin heating the top, using the gas and air Bunsen burner for approximately fifteen minutes. Next, valve off the burner and position the 8-fire, seven-jet lathe burner toward the outside edge of the dome top. Begin pulling holes, the appropriate size for the fitting, in the dome top using a Victor hand torch. Once the first hole is pulled, heat the top portion of the dome until it becomes a rose color. Alternate between reheating the top and pulling the holes until all of the holes made. Seal the center thread first, to minimize the risk of warping and cracking when attaching the other fittings during assembly. Continue with same heating sequence as before, by heating the top after each seal is made. This should keep the top at a constant temperature and prevent hot spots or excessive strain accumulation in the lid. Once all of the fittings are sealed and aligned, place the completed unit into a hot oven and anneal at 565 centigrade for forty-five minutes.

Summary

The completed laboratory scale units performed very well during testing. This test verified the abilities of the anti-foam agent when used during the STTP process precipitation, concentration, and wash steps, using the KTPB slurry. The vessels enabled researchers to observe the reactions of the solutions during normal and extreme agitation, with and without the antifoam agent. One of the most important conclusions reached was the importance of minimizing the introduction of gas into the slurry during processing whenever possible.

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Figure 1

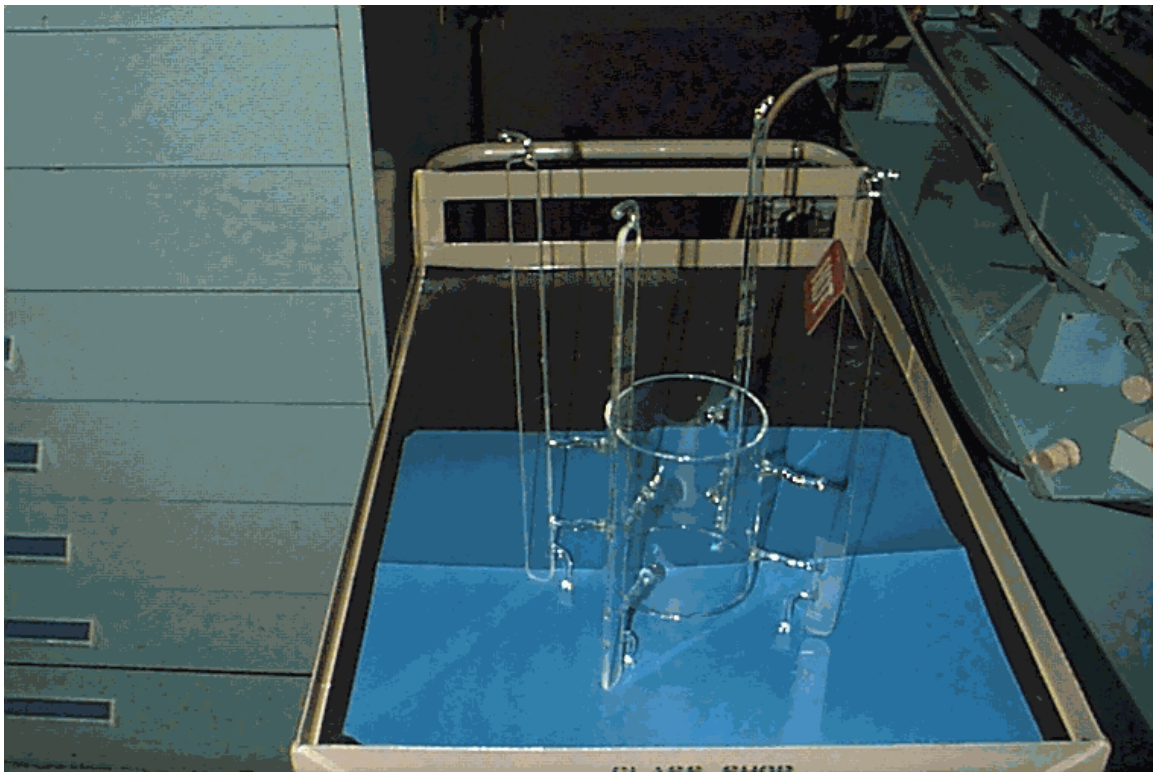

Figure 2