2018 Phenolic Laminate 11
2/23/2018

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DEPARTMENT OF CHEMICAL ENGINEERING
CHE 395E

A STUDY OF PHENOLIC LAMINATE PRODUCTION

I.Object

The overall objective of this experiment is to produce a phenolic/paper laminate, similar to the familiar "Formica" commonly used for furniture and counter tops. This study includes the preparation of a laminating varnish from the condensation of phenol with formaldehyde, the impregnation of paper stock with the varnish, and the lamination of treated paper into a finished product.

PART I: PREPARATION OF PHENOLIC POLYMER SUITABLE FOR LAMINATING

II. References

1. Kirk-Othmer Encyclopedia of Chemical Technology, Vol. 15, 2nd Edition, Wiley (1968), pages 176-208.
2. The Chemistry of Phenolic Resins, Martin, Wiley (1956).
3. Encyclopedia of Polymer Science and Technology, Interscience (1972), Vol. 8, 9, 10.
4. Polymer Science, F. W. Billmeyer, Wiley (1971)
5. Handbook of Industrial Toxicology, Plunkett, Chemical Publishing (1966)
6. P. J. Flory, Principles of Polymer Chemistry, Cornell University Press (1953).

III. Equipment and Supplies

1. Chemical Hood (Room 201)
2. 500 ml, 3-neck, round bottom flask
3. Heating mantle and voltage control for flask
4. Reflux condenser
5. Thermometer, 0-150 ^{o}C
6. Rubber gloves
7. Phenol
8. Formaldehyde solution, 37% by weight CH2o
9. Sodium hydroxide
10. Oxalic acid

11. pH indicating paper
12. One-liter bottle with lid
13. Methyl alcohol
14. Small metal pan, 300 ml capacity minimum
15. Electric hot plate

IV. Procedure

I. Be familiar with the toxicological hazards of phenol and the other chemicals before beginning. Be especially careful when handling phenol. Wear rubber gloves. This experiment is done in a hood.

2. Weigh 94.1 g (1 g mole) of phenol and charge into the round bottom flask. The phenol may be easily removed from its bottle by warming the bottle in hot water.

3. Add 1.3 g mole formaldehyde (105.4 g U.S.P. 37% formaldehyde solution with water) to the vessel.

4. Prepare 25 ml of 3N sodium hydroxide solution and add to the vessel.

5. Seal vessel and heat at reflux (T=85-90ºc) for two hours. Shut off heat and allow to cool somewhat before opening the vessel.

6. Calculate the amount of oxalic acid necessary to neutralize the vessel contents. Add approximately this amount so that the solution pH is about 6.5.

7. Dehydrate the mixture by carefully heating at atmospheric pressure. This is best done by transferring the vessel contents to a shallow pan heated by a hot plate (in the hood, of course). The cloudy product should be heated and stirred frequently until it clears. It will then have a molasses-like viscosity at room temperature. During dehydration, care should be taken to keep the product temperature at or below 100°c. When all of the water is removed, the polymer temperature will begin to move above 100°c, and the pan should be removed from the heat source at this point.

8. Transfer the dehydrated polymer to a tared bottle. Allow the resin to cool to room temperature. Then add sufficient methyl alcohol to make a 50% by weight phenol-

formaldehyde solution. Store this solution in refrigerator in until next lab period. Be sure to label bottle properly.

9. Carefully clean the glass reactor, dehydration pan, etc., for the next group before leaving.

V. Report

l. Briefly describe the pertinent chemical reactions involved in forming the condensation resin.

2. Phenolic resins are usually categorized by terms such as "one-step", "two-step", "resole", and "novolak". Categorize your resin this way.

3. Stages of cure of phenolic resins are usually categorized as A stage, B stage, and C stage. At what stage is your resin when it is diluted with methanol?

4. Polymeric materials are often labeled as thermoplastic or thermosetting materials. What type of material is phenol-formaldehyde? Why?

5. Why is phenol-formaldehyde called a condensation resin? Why then is the dehydration step so critical?

PART II: PREPARATION OF PHENOLIC/PAPER LAMINATES

I. Equipment and Supplies

l. Polyethylene sheeting
2. Rubber brayer
3. Air oven
4. Aluminum foil
5. Kraft paper stock
6. Flat aluminum plates 6" x 6"
7. Compression press
8. Analytical balance
9. Scissors
10. Rubber gloves

II. Procedure

l. Set air oven to 100 ^{o}C.

2. Cut paper stock into 3-inch x 3-inch squares.

3. Weigh paper squares on analytical balance and mark for identification.

4. Place paper stock on polyethylene-covered flat surface.

5. Weigh out sufficient laminating varnish so that the resin content of the treated paper will be about 65% by weight.

6. Pour varnish on paper and use rubber brayer to work the varnish into the paper stock.

7. Allow the paper to air dry for 10-15 minutes. If sufficient varnish has been added to the paper, it will appear to be translucent to light. If treated paper is not translucent, additional varnish may be required.

8. After air drying, again weigh specimens and determine the resin content of the treated paper expressed as %R = (w2-w1)/w1 X 100, where w2 = treated weight and w1 the untreated weight

9. Place air dried sheets into 100°c oven for 3 minutes or until paper is no longer "tacky." Be careful not to exceed 100 ^{o}C.

10. Weigh oven dried samples and determine the volatile content of the treated paper, %V = (w2-w3)/w2 X 100, where w3 = dried weight.

11. Cut oven dried sheet into 1 inch x 1 inch squares and stack together to form a 10 gram sandwich. (Be sure to weigh the sandwich.) Overlay the stack with aluminum foil, place between aluminum plates, and place assembly into the compression press which has been pre-heated to 290 ^{o}F.

12. Apply 1100 psi compressive stress to the package for three minutes.

13. Remove sample, break off the resin which has flowed out of the sample and weigh the cured sandwich. Calculate the percent flow given by %F = (w4-w5)/w4X 100 where w4 is the original weight of paper laminate prior to pressing and w5 = weight of cured sandwich after removal of polymer flash

14. Repeat steps 9-13 for oven residence times of 6 and 9 minutes.

15. Cool press to 250°F. Press paper laminates made of about 16 sheets of treated paper (3" x 3" size) varying pressure and curing time in the press. Suggested starting conditions are 250-300 psi and 6-8 minutes cure time.

III. Report

l. Show calculation for how compressive stress on the laminate is determined from hydraulic press oil pressure readings.

2. Explain the trend, if any, between %F and drying oven residence time.

3. Contrast the apparent properties of cured pure phenolic resin with those of the paper/resin laminate.

4. Is the volatile content observed what you would expect, knowing the amount of alcohol in the varnish? Or is it greater? Explain the difference.

5. Discuss any trends observed in your laminate production in step 15 above.