

PAPERMAKING AND ANALYSIS OF PAPER

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Ancient people used various methods for writing and drawing. Drawings were made on stone using dirt or colored minerals and animal fat, wooden boards coated with wax were used for writing, and animal skins (parchment and vellum) were dried and scraped for writing purposes. The first writing surface made from plant materials was papyrus.

Paper was first reported to be made in China, about 100 A.D., from tree bark, scraps of linen, hemp, and pieces of fishing nets that was beat into pulp and then formed over loosely woven cloth over a wood frame.

Papermaking was spread to the Middle East about 751 A.D. and eventually to Europe about 1151. Those papers were generally made from rags and linen, not wood fibers.

The use of wood fibers to make paper, similar to the process used by wasps, was described by Rene de Reaumur in 1719, but a crude process was not developed until about 1839. The actual use of wood pulp did not come about until 1844. The first newspaper printed entirely on “paper made of wood” was The Boston Weekly Journal on January 15, 1863.

The use of paper to transmit the written word or drawings and other artistic endeavors has been one of the major forms of communication through the 20th century. Only in recent years has the use of the media and multimedia started to replace paper as one of our major forms of communication.

There are several types of papers commonly encountered on a daily basis. Newsprint (or newspaper paper) is mainly ground wood fibers with little or no additives. Bond paper, used for writing, copying, and printing (such as laser printers) will contain sizing to reduce ink bleeding and mineral additives to increase opacity of the paper and brightness. Coated papers, used for magazines, have a thin coating of latex containing mineral additives to produce a bright, shiny paper. Specialty papers are also made for specific purposes such as drawing, painting, and craft applications. The most common mineral additives are kaolin clay and titanium dioxide. Titanium dioxide has the highest brightness (or whiteness) but is significantly more expensive.

Commercial papers are made in a continuous process in which the pulp mixture is deposited on a moving wire (what we call a screen). This causes an alignment of the fibers that produce the tear characteristics observed in papers such as newsprint. The paper transferred to a continuous felt surface and is dried and calendared (compressed between two rollers) before being cut into sheets. Careful examination of a sheet of paper will show that one side is smoother than the other. The smoother side, known as the *felt side*, is the top of the paper that was formed and the side that was in contact with the felt. The rougher side is known as the *wire side* and is side that

formed in contact with the wire. Often, a label on a package of paper indicates that the user should use a particular side first, that is the felt side.



Materials

Paper for pulp:

Pressed pulp board, available from papermaking supply companies.

Newspapers, waste bond or computer paper, old catalogs (do not use shiny, coated papers), or plain paper junk mail.

Cotton linters or other rag-type materials, available from papermaking supply companies.

Retention aid (this is a 0.5% solution of high molecular weight, cationic polyacrylamide, available in both solution and powder form.

To prepare the solution, dissolve 0.5 g of dry retention aid in 3 mL of ethyl alcohol, then dilute to 100 mL with water.)

Liquid sizing (this experiment uses a water emulsion of an alkyl ketene dimer.)

A home made sizing can be prepared by mixing 1 pkg of unflavored gelatin with 30 mL (2 Tbs.) warm water until the gelatin dissolves. Mix 2 Tbs. cornstarch with 30 mL (2 Tbs.) cold water and add to the gelatin solution. Then add 1 cup boiling water and stir to dissolve. Use at the rate of 1/8 tsp. per blender of pulp.

Kaolin clay

Optional: Calcium carbonate

Optional: Titanium dioxide (a filler and brightener for paper)

Papermaking felt sheets. One felt is needed for each sheet of paper made.

Papermaking mould and deckle

Measuring spoons for liquid sizing, retention aid, and mineral fillers

Colander or large strainer

Cheese cloth (if colander has large holes)

Blender

Plastic bags (Zip Lock type or with twist ties)

Large plastic vat to hold pulp solution. (Rubbermaid 10 gallon Rough Tote/storage

containers work well. These have lids so sheetforming solutions can be covered and stored for short periods.)

Plastic wash basins

Newspapers. The newspapers should be separated into separate sheets (full-sized sheets) that are folded in half and in half a second time (The same size as the newspaper when sold at a news stand.)

Optional: Paper press, to press out liquid from paper sheets after forming.

Optional: Iron, (a standard steam or dry iron for clothing) to dry paper sheets

Safety

Wear safety glasses or goggles at all times in the laboratory.

There are no safety hazards associated with the materials used in this experiment.

When using the blender, always make sure the top is in place before turning on the motor.

Due to the heavy use of water in this experiment, floors may become slippery. Clean up all spills as quickly as possible.

Disposal

Dispose of materials in the proper waste containers. Do NOT put pulp solutions in the sink.

PROCEDURE

You will be making several sheets of paper:

The first sheet will use plain pulp or paper fibers.

The second sheet will use plain pulp or paper fibers with some liquid sizing.

The third sheet will use plain pulp or paper fibers with some liquid sizing, retention aid and kaolin clay

Additional sheets will be made using plain pulp or paper fibers with a different filler such as calcium carbonate or titanium dioxide with liquid sizing and retention aid. Your instructor will direct you on this.

Additional sheets using cotton linters or other rag-type materials, either plain or with sizing and, if desired, mineral filler and retention aid. Your instructor will direct you on this.

In each case, the procedure is the same, but additional materials are blended with the pulp for each type of paper.

Preparation of the pulp

Select the paper or pressed pulp. Place it in a large container, cover with cold water, and allow it to soak overnight.

Tear the wet paper into 2.5 cm (one inch) squares. If desired, cook the paper, as instructed below. If cooking is omitted, the paper is ready for processing into pulp. **NOTE:** Cooking is not required for pressed pulp board, recycled paper, or cotton linter board.

OPTIONAL: Cook the paper by placing the torn paper into a pot or beaker of boiling water and simmer for one hour. This process will help the paper fibers to swell and fray for improved bonding in the papermaking process. While the paper is cooking, line a colander with a double or triple layer of cheese cloth. After cooking, drain the paper in the colander. Store the wet paper in plastic bags until ready to use. **NOTE:** The wet paper can be refrigerated or frozen to prolong storage.

Using a blender, process the paper into pulp. For each batch, use at least two cups of water and a ball of paper about the size of a golf ball. **CAUTION:** Using too much paper will cause the blender motor to burn out. It is suggested that the blender be *pulsed* until the paper pieces are broken up, then allow the blender to run until the paper is completely disintegrated. This process is called *beating the paper*.

If you will be making a paper containing sizing, add ½ tsp. of sizing to the blender *after* beating the paper. Blend to mix.

If you are making a paper containing sizing and mineral clay filler, add 1 tsp. of kaolin clay (or other filler material) *after* beating the paper, blend to mix, then add 2.5 mL (½ tsp.) of sizing, blend to mix, and, last, add 5 mL (1 tsp.) retention aid, blend to mix.

After beating the pulp, the mixture can be added to the papermaking vat.

If the pulp is to be reserved for later use, drain the pulp in a large strainer or colander over a wash basin. You should have approximately one-half cup of processed pulp.

NOTE 1: If desired, the pulp can be strained and stored in plastic bags in a refrigerator. (Unrefrigerated pulp should last about one week.)

NOTE 2: If the paper is to be colored, the pigments would be added after beating and before the sheets are formed. Professional pigments, made for paper use, are recommended along with retention aids according to the directions provided by the pigment manufacturer. The worker must wear rubber gloves and an apron when working with pigments.

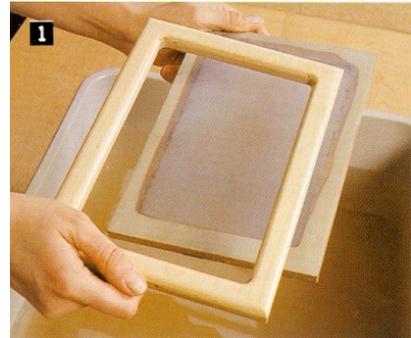
Sheetforming (Making the paper)

The vat to be used should be filled about half full with cold water. Once a sheet of paper is made, it will contain residual pulp. It is not necessary to replace the water for successive sheetforming.

Depending on the size of the vat used, add one-half to one cup of prepared pulp. (Use the amount you prepared in the blender and strained - this will be about one-half cup of pulp. Too little pulp results in sheets that are too thin, too much pulp results in sheets that are too thick.) Stir to disperse the pulp.

Place a pile of newspapers, separated into separate sheets and folded (see the *Materials* section) on your work area. Place a clean, slightly damp felt on top of the stack of newspapers.

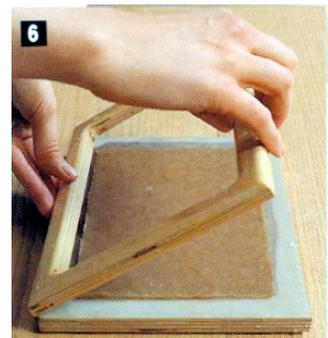
Place the *mould* (the wood frame with the wire or plastic screening) and *deckle* (the wood frame) together with the screening in the center (in contact with the deckle).



Holding the mould and deckle together (make sure your fingers are not touching the screen) place it at the far end of the vat, away from you and slide it into the solution, deckle side up, disturbing the pulp as little as possible. Once the mould is flat on the bottom of the vat, raise it straight up out of the vat, keeping it level, with a *slight* shake to help distribute the fibers evenly. Allow the water to drain while holding the mould over the vat (about 10-15 seconds).

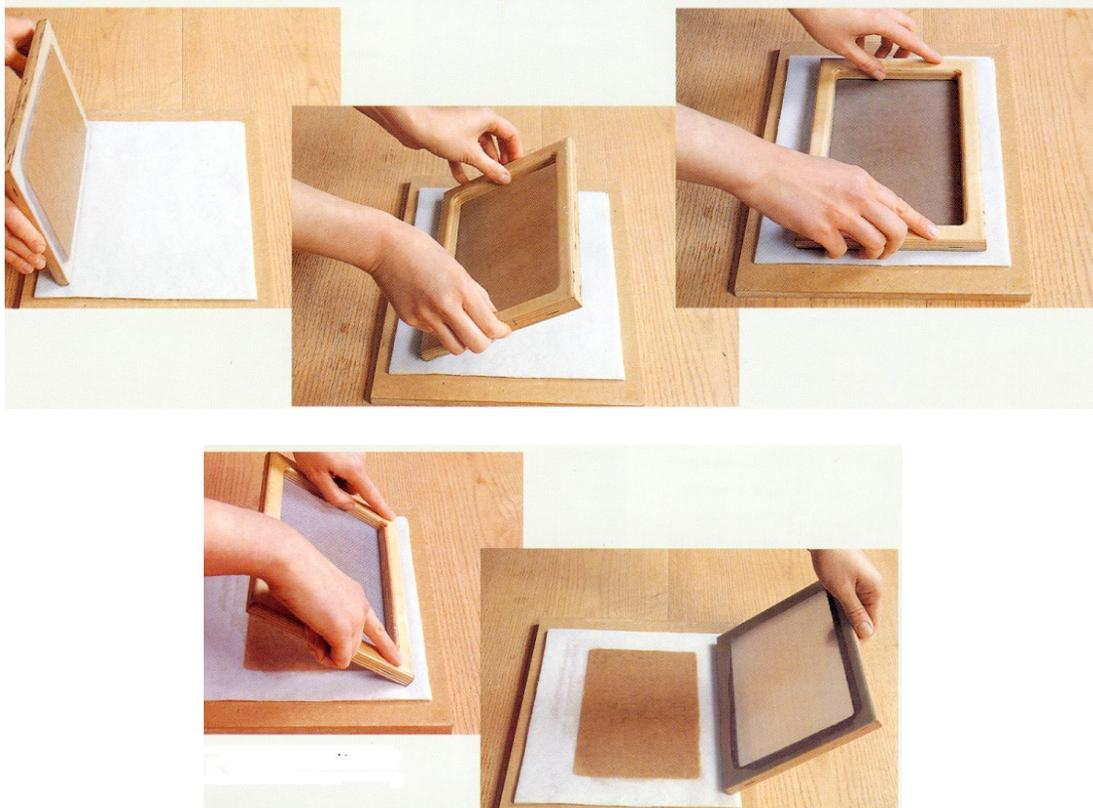


Set the mould down on the felt. Remove the deckle.



Rinse the deckle quickly in the surface of the water in the vat to remove any pulp. Set the deckle aside.

Couch (from the French *couche*, meaning *to lay*) the sheet of paper by inverting the mould and fibers onto the felt. Press the back of the screen with a wet sponge to release the paper, allowing it to adhere to the felt. Lift the mould from one end and slowly “roll” it up and away from the paper. The paper should release from the mould and adhere to the felt.



Place a small piece of paper, with your name on it and identifying the sheet as *plain*, *with sizing*, or *with mineral clay*, ***in pencil***, near the corner of the sheet. All the sheets, in contact with the felts, will be stacked for pressing and drying.

Pressing the stack is done in a press. The papers and felts are pressed for about 10 minutes.

Generally, the paper will adhere to the felt and can be dried by hanging on a clothesline. It will take several hours or overnight to dry. (As an alternative, paper sheets can be dried by sandwiching them between two pieces of cotton cloth and ironing them with a dry household iron.) When dry, remove the dried paper from the felt.

Please clean up your work area at the conclusion of the experiment.

Evaluation and Analysis of Paper

Materials

- Paper brightness samples
- Pencil, pen, and felt-tip markers
- Crucibles and covers
- Pipestem triangle
- Crucible tongs
- Scissors
- Ruler
- Meeker or Fisher burner

Safety

Wear safety glasses or goggles at all times in the laboratory.

There are no safety hazards associated with the materials used in this experiment.

Crucibles, triangles, and support rings will get very hot during the ashing process. Allow sufficient time for these to cool.

Disposal

Dispose of materials in the proper waste containers.

PROCEDURE

Compare brightness (color or whiteness) of the papers you made (plain paper, paper with sizing, and paper with mineral clay) with each other and with some commercial paper or a chart of known brightness. Record your observations.

Evaluate the opacity of the papers by drawing a pencil line on each of the papers. Turn over the papers and look for show through. Is the pencil line visible from the reverse side? Record your results.

Write on each of the papers with a ball point pen. Does the ink spread (bleed)? Does the ink show through from the reverse side? Record your results.

Write on each of the papers with a felt-tip marker. Does the ink spread (bleed)? Does the ink show through from the reverse side? Record your results.

Test the strength of the paper. Tear each of the papers. Does it tear differently in different directions? Compare the tear test with commercial paper and/or newspaper. Record your results.

Ash Content and Mineral Retention

1. Using a gas burner

Several paper samples will be ashed to determine mineral content.

Obtain a crucible with a cover. Make sure it is clean and dry. If necessary, clean the crucible and dry it in a drying oven.

Weigh the crucible and cover and record its mass.

Cut a strip from your plain paper sheet (unbleached wood pulp or plain paper) approximately 1.5 cm wide (slightly more than $\frac{1}{2}$ inch) by 20 cm long (approximately 8 inches). Roll the strip into a loose coil and place it into the pre-weighed crucible.

Weigh the crucible and the paper and record the mass. (The paper sample should weigh at least 0.20 g or more.)

Your instructor will demonstrate how to ash your paper samples using a laboratory burner. This will burn off all the carbon-based material leaving only mineral materials in

the
form of
ash.



Left: Ashing the paper using a laboratory burner

Above: The lid on the crucible is slightly open to allow air to circulate during the ashing process. If the paper is actively burning (i.e., a yellow flame) close the crucible lid until the flame

When the paper has been properly ashed, all the carbon has been burned off and only a grey colored ash remains.

Weigh the crucible with the contained ash. Determine the mass of the ash residue. Calculate the percent of the paper sample that remains as ash. Record your results. NOTE: The mass of the ash may be very small for plain paper samples.

Repeat the ashing process with a second sample of paper containing a mineral filler. If you made paper with two or more different mineral fillers, you may be instructed to ash another sample.

Repeat the ashing process with a sample from the cotton-based paper.



2. Using a laboratory furnace

If a laboratory furnace is used, only three samples will be ashed: plain wood pulp, wood pulp with a mineral filler, and a cotton-based paper.

Obtain three crucibles with covers. Make sure they are clean and dry. If necessary, clean the crucibles and dry them in a drying oven. Mark each crucible by scratching them with the tip of a file on the bottom for later identification. Weigh each crucible and cover and record its mass.

Cut a strip of each paper approximately 1.5 cm wide (slightly more than ½ inch) by 20 cm long (approximately 8 inches). Roll each strip into a loose coil and place them into separate, pre-weighed crucibles. Record which crucible contains which paper sample. Weigh the crucibles and the papers and record their masses. (The paper samples should weigh at least 0.20 g or more.)

Using a laboratory furnace, during the next week, your paper samples, along with some commercial paper samples, will be placed in the furnace and fired at 800°C (1472 F).

Weigh each of the crucibles with the contained ash. Determine the mass of each of the ash residues. Calculate the percent of the paper sample that remains as ash. Record your results.

Report Form

PAPERMAKING AND ANALYSIS OF PAPER

Name _____ Course/Section _____

Partner's Name (If applicable) _____ Date _____

Evaluating the Paper

Compare color (whiteness) of the papers:

Plain paper:

Paper with sizing:

Paper with mineral clay added:

How do the colors of the papers you made compare with commercial papers?

Evaluate the opacity of the papers by drawing a pencil line on each of the papers. Turn over the papers and look for show through. Is the pencil line visible from the reverse side? Describe your results:

Repeat with a ball point pen. Does the ink spread (bleed)? Does the ink show through from the reverse side? Describe your results:

Repeat with a marker. Does the ink spread (bleed)? Does the ink show through from the reverse side? Describe your results:

Strength - tear the paper. Does it tear differently in different directions? Compare the tear test with commercial paper and/or newspaper. Describe your results:

Which paper provides the best writing surface?

Ash Content and Mineral Retention

	<u>Mass of crucible and cover</u>	<u>Mass of crucible, cover, and paper</u>	<u>Mass of paper</u>	<u>Mass of crucible, cover, and ash</u>	<u>Mass of ash</u>	<u>Percent ash</u>
Plain paper	_____ g	_____ g	_____ g	_____ g	_____ g	_____ %
Paper with sizing	_____ g	_____ g	_____ g	_____ g	_____ g	_____ %
Paper with kaolin clay	_____ g	_____ g	_____ g	_____ g	_____ g	_____ %
Paper with calcium carbonate	_____ g	_____ g	_____ g	_____ g	_____ g	_____ %
Paper with titanium dioxide	_____ g	_____ g	_____ g	_____ g	_____ g	_____ %
Cotton based paper	_____ g	_____ g	_____ g	_____ g	_____ g	_____ %

$$\text{Percent ash} = \frac{\text{Mass of ash}}{\text{Mass of paper}} \times 100$$

For comparison purposes, the ash content for the reference brightness papers are: (These values will be supplied by your instructor)

Brightness	84	87	90	92	94	100
Ash content, %						

What conclusions can you draw from the ash information of the reference brightness papers?