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BUILDING RESEARCH NOTE 2

CENTRAL BUILDING RESEARCH INSTITUTE, INDIA

CHARACTERIZATION OF POLYMERS

Plastics are relatively new materials and their properties are different from those of traditional ones such as concrete and metal. Out of several different types of plastics each has its own properties making it suitable for a particular application. Success in the use of plastics in building will depend upon correct choice and care with which they are used. Usually field engineers and builders do not possess the required information about the difference between various plastics such as polyvinyl chloride, polyethylene, polystyrene etc and many a times they appear to be the same with no diffefence from one plastic to another.

A particular plastic is made up of a particular polymer and each type has certain essential features/properties to make it suitable for a particular application. Users should be familiar with this aspect to avoid failures due to incorrect choice and the blame being passed on to the entire group of plastics. For example, commercially available plastic pipes in the country are made of either polyvinyl chloride or polyethylene. Similarly flooring and walling tiles are made of polyvinyl chloride or polystyrene. But each type would not have similar properties. Out of about 40 different types of polymers hardly a dozen are of major importance to building industry (Table 1).

Some times a supplier may himself not be knowing the product he is selling and may provide wrong information. So a user should have a simple scheme by which he may identify a polymer. In one installation, polyvinyl chloride and polyethylene pipe fittings were mixed by the plumber, both being black in colour; resulting in the use of PVC fittings witih PE pipe and vice versa. When water was opened the whole installation started leaking. This could have been easily avoided by separating the two types of fittings by immersing them in a bucket of water. Polyethylene fittings being lighter in weight remain on the surface of water whereas polyvinyl chloride fittings settle at the bottom.

Complete identification of a plastic may mean isolation and identification of a polymer or a copolymer or blend polymer, the isolation and identification of plasticizers, stabilizers, fillers, antioxidants and surface active agents etc., present in the polymer. A thorough and complete identification may need sophisticated and complex characterization techniques such at fractionation, molecular weight determinasion, chemical analysis, Infra red absorption spectroscopy, nuclear magnetlc resonance, differential thermal analysis, thermogravimetric analysis etc. In this note efforts have been made to compile preliminary procedures and simple and rapid qualitative tests which provide at least broad identification of a polymer. Only those polymers which are of major significance to building industry have been included.

There are four simple tests, viz, visual, combustion, pyrolysis and solubility which can be easily performed to ascertain the nature of a polymer from which any end product is manufactured. First thing that one may try to know is whether the given plastic is a thermoplastic or a thermoset. Thermoplastics are less rigid and if bent too far, first crack and then break into

Table 1

Polymers of Interest to the Building Industry

Name of the Polymer	Abbre-viation	Thermoplastic or Thermosetting	Density	Applications
Polyethlene	PE	Thermoplastic	0.92-0.96	Pipes and fittings, electricai conduits, moisture barrier films.
Polyvinyl chloride	PVC	-do-	1.32-1.44	Flooring materials lighting fixtures, roofing and walling panels. pipes and fittings, electrical conduits, water barrier goods.
Polystyrene	PS	-do-	1.04-1.07	Thermal insulation, lighting fixtures, panels, wall tiles, sanitary wares,
Polyvinyl acetate	PVA	-do-	1.17-1.19	Adhesive for wood, additive in concrete.
Polymethyl methacrylate	PMMA	-do-	1.17-1.19	Glazing, lighting-fixtures, panels, siding, wall coverings, bath fixtures,
Polyamides	Nylon	-do-	1.14	Building hardware items
Epoxy	EP	Thermosetting	1.1-1.4	Flooring, grouting, bonding, repairing and patching materials.
Polyester (unsaturated)	PEs	-do-	1.12-1.46	Flooring, GRP sheets for roofing and lighting.
Polyurethane	PU	-do-	1.10-1.50	Thermal insulation flóoring and composite materials.
Urea Formaldehyde	UF	-do-	1.47-1.52	Decorative laminates, plywood, particle board
Phenol Formaidehyde	PF	-do-	1,32-1.55	Decorative laminates, resin bonded boards, building hardwares.
Melamine Formaldehyde	MF	-do-	1.47-1.52	Decorative laminates, resin bonded boards.

fragments. Thermosets are rigid and any attempt to bend them usually results in sharp fracture. An electric soldering iron is a good tool for identification. Before it gets red hot press it against the sample. If it sinks into the sample the material is thermoplastic; if not, it is thermosetting.

Some polymers can be easily identified by the sound produced when dropped. For example, plastic products made from polystyrene give a peouliar metalic ring when dropped. Polyethylenes may be identifjed by their light weight and waxy feel. Similarly moulded articles in phenolic material are invariably in dark opaque colour while those made of urea and melamine formalpehyde are usually in pastel shades.

Melting and Combustion Test

A small non-luminous flame, preferably from a micro bunsen burner is employed and the behaviour of a small sample of the material to be identified is observed. Ease of combustion smoke, residue and the odour left after extinction of the burning sample can supply useful information. General results are summarized in Table 2.

Table 2

Classification of Polymers by Melting and Combustion Test

Combustion Test	Does not melt	Softens, melts in parts	Melts
Burns only when sufficiently heated	amino and phenol formaldehyde resins	Polyvinyl chloride	Polyamides (nylon), Polyurethanes
Burns with small flame or chars	polyesters (cross-linked type)	Epoxy resins	Polyethylene Polyvinyl-acetate, polymethyl methacrylate
Burns fairly readily with sooty flame	—	—	Polystyrene, Phenol formaldehyde (novolak-type)

Table 3

Classification of Polymers by Pyrolysis Test

Reaction of volatile products	Does not melt. may char	Chars, melts in part	Melts, may char later
Acidic	Phenol formaldehyde	Polyvinyl chloride Epoxy resins	Polyvinyl acelate, phenol formaldehyde (novolak type)
Alkaline	amino formaldehyde resins	—	Polyamides (nylon), Polyurethanes
Neutral	Polyesters (cross linked type)	—	Polyethylene, Polystyrene, Polymethyl methacrylate.

Pyrolysis Test

In this test, behaviour of a sample, a few mg in weight, when slowly heated in a small ignition tube is observed, and the reaction of the fumes is tested with an indicator paper. Results are summarised in Table 3.

Special Tests

Having found out a broader identification of polymer by above tests it is desirable to supplement the preliminary information by special tests. Typical colour tests alongwith solubility, combustion and pyrolysis behaviour of an individual polymer are given below :

Polyvinyl Chloride

Solubility : Soluble in liquids such as tetra hydrofuran, mixtures of acetone and carbon disulphide and to a less extent in methyl ethylketone.

Combustion : Softens and chars but self-extinguishing. When kept burning in a flame produces a sooty yellow flame and leaves black residue.

Pyrolysis : Chars, evolves ethylenic products and hydrochloric acid. No monomer is liberated.

Colour Test : About 50 milligram of the sample is placed in a test tube and dissolved in 5 c.c. pyridine by heating on a water bath. The addition of a few drops of methanolic sodium hydroxide produces a brown colour and eventually a brown precipitate is produced.

Polyethylene

Solubility : Insoluble at room temperature but soluble in aromatic and chlorinated solvents at 60 to 70°C and in molten paraffin wax. Xylene (at 60-70°C) is a convenient solvent. The polymer precipitates from solution on cooling to room temperature.

Combustion : Melts and burns with yellow tipped blue flame with little smoke, gives waxy smell on extinction, dripping usually noticed. Wire can be pulled which is extensible.

Pyrolysis : Melts and becomes transparent, evolves ethylene together with neutral white fumes that condense to fluid or waxy polymers of low molecular weight.

Other Tests : Polyethylene is one of the few polymers that float in water. Extremely limited solubility and lack of chemical reactivity are indicative of- this material. There are no simple chemical tests.

Polystyrene

Solubility : Soluble in aromatic and chlorinated hydrocarbons. Polymers of low molecular weight are also soluble in ether and acetone.

Combustion : Softens and burns fairly readily with an extremely sooty flame; characteristic odour of styrene noticeable on extinction.

Pyrolysis : Becomes elastically extensible (rubber like) at 80°C, then melts and distils as monomer and other products, neutral condensate with characteristic smell of styrene pervading.

Other Test : Rremarkable metallic 'tinkle' produced by moulded polystyrene, when struck or dropped, is unique for this class of material.

Colour Test : Asmall sample (0.1g) is refluxed with concentrated nitric acid (5ml) and the clear solution tnat results after about one hour is poured into 20 ml water, producing a pale yellow precipitate of p-nitrobenzoic acid which can be reduced, diazotised and coupled with β naphthol to produce a red colour.

Poyvinyl Acetate

Solubility : Soluble in aromatic or chlorinated hydrocarbons,acetone, alcohols and esters.

Combustion : Melts, burns freely with yellow smoky flame, characteristic vinyl odour on extinction. The sticky residue can be drawn into long threads which become brittle as they cool.

Pyrolysis : Melts, chars, evolves acid fumes.

Colour Test : Polyvinyl acetate stains deep brown on contact with dilute iodine solution (composed of 0.1g iodine and 1.0g potassium iodide dissolvcd in a mixture of 10 ml water and 10 ml alcohol and made upto 100 ml with 2N HCl) and the depth of colour increases on washing with water.

Polymethyl Metha Crylate

Solubility : Soluble in aromatic and chlorinated hydrocarbons (benzene, chloroform), esters (butyl acetate); 80/20 toluene/methyl alcohol mixture, gives low viscosity solutions. PMMA slowly disperses in acetone.

Combustion : PMMA once ignited burns with an almost smoke free blue based candle like flame. There is practically no residue left. The flame is easily extinguished and the characteristic fruity odour of the monomer then becomes apparent.

Pyrolysis : Distils largely as monomer in which formaldehyde may also be detected.

Colour Test : Polyacrylic acids and their water soluble salts are precipltated from solution of polymer on addition of alum, copper sulphate and ferric chloride. With the help of a needle or rod, merge one drop of solution of polymer into an adjacent drop of saturated alum solution. Clotting in the form of a characteristic ropiness of the bridge between the two drops is at once apparent. If the alum solution is replaced by one of copper sulphate or ferric chloride the ropy product is coloured.

Nylon

Solubility : Soluble in phenols hot formamide, hot formic or acetic acid, hot aniline.

Combustion : Melts, darkens, boils and finally burns with a small flame that is easily extinguished giving a white smoke of characteristic odour.

Pyrolysis : Decomposes at 260 to 300°C with alkaline distillate and characteristic odour.

Colur Test : When a test paper soaked in a freshly prepared saturated solution of O-nitro benzaldehyde in dilute caustic soda is presented to the hot vapours from pyrolysis of common nylon (nylon 66) it becomes deep mauve black in colour, the colour slowly fading on exposure of the paper to air. Other nylons (nylon 6) give only a grey colour.

Phenol Formaldehyde

Solubility : Novolaks and low mol. wt. polymers are soluble in acetone, ether, esters and to a less extent in hydrocarbons. Cured resins are practically insoluble in common solvents.

Combustion : Novolaks and low mol. wt. resins melt; cured resins char, ultimately burn leaving a strong odour of formaldehyde and phenol on extinction.

Pyrolysis : Novolaks readily melt, decompose and distil as phenolic alcohols; cured resins decompose, char, evolve phenol and formaldehyde. The volatile products have an acid reaction.

Other Test : All phenolic resins and articles moulded therefrom give small of phenol and formaldehyde when scraped with a knife or file.

Colour Test : On warming finely divided polymer or its distillation with dilute sulphuric acid phenolic resins lose formaldehyde. To one or two ml solution of this add a few drops of 5% aqueous chromotropic acid solution, followed by an excess of conc. H_2SO_4 and if necessary warm tc 100°C for 10 minutes. Formaldehyde produces a bright violet colour which is also given by UF and MF resins.

Phenols are released on dry distillation especially if the test sample is mixed with soda lime. They also result from fusion with alkali. A small sample of phenol is mixed on a spot plate with a few drops of conc. sulphuric acid, then treated with a few crystals of sodium nitrite. Phenols give a blue or green colour that becomes red on dilution with water and blue or green again when made alkaline.

Urea formaldehyde and Melamine formaldehyde Resins

Solubility : Polymers of low mol. wt. are soluble in water, alcohol, formaline, pyridine, formic acid. Final cured resin insoluble.

Combustion : The resins crack and char but are not easily set alight. Residues give smell of formaldehyhe and amine derivatives (resembling burnt fish).

Pyrolysis : The resins crack, char and give off alkaline vapours in which formaldehyde and a characteristic fish like odour are present.

Other Test : When refluxed with an excess of aniline, urea resins evolve ammonia and on cooling to room temperature the solution deposits needle shaped crystals of S-diphenyl urea (m.p. 235-239°C) which is almost insoluble in water but can be recrystallized, if necessary, from alcohol. Melamine resins treated similarly give only a small yield of a white powder (m p.>350°C) which is freely soluble in water.

Polyesters (cross-linking type)

Solubillty : Set products are insoluble. Swollen by acetone, chloroform and alcohol to some extent.

Combustion : Do not melt but may burn with a candle like or sooty flame and evolve characteristic monomers.

Pyrolysis : Do not melt, do not not readily char; a strong fruity but biting odour may be present.

Other Tests : There are no simple chemical tests. Styrene may be distinguished from some polyesters as per the colour test described under polystyrene. In glass fibre reinforced polyester sheets the glass fibre mat or cloth may be seen after the resin is burnt off.

Epoxy

Solubility : Final set products are insoluble.

Combustion : Softens, melts in part, burns with a smoky flame and leaves a charred residue.

Pyrolysis : Chars, melts in part, evolves acidic brown fumes.

Colour Test : Dissolve 0.1gm powdered resin in 10 ml conc. H_2SO_4. To one ml clear solution of this add (1) one ml. conc. nitric acid and after a few minutes pour it into 100 cc dil. NaOH to obtain a bright orange or red colour. (2) 5 ml of Deniges reagent (0.25 gm HgO dissolved in one ml of conc. H_2SO_4, then diluted with 5 ml water). The solution slowly becomes orange coloured and may subsequently yield an orange precipitate.

Polyurethanes

Solubility : Final set products are insoluble

Combution : Softens, melts, may char later; combustion may cease when unassisted.

Pyrolysis : Melts and chars, evolves alkaline vapours.

Colour Test : P-dimethyl amino benzaldehyde in solution or added as powdered to a sample dissolved in or swollen with glacial acetic acid produces a bright yellow colour.

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