

Dinoop Lal S. “Photodegradation of polystyrene by nano titanium dioxide and photosensitizers.” Thesis. Research & Postgraduate Department of Chemistry, St. Thomas’ College (Autonomous), Thrissur, University of Calicut, 2020.

Chapter 2

Materials and Methods

2.1 Materials

The following chemicals were used for the preparation of various photocatalysts.

Chemicals	Manufacturer
• Titanium(IV)isopropoxide	: Sigma Aldrich
• Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$)	: Sigma Aldrich
• Aniline	: Merk, India
• Ammonium peroxodisulphate	: Merk, India
• Graphite (150 mesh)	: Merk, India
• Sodium nitrate ($NaNO_3$)	: Merk, India
• Hydrogen peroxide (H_2O_2) (30% W/V)	: Merk, India
• Potassium permanganate ($KMnO_4$)	: Merk, India
• Silver nitrate ($AgNO_3$)	: Sigma Aldrich
• Iron (III) nitrate nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$)	: Sigma Aldrich
• Copper sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$)	: Sigma Aldrich
• Benzophenone	: TCI chemicals, India
• 2-hydroxy-4-methoxybenzophenone	: TCI chemicals, India
• 4-methoxybenzophenone	: TCI chemicals, India
• 2-chlorobenzophenone	: TCI chemicals, India
• 4-nitrobenzophenone	: TCI chemicals, India
• Malachite green	: TCI chemicals, India
• Methyl blue	: TCI chemicals, India
• Sulphuric acid	: Merk, India
• Hydrochloric acid	: Merk, India
• Nitric acid	: Merk, India
• Ammonium hydroxide	: Merk, India

All the above chemicals were used directly without further purifications for the photocatalyst preparation. Ethanol was distilled in the presence of magnesium ribbon and iodine solution in order to remove the water content.

Detailed Methods of preparation of various photocatalysts are described in the corresponding chapters (chapters 3-7)

PS specimens for photodegradation studies were prepared using the following chemicals.

Chemicals	Manufacturer
• Polystyrene beads	: LG Polymer India Pvt. Ltd
• Toluene	: Merk,India

UV tube (253 nm. 30 W, Phillips Holland) fit inside a wooden chamber (96 cm x 26cm x 34cm) was used as UV light source for the photodegradation studies.

2.2 Preparation of PS and PS-photocatalyst composite sheets

2.2.1 Preparation of PS sheets

PS sheets were prepared through the method of solvent casting. Typically 5g of PS beads were dissolved in 20 ml toluene and the resulting viscous solution was homogenized using ultrasonic probe sonicator (750 W) for 30 minutes. The solution was immediately poured into petri dishes of uniform dimensions and kept overnight in a vacuum oven. The obtained PS sheets were allowed to dry at room temperature for seven days. The PS sheets hence obtained were subjected to photodegradation studies.

2.2.2 Preparation of PS-composite sheets

PS-composite sheets were also prepared through solvent casting method. Photocatalysts of varying weight percentages were loaded into 5g PS dissolved in 20 ml toluene. The viscous solutions were sonicated for 30 minutes, casted in petri dishes, dried in a vacuum oven followed by room temperature drying as explained above. The polymer composite sheets were subjected to UV irradiation for photodegradation studies.

2.2.3 Preparation of PS and PS-composites for mechanical studies

Mechanical tests included determination of tensile and flexural properties. Polymer composites for mechanical studies were prepared by injection moulding technique. Finely chopped PS and PS composite sheets- prepared by solvent casting method discussed above, were fed into injection moulder (Windsor, India) which consisted of moulds corresponding to tensile and flexural test specimens. Dimension of polymer specimens for tensile test was as per *ISO-527-2-1A* standard while that of flexural was as per *ISO-178* standard (Figure 2.1). Two other duplicates were also moulded for each sample. The samples were subjected to UV irradiation after which their tensile and flexural properties were measured using a universal testing machine (UTM).

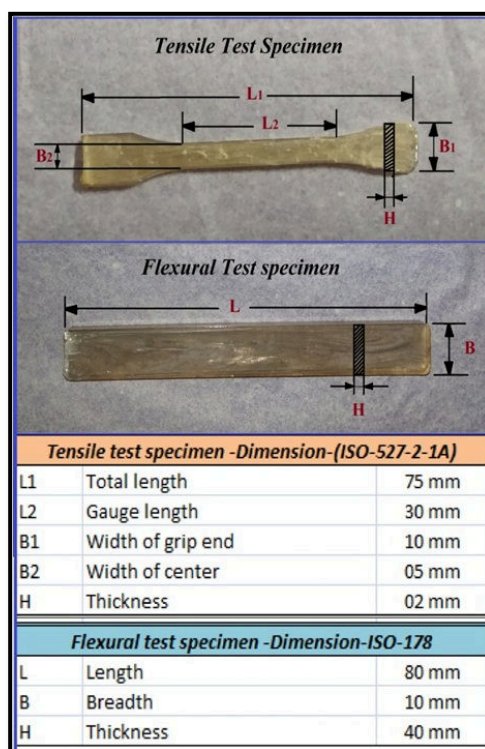


Figure 2.1. Dimensions of injection moulded specimens for tensile and flexural measurements as per ISO standards.

2.2.4 Preparation of PS and PS-composites for electrical studies

Electrical studies included dielectric breakdown (break down voltage) (BDV) measurement and determination of dielectric permittivity (ϵ_r) through capacitance

measurement. Specimens for BDV were moulded by the aid of hydraulic hot press to obtain uniform discs of thickness 1 mm and diameter 75 mm. Polymer sheets were cut out into small circular button shaped discs of diameter 10 mm for the determination of dielectric permittivity. The samples were then exposed to UV irradiation for photodegradation studies followed by electrical testing along with their duplicates.

2.3 Photodegradation reaction setup

Photodegradation of PS and PS composites were studied under artificial UV irradiation. Artificial UV irradiation of the polymer specimens in an isolated chamber avoided the possibility of their degradation through any other weathering process. UV chamber also protected the polymer specimens from natural harsh conditions and hence the impact of UV radiations alone, on photodegradation of PS could be investigated. The polymer specimens were subjected to a uniform UV exposure throughout the study and the rate of photodegradation of each specimen were compared.

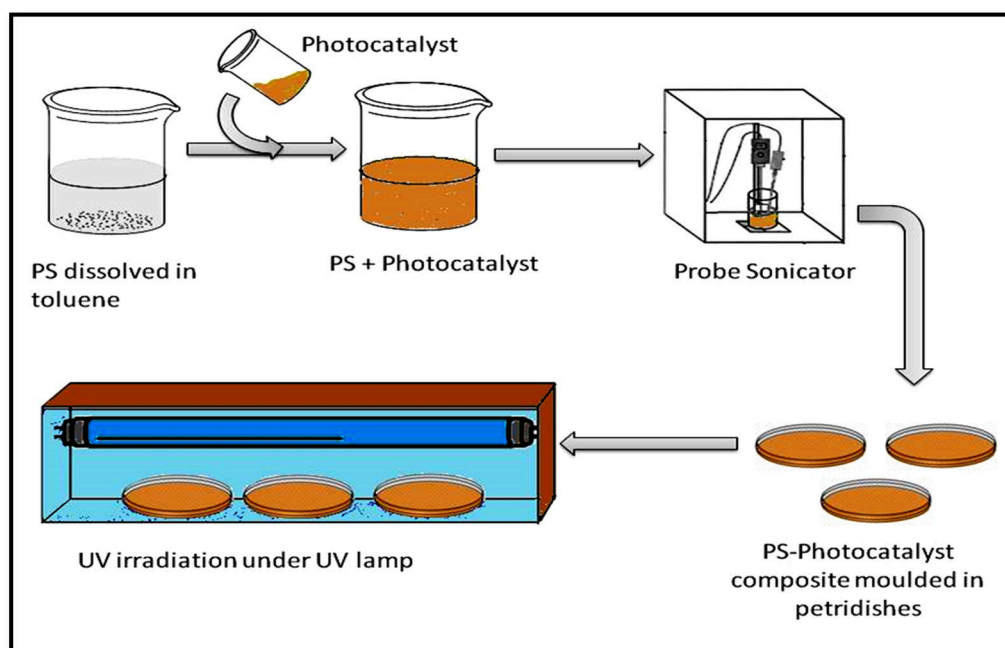


Figure 2.2. Illustration of PS composites preparation and their photodegradation setup

The UV irradiation chamber contained a UV tube (30 W, Philips Holland) of wavelength 253 nm and the specimens were irradiated maintaining an exact distance

of 8 cm from the tube. Irradiation was carried out continuously for 1000 hours. The specimens were subjected to various analyses at regular intervals of 200 hours. Figure 2.2 illustrates the scheme starting from polymer composite preparation to their UV exposure.

2.4 Characterisation techniques

Powder XRD analysis of the photocatalysts was done using X-Ray diffractometer Aeris, Panalytical -with Copper-K alpha radiation (1.5406\AA wavelength) as the source. The particle size and morphology of the prepared photocatalysts were determined using Scanning Electron Microscope (FESEM) Hitachi SU6600 Variable Pressure Field Emission. Elemental analyses were done using energy dispersive X-ray (EDX) instrument JED 2300, Jeol. The surface morphology of PS-composite sheets was determined through SEM instrument, JSM-6390LV, JEOL. High resolution transmission electron microscope (HRTEM) and selected area electron diffraction (SAED) analysis were done using JEM 2100, Jeol. Molecular weight determination of the PS-composites was done using Gel Permeation Chromatography (GPC) LC-20AD, Shimadzu, Japan, with stationary phase silica gel and mobile phase tetrahydrofuran. The samples were dissolved in tetrahydrofuran for GPC analysis. IR spectroscopic analysis was done using FTIR-ATR spectrometer IRAffinity-1S, Shimadzu, Japan. PS composite sheets as such were used for FTIR analysis. UV-DRS analyses were conducted using UV-visible spectrometer UV-2600, Shimadzu, Japan. PS sheets were directly loaded into the instrument for UV-visible spectroscopic analysis. Thermal studies were conducted in a thermogravimetric analyzer (TGA), STA 6000, Perkin Elmer. The TG analysis was done in nitrogen atmosphere. Mechanical measurements were taken using universal testing machine (UTM), Autograph AG-X plus, Shimadzu. The samples moulded as per ISO standard as mentioned above were tested for tensile and flexural properties. Capacitance was measured using LCR meter of frequency range 20 to 10^7 Hz. The value of dielectric constant was determined using the measured value of capacitance. Dielectric breakdown or Breakdown voltage (BDV) was measured using specially designed wooden chamber with two copper electrodes aligned in a head to head fashion with respect to each other (Figure 2.3). The electrodes were connected to the two terminals

of a high power alternating current (AC) voltage source. The disc shaped sample (1 mm thickness) was placed in between the round heads of the electrodes. The chamber was filled with transformer oil so as to immerse the electrodes and sample completely. Transformer oil served as insulator in order to suppress arcing or discharges of electricity which may lead to current leakage and false measurement. Voltage needed to break the sample (BDV) was measured for the polymer samples and their duplicates.

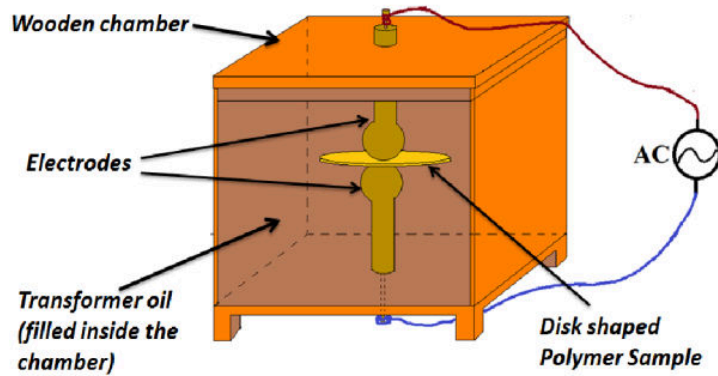


Figure 2.3. Setup for BDV measurement