# Purpose:

To define the process used to manufacture ceramic powder batches.

# Responsibilities:

Crystals Department Engineering is responsible for maintaining this procedure.

Crystals Department Management and Technicians are responsible for carrying out this procedure.

# Associated Documents:

ISO 9001, QAM, AS9100

# Safety Precautions

In certain operations in the following procedure, the operator will come in contact with the following situations that require due caution:

Warm to hot drying ovens (80°C - 130°C)

Hot Plates

Furnaces

Extremely Fine/Toxic Powders

Common sense and good laboratory procedures should always be used.

In case of any accident, inform your supervisor immediately.

## Working with Furnaces

Any furnace and contents should be allowed to cool below 200°C before anything is inserted or removed from the furnace.

When inserting or removing anything from a furnace that is warmer than 65°C (150°F), use a set of gloves, mitts, or other utensils that allow the operator to work without risk of burns. Furnaces with exposed heating elements must be turned OFF by the operator prior to reaching into the furnace.

## Handling Fine and/or Toxic Powders

When working with fine powders, it is good practice to take the appropriate steps to avoid inhaling or ingesting the dust generated by the handling process. Two methods used are the use of a dust collection bench as the primary work area when handling the powders, and the wearing of the appropriate dust masks. It is also good practice to wear lab coats and gloves when handling the powders. It is imperative that the operator washes his /her hands thoroughly before eating, drinking, or smoking in order to prevent ingesting any dust. Very fine dust is found mostly in operations dealing with raw materials, dried, ball-milled powders, and very fine, granulated powders.

The most toxic powder used in this process is orange-colored PbO, lead oxide, also known as Litharge. Long-term exposure to lead and lead compounds, such as lead oxide, can result in a buildup of lead in the body, due to absorption of lead into the bloodstream through inhaling dust or fumes, or ingesting by mouth. While lead oxide should be handled in the same way as any other fine powder, there are specific dust mask types which are approved by OSHA for working with lead. The 3M Model 8233 is one of these dust masks, and is available for operators to wear on a voluntary basis when handling lead-based powders. (Note: The primary prevention control for lead exposure is the use of the dust bench when handling lead-based powders.) Again, thoroughly washing the hands after working with lead-based powders is required to avoid ingesting lead.

Do not eat or drink in the area where powders are stored, handled or processed.

# Laboratory Practice

The object of the batching operation is to produce high quality, high purity ceramic powders. The three keys to this are cleanliness, raw material handling, and proper use of the equipment. If there are ever any questions about the correct method of doing something, always ask your supervisor.

## Cleanliness

Keep work surfaces clean and free of materials. Clean after each process and before you start the next operation. This includes bench tops, balances, sinks, and shelves. Vacuum the floor as needed. Do not use compressed air or a dust broom to clean powder off of equipment or floors. Always use the HEPA vacuum cleaner or wet mop and wet wipes to clean.

Dispose of any lead-contaminated paper towels in the appropriate hazardous waste barrel. Discard any lead-containing scrap powder in these barrels also.

The sink in the batch room drains to a settling tank and then to a wastewater recovery / recirculation system. It does not drain to the sewer due to the lead-containing powder waste. Always use this sink for cleaning utensils from the batching process.

Keep utensils clean. When finished with an operation, wash your dishes so they will be clean and dry the next time you need them. In general. clean utensils with liquinox and tap water, and rinse with Dl water. Dry with a hand towel or Kimwipe, or in the case of screens, place in the batch room oven to dry. Store all utensils in a cabinet so that dust does not settle on them. When washing utensils, always final rinse with Dl water and blot dry with a Kimwipe. Before using utensils, check to see that they are clean.

## Raw Material Supply

Always maintain a sufficient supply of tested raw materials on hand to last at least six months. If the raw material level looks low, tell your supervisor.

Before a new raw material is used for production batches, a test batch must be made to determine if the new raw material makes acceptable batches. CR1027 describes the procedure used to qualify batches for release to production.

## Proper Use of Equipment

Mill jars should only be used for one operation and one material. This means that for calcined materials, 2 mill jars are needed; one for first (precalcine) mill, and one for second (post-calcine) mill. Mill jars should be charged with zirconia grinding media, and should be approximately one-half full of grinding media. When new media is added to a mill jar, the mill jar should be half filled with DI water and rolled for 60 minutes to clean the new media. Dump the water and rinse the mill out with Dl water.

Only screens that are in good condition should be used. If they are handled with care so that the rims and edges are not bent, the screens will come apart without prying. Avoid placing too much material on a screen at one time. The finer the mesh size of the screen, the smaller the amount of material that can be screened at one time. Make sure the screens and material are dry before use.

## Process Traceability

Each ceramic batch to be produced will have a Ceramic Powder Production Traveler (CR011) that will serve as a router for the process. A unique version of CR011 is generated by engineering for each recipe and will specify the raw materials and amounts to be used, the sequence of steps, and other pertinent process details for that batch. Operators will use the traveler as a record for that batch by signing off steps as the operations are completed, recording pertinent information, and noting observations as required.

# Procedures

1. **Weighing**

NOTE: These batches are high-grade piezoelectric ceramic compositions that are extremely sensitive to the precise ratio of raw materials, impurities in the raw materials, and impurities added in the batching process. Extreme care must be taken to maintain the quality of these materials.

* 1. Equipment
* Electronic Scale - capable of 5 kg +/- 0.1 gm
* Electronic Scale - capable of 200 gm +/- 0.01 gm
* Various Weighing Pans
* Small plastic scoops for raw materials
* 10 liter, wide-mouth nalgene carboys, with media
* Size 1 urethane lined mill jars, with media
* 12” Funnel
* Material/batch-specific brushes
  1. Use the appropriate electronic balance to weigh all materials. Make sure the electronic scales are level and properly “zeroed”. Place the appropriate weighing pan on the scale and “tare” the scale to zero before weighing each material.
  2. As the various raw materials are taken from their storage containers, make entries on CR011 for the raw material lot number.
  3. Weigh the correct number of grams for each raw material listed on CR011. This step is extremely critical. Repeat this process for all the materials defined by CR011.
  4. Refer to the Powder Production Traveler for the next step. Either blend the powder in the V-Blender or proceed to the milling steps accordingly.
  5. For V-blender Processing: Transfer each of the pre-weighed materials from the weighing pans to the V-Blender. Set the V-blender shell drive time to 5:15. Set the agitator bar time to 5:00. Afterward, remove the V-blender shell and move it to the down draft hood. Transfer the the material to plastic storage containers for weighing.

1. **Dry Milling**
   1. Equipment

* Mill Roller
* 10 liter, wide-mouth nalgene carboys, with media
* Size 1 urethane lined mill jars, with media
* 10-Mesh Screen and Screen Pan
* Plastic Storage Container with Lid
  1. Transfer the powder into the appropriate mill jar using the 12” funnel to assure all the material is transferred to the mill. Use the small weighing brush for that material to brush all the material into the funnel.
  2. Secure the lid to the mill jar.
  3. If the mill roller is running, turn it off, then place the mill jar on the mill roller.
  4. Turn on the mill roller and note the time. Check the speed of the rolling mill jar. Adjust to about 60 rpm as necessary.
  5. Allow the mill to roll for the time indicated on the Batch Control Sheet.
  6. During the milling cycle, occasionally stop the mill roller, stand up the carboy, and tap with a rubber mallet to dislodge any material from the neck of the carboy. This is not necessary if using Size 1 Mill Jars.
  7. After the time specified on CR011, turn off the mill roller, remove the carboys or mill jars, and turn roller back on if there is anything else on the rollers.
  8. Assemble a clean 10-mesh screen and pan.
  9. Slowly dump the milled material onto the screen stack. The 10-mesh screen will separate the powder from the milling media.
  10. Put the media back into the mill and screw on the mill lid. The milled powder should be placed in a plastic storage container and the lid attached.
  11. Label the plastic storage container with the batch number.

1. **Calcining**
   1. Equipment

* 4” diameter, 6” high, alumina crucibles with lids
* 3-1/2” diameter 6” high, Ca-stabilized zirconia crucibles
* Calcine Furnace
* Stainless Steel Drying Pan
* Mortar & Pestle
* 30-Mesh Screen and Screen Pan
  1. Transfer the material to be calcined from the plastic storage container into the appropriate calcine crucibles defined by the Batch Control Sheet using the 12” funnel as an aid. The crucibles should be approximately 80% full. Put a lid on the crucibles when calcining PZT.
  2. Place the loaded calcine crucibles in the appropriate calcine furnace. Set the crucibles so they do not touch the furnace wall.
  3. If main power is off, turn it on at this time. Refer to the controller manual to select the calcine profile program defined on the batch control sheet.
  4. If the furnace has an over-temperature protection circuit, check the OVERTEMP/RESET switch to confirm that the indicating lamp is off.
  5. Open the Nabertherm software program on the batch room PC.
  6. Select the appropriate furnace. Label the profile with the batch number being calcined and the date.
  7. Close the furnace door and start the calcine profile via the ControlTherm software.
  8. After the furnace has cooled to below 150ºC, the crucibles can be removed from the calcine furnace.
  9. Review the calcine profile recorded for the proper maximum temperature and time at that temperature.
  10. Inspect the calcined material for the appropriate color and texture. Call your Supervisor or Engineering if either the recorded profile or the material shows any deviations from normal.

NOTE: Properly calcined PZT material is cream colored. If the material in the center of the calcining crucible is still orange, the batch should be recalcined.

* 1. If the material and profile look normal, remove the calcined material from the calcine crucible and place the calcined material in a clean drying pan.
  2. Refer to the Powder Production Traveler for the next step. Either grind the powder in a mortar and pestle or process in the V-Blender accordingly.
  3. For Mortar and Pestle Processing: Transfer a manageable portion of the calcined material from the drying pan to the mortar. Use the mortar and pestle to break apart the large lumps. Process the broken-up material through a clean 30 mesh screen. The material that does not pass through the screen should be emptied into the mortar to be broken up. The material that does pass through the screen should be emptied into a plastic storage container. Continue to break apart and screen the material until it all passes through the 30 mesh screen and is loaded into the plastic storage container.
  4. For V-Blender Processing: Transfer the calcined material from the drying pan to the V-Blender. Set the V-blender shell drive time to 5:15. Set the agitator bar time to 5:00. Remove the V-blender shell and move it to the down draft hood. Transfer the the material to plastic storage containers.
  5. Transfer the batch from the plastic storage container to the weighing pan and determine the weight of the batch.
  6. Transfer the batch from the weighing pan back to the plastic storage container and label the container with the batch number.

1. **Wet Milling**
   1. Equipment

* Electronic Scale - capable of 200 gm +/- 0.01 gm
* Small plastic weighing pans
* Small plastic scoops for raw materials
* 10 liter, wide-mouth nalgene carboys, with media
* Size 1 urethane lined mill jars, with media
* 12” diameter funnel
* Material/batch-specific brushes
* Various Beakers
* Mill Roller
  1. If CR011 calls for solid materials to be added, use the appropriate electronic scale and weighing dish to weigh the material. Make sure the electronic scale is level and properly “zeroed”. “Tare” the scale to zero before weighing each material.
  2. Transfer the contents of the weighing pan into the appropriate mill jar using the 12” funnel to assure all the material is transferred to the mill. Use the small weighing brush for that material to brush all the material into the funnel.
  3. Use the 1000 ml Tripour beaker and add the amount of liquid specified on CR011 to the mill.
  4. Secure the solid lid on mill jar.
  5. If the mill roller is running, turn it off.
  6. Place the mill on the mill roller.
  7. Turn on the mill roller and note the time.
  8. Allow the mill to roll for the time indicated on CR011.
  9. After the specified time, turn off the mill roller, remove the mill, and turn the roller back on if there are any other mills on the rollers.

1. **Vacuum Filtering and Drying**
   1. Equipment

* 4000 ml Filtering Flask
* #6 Coors Porcelain Buchner Funnel
* Whatman #5 filter paper
* Vacuum Pump and Hose
* perforated carboy mill lid
* perforated SS Size 1 discharge lid
* aluminum foil
* plastic spatula
* Stainless Steel Drying Pan
* Batch drying oven
* 12” diameter funnel
* Mortar & Pestle
* 30-Mesh Screen
* Screen Pan
* Plastic Container with Lid
  1. Make sure the filter flask is empty.
  2. Set the Buchner funnel on the filter flask.
  3. Place a filter paper circle (Whatman #5) in the Buchner funnel.
  4. Wet the filter paper with Dl water while making sure it lays flat and smooth.
  5. Turn on vacuum pump and make sure that the paper forms a good seal.
  6. Remove the mill lid from the mill, and replace with the perforated lid for straining the media from the batch.
  7. With the vacuum pump running, turn the mill over slowly and pour the batch from the mill into the Buchner funnel.
  8. After the majority of the material has been poured from the mill, add a small amount of DI water to the mill. Agitate the mill, then dump the material into the funnel. Repeat this process as necessary to recover as much of the batch as possible from the mill. Be careful not to use too much water, as the filtering funnel may overflow, or the water level in the vacuum flask will become too high and water will be drawn into the vacuum pump.
  9. Cover the funnel with aluminum foil. Allow the vacuum pump to pull the water from the batch. As the last of the water is removed, cracks will form or the cake will pull away from the sides of the funnel. This generally takes about 90 minutes.
  10. Five to ten minutes after the cracks form, when virtually no water is coming out of the Buchner funnel, turn off the vacuum pump. The cake can be removed from the Buchner funnel at this time, but it will be easier to remove if allowed to dry overnight. Run a spatula around the Buchner funnel between the cake and the funnel, place a drying pan over the Buchner funnel, and flip the two simultaneously so the cake falls into the drying pan.
  11. Remove the filter paper from the filter cake and use a spatula to remove excess material remaining on the filter paper.

NOTE: Removal of the filter paper at this time is critical to prevent defects in the fired ceramics.

* 1. Cover the pan with aluminum foil, and label with the batch number. Poke some small holes in the foil. Place the drying pan in an 80°C drying oven for a minimum of 16 hours or until dry.
  2. Clean the vacuum filtering area, Buchner funnel, and mill. Empty the water from the filter flask.
  3. Remove the dried batch from the drying oven. Obtain a funnel, mortar and pestle, 30 mesh screen, pan, and plastic storage container with lid.
  4. Use the mortar and pestle to break apart the large lumps into a manageable size. Generally, it’s easiest to work with part of the batch at a time.
  5. Screen the broken-up material through a 30 mesh screen. Screens are meant for shaking only. The material that does pass through the screen should be poured through the funnel into the plastic storage container.
  6. Continue to break apart and screen the cake until all the material passes through the 30 mesh screen and is transferred into the plastic storage container.
  7. Label the plastic storage container with the batch number.

1. **Granulating**
   1. Drying
      1. Equipment

* Drying Oven
* Large electronic balance
* Stainless Steel Drying Pan
* Plastic storage container containing batch material
  + 1. Transfer the batch to be granulated from the plastic storage container to a clean drying pan and place in the drying oven at 80ºC for a minimum of 4 hours.
    2. Tare the large balance to “zero” with the Tripour beaker on the balance.
    3. Transfer the dried material from the drying pan to the Tripour beaker.
    4. Add binder using either Section 6.2 or Section 6.3, as specified on CR011.
  1. **Binder Addition Using Hobart**
     1. Equipment
* Hobart mixer and pan
* 8” diameter, 30 mesh stainless steel screen
* 8” diameter, screen pan
* #5 size Coors Porcelain Mortar and pestle
* Various Beakers
* Stainless Steel Drying Pan
* Drying Oven
  + 1. Determine the weight of the material in the Tripour beaker and, based on this weight, the amount of binder that should be added to the batch. The number of grams of binder that should be added is calculated using the following formula:

Binder (gm) = (Wt of material (g) / 100) x % binder from CR011

* + 1. Transfer the material from the tripour beaker to the Hobart mixing pan and set the mixing pan on the Hobart mixer.
    2. Using a glass beaker, weigh the amount of binder determined in 6.2.2.
    3. Insert the beater for the Hobart mixer, set the mixer on low speed, and turn on.

NOTE: The following steps using the Hobart mixer should be done in the shortest period of time possible to prevent the ceramic powders from abrading steel from the mixer into the batch.

* + 1. Slowly raise the mixing bowl of material so that the mixer starts to slowly stir the material. Slowly add the binder to the material, pouring in about 20 gm every minute. Tap the side of the bowl periodically to redistribute the material. Continue to mix until the entire batch has the same consistency, usually about 20 minutes.
    2. Stop the mixer, lower the bowl, and transfer the batch from the mixer pan to the drying pan.
    3. Bang the mixer pan on the work surface to loosen any material that has accumulated on the sides of the pan. Do not scrape the material from the sides as this can contaminate the batch. Transfer this loosened material to the drying pan.

NOTE: If the batch is too wet to screen after the binder is added, it may be placed in the drying oven until it dries to a workable consistency.

* + 1. Using the pestle, crush the granules together and against the sides of the mortar.

NOTE: Shear motion between the mortar and pestle is used to disperse the binder evenly and force it to wet the batch.

* + 1. Use the 30 mesh screen and pan for the particular material. Start by transferring a couple of small scoops of material onto the screen. Vigorously, shake the screen horizontally with some vertical agitation. Transfer the material that passes through the screen onto a drying pan. Transfer the material that does not pass through the screen into the mortar. Use the pestle to gently break up the lumps of material until all the material passes through the 30 mesh screen.
    2. Repeat this process until all the material passes through the 30 mesh screen and is in the drying pan.
    3. Place the drying pan in an 80°C drying oven for a minimum of 16 hours and a maximum of 24 hours. If the batch is still wet, inform your supervisor.
  1. **Binder Addition Using V-Blender**
     1. Materials:
* Patterson Kelly V-Blender
* 8oz Funnel with attached tube
* 2000ml beaker
* Binder #3
* Stainless Steel Drying Pans (4)
* large electronic balance
* rubber mallet
  + 1. If necessary, clean the V-blender. Remove the intensifier bar, shell, and liquid feed tube and clean as necessary. The intensifier bar may need to be disassembled for cleaning.
    2. Check that the total shim height is 0.060+/-0.005 inches on the intensifier bar.
    3. After cleaning, load the weighed material into theV-Blender in the down draft hood, install the shell onto the shaft, reassemble and install the intensifier bar, making sure it is fully seated onto the shaft. The lock screw will need to be tightened with the appropriate wrench:
* Shell Bolt – 14mm Wrench
* Intensifier Bolt – 7/16” Nut Driver or Wrench
  + 1. Install the feed tube, and attach the tubing and funnel.
    2. Tightly secure both solid covers.
    3. To run the blender, the powder must be the same height as the top of the intensifier bar. To check the height, load the powder and run the V-Blender for one minute, remove the solid cover and observe the height. If the height is any lower than the top of the intensifier bar, inform your supervisor.
    4. Calculate the amount of binder that should be added to the batch by using the following formula:

Binder (gm) = (Wt of material (g) / 100) x % binder from CR011

* + 1. Using room temperature binder, weigh out the appropriate amount of binder as calculated above.
    2. Set the shell timer for 17 minutes, set the switch to auto, and turn on the shell. Allow the shell to turn for 30 seconds prior to adding the binder.
    3. Set the intensifier timer for 12 minutes, set the switch to auto, and turn on the intensifier bar. Begin the binder addition process by filling the funnel with binder. Keep the funnel elevated as high as possible.

NOTE: It is important to add the binder as quickly as possible so the machine does not heat up. Some things that will help add the binder as quickly as possible are: using warm binder, keeping the funnel elevated, and keeping the funnel topped off.

* + 1. Continue to add binder to the funnel until all of the binder has been used. After all the binder has been added, allow the shell and intensifier bar to run for one more minute.
    2. After the blender has stopped, set the shell switch to jog, and jog the shell to the upright position. Use the rubber mallet to tap the blender shell and solid cap to loosen any powder stuck to the sides. Remove the front solid cap. Inspect the powder for a sand-like consistency. If the powder is too fine, run the shell for 1 minute intervals until the desired consistency is achieved. Do not let the shell run continuously. It will continue to granulate into larger pieces as it runs.
    3. Remove both covers from the shell. Use a plastic policeman to lightly scrape the surface of the covers into the shell. Carefully remove the intensifier bar from inside the shell. Use a plastic policeman to lightly scrape off any material into the shell. Remove the shell locking bolt. Remove the shell and transfer it to the down draft hood. Empty the shell into 4 separate stainless steel pans somewhat evenly.
    4. Cover the pans with foil and label with the batch number. Poke several holes in the foil, and place the pans in the oven to dry for 30-60 minutes.
    5. While the powder is drying, disassemble the blender and clean the shell, intensifier bar and feed tube.
    6. Use the 30 mesh screen and pan for the particular material. Start by transferring a couple of small scoops of material onto the screen. Vigorously, shake the screen horizontally with some vertical agitation. Transfer the material that passes through the screen onto a drying pan. Transfer the material that does not pass through the screen into the mortar. Use the pestle to gently break up the larger lumps until they pass through the 30 mesh screen.
    7. Repeat this process until all the material passes through the 30 mesh screen and is in drying pans. Cover the pans with aluminum foil, label with the batch, and poke some holes in the foil.
    8. Place the drying pans in an 80°C drying oven for a minimum of 16 hours and a maximum of 24 hours. If the batch is still wet, inform your supervisor.

1. **Screening**
   1. Equipment

* 8” diameter, xx mesh stainless steel screen (xx from CR011)
* 8” diameter, xx mesh stainless steel screen (xx from CR011)
* 8” diameter, screen lid
* 8” diameter, screen pan
* Material/batch-specific brushes
* 1 gallon, plastic storage containers
* large electronic balance
* appropriate weighing pans
* plastic material scoop
* Stainless Steel Drying Pan
  1. Obtain a clean batch storage container for the material type needed and label the container with the batch number, screened size, and date. Label the container tare weight also for future reference.
  2. Remove the dried batch from the drying oven.
  3. Assemble the appropriate stack of screens as detailed on the batch control sheet plus a bottom pan for that material.
  4. Place the weighing pan on the large electronic balance and “tare” to zero.
  5. Transfer a couple of small scoops of material onto the top screen in the stack.
  6. Shake the screen stack vigorously, horizontally with some vertical agitation. Screens are meant for shaking only. Agitate until as much of the material as possible has passed through the top screen.
  7. Remove the top screen from the stack. Empty the material that does not pass through the top screen into the mini milling jar. This is the course material.
  8. Continue to agitate the screen stack by shaking the material from one side of the screen to the other. Occasionally, tap the stack lightly against the table top to shake out the granules that are trapped in the wire mesh. Agitate until the fines have passed through the screen.
  9. Remove the top screen from the screen pan. Empty the material that is on the top screen into the batch storage container marked for that batch material.
  10. Transfer the fines in the bottom pan to the weighing pan on the electronic balance.
  11. If the holes in the bottom screen are clogged with material, remove the material with the appropriate screen brush. Brush out the material from the bottom side of the screen.
  12. Repeat this process until all of the batch material has been screened.
  13. The course material is in the mortar. Break the course material with the pestle. It is also acceptable to place the coarse material into a jar mill with some media and roll for several minutes to reduce the size, instead of grinding by hand with the mortar and pestle. Process the material starting at Step 7.1.6. If there is no course material remaining then proceed to Step 7.1.15.
  14. Record the weights of the good batch material and fine material on CR011.

1. **Combining Approved Batch Materials into a New Batch**

NOTE: Powder from several approved batches can be combined into a new batch of material. This should be done by first estimating the d33 of the combined batch, using a calculation of the mixture by weight, as appropriate for the target material specification. Afterwards, powder granules that have been passed through sieves and are acceptable for pressing can be blended together either by tumbling in the V-blender (agitator bar off) or rolling in a carboy (with no media). This combined batch should then go through the Batch Qualification process and tested against the appropriate material specification.

1. **Combining Fines into a New Batch**

NOTE: Fines from several approved batches can be combined into a new batch of material. This should be done by first estimating the d33 of the combined batch, using a calculation of the mixture by weight, as appropriate for the target material specification. Afterwards, the binder material must be burned out from the fines before they can be blended together. This fines batch should then go through entire Granulation process again, followed by the Batch Qualification process, tested against the appropriate material specification.

1. **Binder #3 Preparation**

NOTE: This section covers the procedure to be used in the preparation of binder solution. Binders are, in general, the ingredients that are added to batches to give the pressed green ware handling strength. Most binders are very sensitive to heat.

* 1. Equipment
* Electronic Scale - capable of 5 Kg +/- 0.1 gm
* Appropriate weighing pan
* Small plastic scoop for PVA and/or PEG
* 250 ml graduated cylinder
* 1000 ml graduated cylinder
* Electric stirrer motor with mixing blade and stand
* 600 ml beaker
* 1000mL beaker
* 2000 ml beaker
* Hot plate
* Thermometer that can measure temperature up to 100°C
* 1000 ml screw cap Erlenmeyer flask for binder storage (2)
* Refrigerator
* Plastic Stirring Rod
  1. Materials
* Polyethylene Glycol (PEG) 600
* DI Water
* DuPont Evanol 51-05 Polyvinyl alcohol (PVA)
* Ethylene Glycol
  1. **20% PEG 600 Solution**
     1. Place the container of PEG 600 into an oven and heat to approximately 40°C. Allow the PEG to be heated for several hours in the oven in order to melt the solid PEG.
     2. Place a 600 ml beaker in the oven to be heated along with the PEG. This will be used as a weighing dish for the melted PEG.
     3. Using the 1000 ml graduated cylinder, measure and add 800 ml of Dl water to the 1000 ml beaker.
     4. Place the beaker of water onto the hot plate. Insert the mixing blade of the stirrer into the water.
     5. Turn on the hot plate and set the temperature control knob approximately 1/5 of the way between low and high.
     6. Insert the thermometer into the beaker.
     7. Heat the water to 45-50°C.
     8. Once the PEG 600 has melted, remove it from the oven. Also remove the pre-heated 250 ml beaker at this time.
     9. Place the pre-heated 250 ml beaker onto the scale, and tare to zero.
     10. Pour out 200 gms of liquid PEG into the 250 ml beaker.
     11. Add the 200 gms of liquid PEG to the beaker of water on the hot plate. Turn on the stirrer motor.
     12. Cover the beaker with aluminum foil, and set the stirrer motor speed to medium. Monitor the temperature to insure that the mixture does not exceed 50°C. (Doing so will scorch the PEG.)
     13. Allow the PEG to mix and dissolve for four hours. Use a timer as required. Reduce the setting on the hot plate if the temperature starts to exceed 50°C.
     14. After the mixing is completed, remove the foil from the beaker, and remove the stirring rod from the solution. Turn off the hotplate.
     15. Remove the beaker from the hotplate, cover with foil, label “20% PEG 600” and set aside.
  2. **20% PVA Solution**
     1. Using the 1000 ml graduated cylinder, measure and add 800 ml of Dl water to the 2000 ml beaker.
     2. Place the beaker of water onto the hot plate. Insert the mixing blade of the stirrer into the water.
     3. Turn on the hot plate and set the temperature control knob approximately 1/3 of the way between low and high.
     4. Insert the thermometer into the beaker.
     5. Heat the water to 70-80°C.
     6. Place the weighing pan on the scale and tare it to zero.
     7. Weigh out 200 gms of PVA into the weighing pan.
     8. When the water has reached a temperature of 70-80°C, turn on the stirrer motor to medium and slowly add the 200 gms of PVA to the water.
     9. Cover the beaker with aluminum foil. Monitor the temperature to insure that the mixture does not exceed 80°C. (Doing so will scorch the PVA)
     10. Allow the PVA to mix until it is completely dissolved. This usually takes about three hours.
     11. Turn the hot plate to 60ºC, and allow the solution to stir until most of the air bubbles have been removed. This should take from 4 - 6 hrs.
     12. After the solution has cleared of air bubbles, check the temperature of the PVA solution. If it is below 40ºC, adjust the hotplate and heat it to 40-50ºC.
  3. **Combining the PVA and PEG Solutions**
     1. Turn the hot plate to 60ºC.
     2. Pour the PVA solution through an 80-mesh sieve into another 2000mL beaker to separate any unmelted particles from the liquid.
     3. Insert the stirrer into the PVA solution, and turn on the stirrer to medium. Add the 20% PEG 600 Solution to the warm PVA solution.
     4. Measure out 20 grams of Ethylene Glycol, and add to the PVA / PEG mixture.
     5. Cover the beaker with aluminum foil and allow the solutions to mix for 2 hours at 40-50ºC.
     6. Turn off the hotplate and the mixer, and remove the stirrer from the solution.
     7. Pour the binder solution into two Erlenmeyer flasks and screw the tops onto the flasks. Label the flasks “Binder #3”, and store in the refrigerator.

**Referenced Documents: CR011, CR1027**