Owensboro Specialty Polymers, Inc.
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PVAc latex Analytical Tests

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Test for polyvinyl acetate

Polyvinyl acetate emulsions in adhesive formulations can often be identified by their odor. If equal parts of adhesive formulation and a 55% solution of sulfuric acid are boiled together in a flask, the polyvinyl acetate will be hydrolyzed to vinyl alcohol and acetic acid. The acetic acid smells like vinegar. Blue litmus paper held in the effluent vapors of the flask turns red.

Test for polyvinyl acetate copolymers

Take a small piece of the adhesive solids and heat with a small amount of 72% sulfuric acid and a few crystals of chromotropic acid at 65°C for ten minutes. Then allow it to stand for one hour. The solution will remain colorless or turn pale violet or pale orange if the resin is a polyvinyl acetate copolymer. If the resin is a homopolymer, a red color will be observed.

Test for polyvinyl alcohol

An adhesive formulation which is not too acid will coagulate if a saturated borax solution is added to it. The acidity of the formulation may be decreased by adding a few drops of ammonia.

Test for starch

Starch can be determined with a potassium iodide/iodine solution (6 grams KI and 2 grams iodine in 100 ml of water). If starch is present in an adhesive film, a drop of iodine solution on the film will turn blue.

Test for casein

A drop of 3% formaldehyde is added to a test tube containing a small amount of adhesive formulation. This mixture should be poured dropwise down the side of another test tube containing one drop only of 10% ferric chloride solution in 2 cc of concentrated sulfuric acid. (CAUTION: The adhesive formulation should be poured into the sulfuric acid very carefully. It is it added too fast, the mixture could boil violently, splattering the concentrated sulfuric acid.) A violet ring will form if casein is present.

Test for animal glue

Extract a small portion of the adhesive formulation with water, then divide the extract into two equal parts. If a precipitate is formed when a freshly filtered 10% sodium chloride solution saturated with tannic acid is added to the first part, animal flue or casein may be present. To confirm the presence of animal glue, acidify the second portion to a

pH of less than four. If no precipitate forms, animal flue is present. The presence of a precipitate indicate the presence of casein.

Test for plasticizers

Plasticizers are generally pure substances. They can be removed from the adhesive film by extracting with petroleum ether or ethyl ether. Both materials should be used because certain plasticizers which are insoluble in one material will be soluble in the other. After the extraction is complete, the solvent should be driven off. (CAUTION: These solvents are extremely flammable.) It is desirable to test the extract for these materials because by far the most common types of plasticizers used are phosphates and phthalates. Presence of phthalate plasticizer can be determined by heating equal parts of dried extract and phenol with a drop of sulfuric acid (5 min. at 60°C). If a pink color is observed when the mixture is made alkaline with dilute sodium hydroxide, phthalate ester is present. If the pink color disappears when the solution is reacidified, the presence of phthalate is confirmed.

Presence of phosphate ester is determined by fusion of the extract with potassium hydroxide. The fusion product is dissolved in water and ammonium molybdate solution is added (15 grams ammonium molybdate in 300 ml water; add to this 100 ml nitric acid [33%] and saturate with ammonium nitrate). Then heat. If the ammonium molybdate solution gives a bright yellow precipitate, the presence of phosphate ester is confirmed.

Test for solvents

Solvents are easily separated from the adhesive formulation by steam distillation, i.e., blowing live steam into the adhesive formulation and condensing the effluent vapor in a cooled flask. Chlorinated hydrocarbons are the most common solvents used in adhesive formulations. Many of them may be identified by their distinctive odors and physical properties. Presence of chlorine in the solvent may be determined by the Beilstein test. A clean copper wire is dipped in the solvent and held in a gas flame. A green flame indicated the presence of chlorine.

Presence of hydrocarbon solvents is easily established by the fact that these materials are less dense than water and are immiscible with water. A mixture of these materials will separate into two phases, the hydrocarbon phase floating on the aqueous phase.

Test for fillers

The presence of a filler may be determined by observing a dry film of the adhesive formulation. If the film is opaque, a filler is present.

Another quick way to confirm the presence of pigments, starches or dextrins is to obtain a weight/gallon of the adhesive. The density of PVAc emulsions is about 9.1 pounds/ gallon and anything significantly greater suggests the presence of fillers.

The tests outlined above are relatively simple to perform, and give quick results. Because they are simple, they will not give a complete analysis of the formulation. Unless the adhesives formulator has a complete analytical laboratory, a complete analysis may require considerable time and cost. On the other hand, simple determinations such as the type and amount of plasticizer or solvent can often be handled quickly at a nominal cost by a well equipped laboratory. Cost and expediency will dictate the best method of performing a particular analysis.