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Making a Surface Coating

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Description

In this activity students prepare a nonaqueous surface coating by first making a polymeric resin and then adding an organic solvent. The product is a basic varnish that hardens by loss of organic solvent to the air. A simple paint can be made by adding pigment to the varnish. An oleoresinous varnish can be made by adding an oil, such as linseed. Certain properties of the varnishes and paint film can be tested and compared: drying time, hardness, flexibility, and degradation of film or underlying substrate due to long-term exposure to ultraviolet radiation.

Materials

- 0.4 g ethylene glycol (IUPAC 1,2-dihydroxyethane)
- 0.25 g glycerine (IUPAC 1,2,3-trihydroxypropane)
- 1.3 g phthalic anhydride (IUPAC 1,2-benzenedicarboxylic anhydride)
- 2.5 mL 60/40 (volume) mixture of xylene/butanol
- approximately 3 g titanium oxide
- approximately 3 g red iron oxide
- balance sensitive to 0.01 g
- thick-walled Pyrex test tube (about 12- to 15-mm by 150-mm) with cap or stopper
- test-tube holder
- 250-mL or larger beaker
- stirring rod
- Bunsen burner
- tongs
- mortar and pestle
- several wooden tongue depressors or craft sticks
- small metal panel (from empty, clean metal soup can)
- small plastic panel (from coffee can lid or 1-gallon water jug)
- cotton swabs (e.g., Q-Tips®)
- pencils having a range in hardness (optional)

Safety, Handling, and Disposal

It is your responsibility to specifically follow your institution’s standard operating procedures (SOPs) and all local, state, and national guidelines on safe handling and storage of all chemicals and equipment you may use in this activity. This includes determining and using the appropriate personal protective equipment (e.g., goggles, gloves, apron). If you are at any time unsure about an SOP or other regulation, check with your instructor.

Carry out all mixing and heating procedures in a fume hood. In general, work with paint and varnishes in a well-ventilated area. Use tongs to handle the hot equipment in this procedure. The heated mixtures reach temperatures of 150°C to 220°C (much hotter than the temperature of boiling water). Do not stir with a thermometer; as a thermometer can easily break, resulting in spilled mercury. Use only a thick-walled Pyrex® test tube for carrying out the high-temperature reactions of this procedure.

Immediately clean up equipment using xylene/butanol washes. Dispose of the used solvent mixture in a specified recovery container.

Procedure

To prepare varnish

1. Place the Pyrex test tube and its cap or stopper inside the beaker, and measure their combined mass. Record this as the tare mass (A).

2. To the test tube add 0.4 g ethylene glycol, 0.25 g glycerine, and 1.3 g phthalic anhydride.

3. Using a small Bunsen burner flame, gently heat the test tube. Handle the test tube and its contents carefully; the temperature of the reaction becomes high.

4. Observe the elimination of water, which collects at the top of the test tube. To remove the water, gently heat that part of the test tube.

5. Heat the test tube until no further elimination of water is observed. When the mixture seems to be thickening, stop heating. Allow the test tube to cool slightly (approximately 2 minutes).

6. Put the test tube in a test-tube rack. Place a stirring rod in the test tube and carefully add a small amount of the 60/40 xylene/butanol mixture by pouring it down along the stirring rod; carefully stir. (Do not pour cold solvent onto the hot glass.) Mix the contents before adding more solvent. Repeat the procedure. Add a total of 2.5 mL of the solvent mixture. Loosely cap the cooling test tube.

7. Allow the test tube and contents to cool to room temperature, and then tighten the cap or stopper on the test tube. Measure the combined mass of the beaker, test tube, cap, and prepared varnish, and record (B). Calculate the mass of prepared varnish (B–A). Label the test tube to identify its contents.

To prepare paint

1. Determine the mass of the varnish that is to be used to make the paint.

2. To a clean, dry mortar, add a little more pigment than one-half the mass of varnish being used. Grind with a pestle to yield a fine powder.

3. To the varnish, add pigment in a mass ratio of 1 to 2, pigment to varnish. Stir the mixture and store in a labeled, capped test tube (or screw-cap storage vial). Before use, this prepared paint should be thoroughly remixed.

To prepare oleoresinous varnish

1. Determine the mass of the varnish that is to be used to make the oleoresinous varnish.

2. To the varnish add raw or cooked linseed oil in a mass ratio of 1 to 5, oil to varnish. Thoroughly stir the mixture. Store the product in a labeled capped test tube (or screw-cap storage vial).

Test the prepared coatings

1. Using cotton swabs, apply the coating to be tested to one side of three wooden tongue depressors, three plastic pieces, and two metal pieces.

2. Use one sample of each substrate to determine drying time. After most of the solvent seems to have evaporated, lightly touch the coating. Note and record the tackiness and hardness. Repeat the observations after 1, 2, 3, 24, and 48 hours.

3. Allow all the test samples to dry completely. In a dark, clean, dry place, set aside one sample of each coated substrate as a control for future comparisons.

4. To test for flexibility of the coating, use one of the plastic samples to see how much it can be bent before the coating shows any cracks.

5. Use one sample of coated wood to test for hardness. To perform a pencil hardness test, use a set of pencils having a range in hardness. Starting with the pencil of softest lead, push the pencil lead along the surface to see which hardness just damages the finish. Alternatively, use a fingernail or another relatively thin edge to test the hardness.

6. Expose one coated sample of each substrate to direct sunlight over a period of 1 to 2 months or more. Compare the coating and substrates to the control samples stored in step 3 above. Wipe the surface of the coatings with a clean, dark cloth to check each for chalking (degradation of the binder). Check for changes in color of the coating and substrate, flexibility, and hardness.

Discussion

1. Once the resin has been prepared (steps 1–5 of "To prepare varnish"), what kind of a product would be expected if you allowed it to harden without adding a solvent? How does this differ from the dry product left after the evaporation of the solvent?

2. This lab activity has presented the basic components of a paint: the film former, pigment, and liquid (the solvent or diluent). There are other components of paints known by the collective name of additives. Additives include small amounts of substances that impart particular properties to the paint not supplied by the main components. The following are typical additives:
    - thickeners—improve consistency of the wet paint
    - defoamers—help break foam formed by agitation or application
    - antisettling agents or dispersion aids—keep the pigments dispersed
    - antiskinning agents—prevent formation of skin on the paint in the can
    - in-can preservatives and fungicides—kill organisms that might grow in the wet paint during storage
    - in-film preservatives and fungicides—kill organisms that might grow in the cured paint film
    - driers—improve the drying rate of the paint
    - ultraviolet absorbers—reduce degradation due to sunlight

Consider the properties of the basic paint produced in this activity and discuss possible additives to make the product more practical.

Explanation

This activity illustrates how a simple paint can be made starting with a polymerization reaction. The final stage of the polymerization results in a cross-linked polymer (the synthetic resin), which is the film former. A solvent is added so that the resin can be applied to surfaces as a thin film. The film former/solvent mixture is a basic varnish. When the varnish is applied to a substrate, as the solvent evaporates from the wet film, the resin hardens. If a pigment is added to the film former/solvent mixture, the product is a paint. When the solvent is lost from the paint, the resulting dry film contains pigment particles held in the hardened polymer network. If a drying oil is added to the film former/solvent, the product is called an oleoresinous varnish.

The polymerizations that occur when ethylene glycol, glycerol, and phthalic anhydride react yield linear chains as well as a cross-linked network of chains. Ethylene glycol has no secondary hydroxyls, so its polymerization with phthalic anhydride can yield only linear chains. (See Figure 1.)

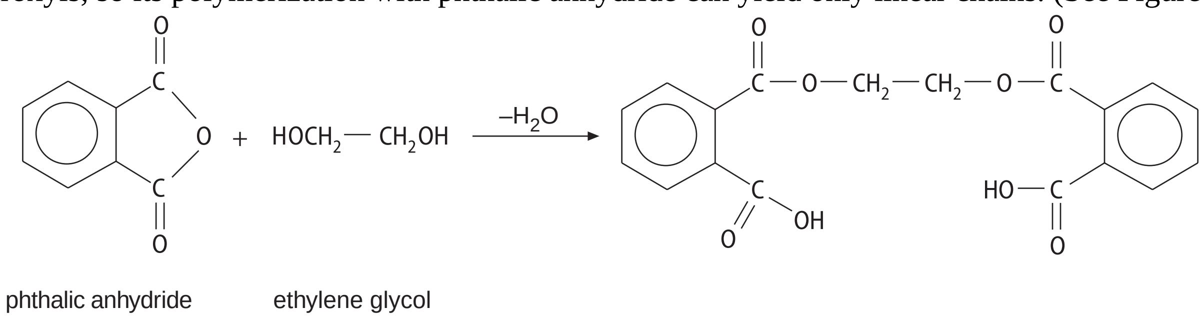

Figure 1: Polymerization of ethylene glycol with phthalic anhydride

When glycerol and phthalic anhydride are condensed at about 150°C, only the primary hydroxyls (from glycerol) are involved in the reaction, resulting in long, linear chains of polyester.
(See Figure 2.)

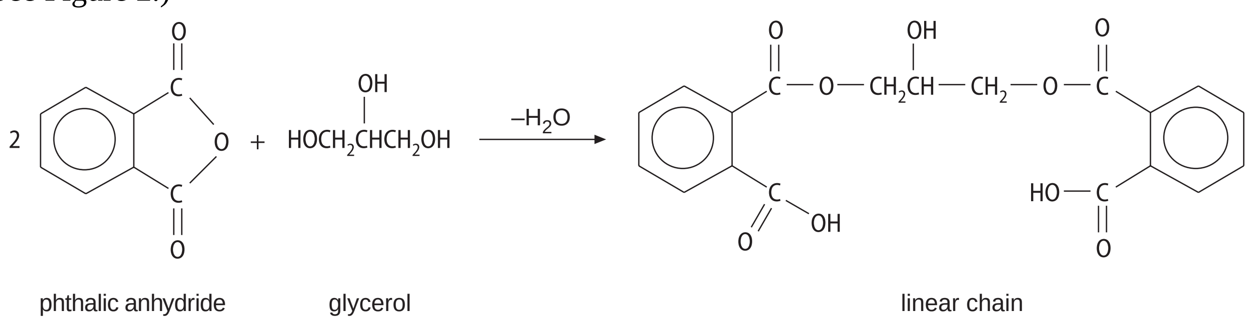

Figure 2: Reaction forming linear chains of polyester

However at higher temperatures, a second stage of polymerization occurs in which the secondary hydroxyls produce a cross-linked system. (See Figure 3.) The amount of cross linking is limited by reaction conditions.

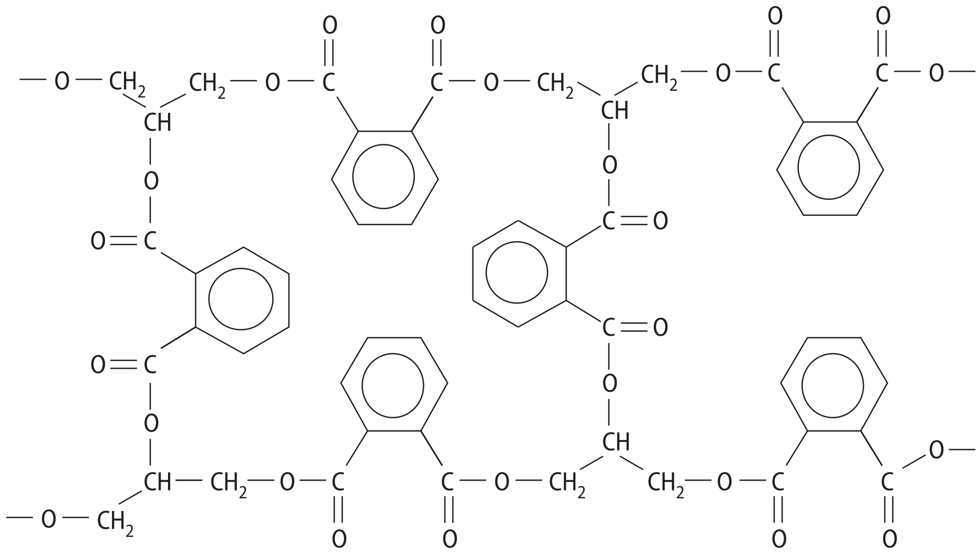

Figure 3: Cross-linked system produced by second-stage polymerization

The cross-linked polyester that is formed is more rigid than the linear one prepared using ethylene glycol. It is desirable to have linear as well as cross-linked chains since the linear chains provide some flexibility to the product.

To make the resin into a varnish that can be applied as a thin film, a solvent is added. The solvent used for this polyester resin is a mixture of organic solvents, a 60/40 mixture by volume of xylene/butanol. Sometimes a resin may not dissolve in either of two solvents alone but will dissolve in a mixture of the two. Polymer solvency is not necessarily easy to predict. The general rule “like dissolves like” is limited and does not give an indication of what will happen when a mixture of solvents is used.

The addition of pigment to the varnish results in a paint with properties that are different from those of the varnish. First and most obvious, the pigment provides color and opacity to the film. Pigments also absorb or reflect UV radiation that would otherwise cause degradation of the polymer systems in exterior paints. The presence of pigment may also improve the hiding power, strength, and durability of the film.

The combination of a natural oil such as linseed with either natural or synthetic resins yields a product called an oleoresinous varnish. Oils themselves have a relatively slow drying rate. They exhibit oxidative drying; the cross-linking results from a chemical reaction with oxygen from the atmosphere. Linseed oil has a modifying effect on the properties of a synthetic resin, influencing drying rate, film flexibility, and water resistance. Although, in general, oleoresinous products have declined in use since they have been displaced by synthetic resins, some specific applications use them to advantage. Oils might find use in products such as clear varnishes, primers and undercoats, aluminum paints, and marine coatings to optimize properties such as penetration into the surface of a wood substrate.

References

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