**LIST OF EXPERIMENTS**

**TPL-303 : RHEOLOGY AND TESTING OF POLYMERS Lab**

1. Determination of Tensile Strength and Percent Elongation of polymer film/sheet
2. Determination of the Vicat Softening point of given plastic sample on Vicat Softening Point apparatus
3. Determination of Tensile strength, Modulus and Percent Elongation of moulded plastic test specimen on Microprocessor Controlled Universal Testing Machine (U.T.M)
4. Determination of the Izod/Charpy Impact Strength of given specimen
5. Determination of the Melt Flow Index of polymer raw material by MFI tester
6. Determination of the Shore A Hardness of Rubber Sheet
7. Determination of the Percent Water Absorption in 24 hours of Moulding Plastic samples
8. Determination of the Falling Dart Impact Strength of polyethylene film using Falling Dart Impact Tester
9. Determination of viscosity of polymer by Brookefield viscometer
10. Determination of polymer yield by microwave synthesizer

**EXPERIMENT NO. -1**

**Object:** Determination of Tensile Strength and Elongation at break of film sample.

**Equipment:**

* + Tensile testing Machine or UTM
  + Thickness Gauge
  + Vernier caliper**s**

**Significance:** The tensile modulus of elasticity is an index of the stiffness of plastic films and can be used to compare the stiffness of different materials. The tensile energy to break (TEB) is the total energy absorbed per unit volume of the specimen up to the point of rupture and can be considered as a measure of toughness. Ultimate elongation values of several hundred percent are common for polymer films. The rate of strain, specimen parameters, and especially flaws may cause large variations in the results. In that sense, caution is advised in utilizing TEB test results for end-use design applications.

**Procedure:**

* Rectangular sample of dimensions 2.54cm width and 15.24 cm length samples are cut in length wise and cross wise direction of film. 5 samples in each direction are taken.
* Each sample is inspected and with the help of thickness gauge the thickness is measured at various points and average thickness is considered.
* Samples are placed and fixed at upper jaws of tensile machine and movable jaws are moved up to fix the lower end of film
* Initial length L0 of film between the jaws is measured with the help of Vernier caliper**s**
* The stretching speed is fixed on the machine and the movable jaw is moved down at constant speed.
* At the breaking of sample, the reading of load in Kgf noted and final length L1 between the jaws is measured with the help of Vernier calipers.

**Calculations:** The following formulae are used for calculation of tensile strength and %

elongation:-

Tensile Strength ( Kgf/cm2  ) = W / ( b x t )

W = Load in Kgf.

B = Width in cm.

T = Thickness in cm.

% Elongation = [ (L1 - L0)/ L0] x 100.

L0 = Initial length of film between the grips in cm.

L1 =Final length of film between the grips in cm.

**Results:** TheTensile Strength of film sample is ------------- Kgf/cm2  and Elongation at break

of film sample is -----------%

**EXPERIMENT NO. - 2**

**Object:** Determination of Vicat Softening Temperature of given plastic sample

**Equipment:** Vicat Softening Temperature Measuring Apparatus

**Specimen**: Use at least two specimens to test each sample. The specimen shall be flat,

between 3 and 6.5 mm thick, and at least 10 by 10 mm in area or 10 mm in

diameter. When necessary to use multiple layers, no more than three layers of

material may be stacked in order to achieve the minimum thickness. The

specimens may be cut from sheet or molded material.

**Significance:** The Vicat softening temperature is the temperature at which a flat-ended needle

of 1mm2 penetrates the specimen to the depth of 1 mm under a specific load. The

temperature reflects the point of softening to be expected when a material is used in an

elevated temperature application. Data obtained by this test method may be used to compare

the heat-softening qualities of thermoplastic materials.

**Procedure:**

* Polymer sheet samples of dimensions 3/4 inch. wide and 1/8 inch. thick are taken. Three specimens are necessary.
* The apparatus for testing Vicat Softening Point consists of a temperature regulated oil bath with flat ended needle penetrater so mounted as to register degree of penetration on a gauge.
* A specimen is placed in the testing apparatus so that penetrating needle rests on its surface at least 1mm from the edge
* A load of 10N or 50N is applied to the specimen
* The specimen is then lowered into an oil bath at 23 degrees C.
* The temperature on the bath is raised at the rate of 500C/ hr. or 1200C/ hr
* The temperature at which the needle penetrates 1 mm is indicated on graduated scale of the apparatus and is considered as the Vicat Softening Point.

**Results:** TheVicat Softening Temperature of given plastic sample is -------------0C

**EXPERIMENT NO. - 3**

**Object:** Determination of Tensile Strength and % elongation of plastic specimen of dumbbell

**Equipment:**

* + Equipment: Tensile testing Machine or UTM
  + Thickness Gauge
  + Vernier caliper**s**

**Test Method:** ASTM D638 or ISO 527

**Significance:** The ability to resist breaking under tensile stress is one of the most important and widely measured properties of materials used in structural applications. The force per unit area (MPa or psi) required to break a material in such a manner is the **ultimate tensile strength** or **tensile strength at break** . The rate at which a sample is pulled apart in the test can range from 0.2 to 20 inches per minute and will influence the results. The test standard to measure tensile properties in the ASTM system is ASTM D638 and in ISO system is ISO 527.

### Tensile Elongation:The ultimate elongation of an engineering material is the percentage increase in length that occurs before it breaks under tension. Ultimate elongation values of several hundred percent are common for elastomers and film/packaging polyolefins. Rigid plastics, especially fiber reinforced ones, often exhibit values under 5%. The combination of high ultimate tensile strength and high elongation leads to materials of high toughness.

### Tensile Modulus of Elasticity:The tensile modulus is the ratio of stress to elastic strain in tension. A high tensile modulus means that the material is rigid - more stress is required to produce a given amount of strain.

**Procedure:**

* The dumbbell sample is taken and its thickness is measured with the help of Vernier caliper**s** in cm.
* Dumbel edges are clamped in fixed and movable grips on Tensile tester.
* Initial length between grip is noted with the help of Vernier caliper**s** in cm.
* The movable grip is moved at constant speed.
* Load and final elongation reading are noted at breaking of specimen with the help of load scale and Vernier caliper**s,** respectively

**Calculations:** The following formulae are used for calculation of tensile strength and %

elongation:-

Tensile strength ( Kgf/cm2 ) = W / (b x t)

W = Load in Kgf.

b = Width in cm.

t = Thickness in cm.

% Elongation = [ (L1 - L0)/ L0] x 100.

L0 = Initial length of film between the grips in cm.

L1 =Final length of film between the grips in cm.

**Results:** TheTensile Strength of polymer sample is ------------- Kgf/cm2  and

Elongation at break is -----------%

**EXPERIMENT NO. - 4**

**Object**: Determination of Izod/ charpy Impact Strength of given plastic sample

**Equipment:** Izod Impact Tester

**Test Method**: **ASTM D256 or** ISO 180

**Specimen**:**:**. Sample thickness is usually 1/8 in. (3.2 mm) but may be up to 1/2 in. (12.3 mm).

**Significance:** A pendulum swings on its track and strikes a notched, cantilevered plastic

sample. The energy lost (required to break the sample) as the pedulum continues on its

path is measured from the distance of its follow through. The impact value of the material

is determined from the energy required to break the specimen. The impact value can be

used as a rule of thumb for determining the load bearing capacity of a material against

momentary stress from impact strength and fracture energy. The higher the impact value of

a material is, the higher the toughness or tenacity of the material is. The result of the Izod

test is reported in energy lost per unit of specimen thickness (such as ft-lb/in or J/cm) at the

notch. Polymeric materials that are sensitive to the stress concentrations at the notch ('notch-

sensitive') will perform poorly in the notched izod test. Engineers use this knowledge to avoid

using such polymers in designs with high stress concentrations such as sharp corners or cutouts.

Unnotched specimens are also frequently tested via the Izod impact method to give a more

complete understanding of impact resistance. Izod impact tests are commonly run at low

temperatures - down to -40°F (-40°C) or occasionally lower - to help gauge the impact resistance

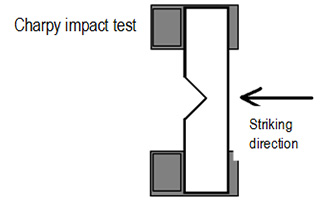
of plastics used in cold environments.

**Procedure:**

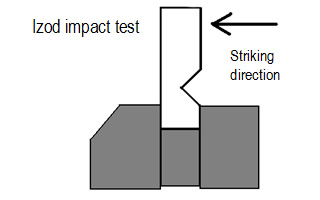
* Sample is clamped in the base of pendulum testing machine so that it is cantilevered upward in notch facing the direction of impact.
* Pendulum is released and the force consumed in breaking the sample is calculated from the height. the pendulum reaches on the follow through.
* Impact load is reported as Kg/ inch of notch.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Testing method | Testable ranges | Test specimen | Data to be obtained | Corresponding standards |
| Charpy impact | Hammer capacity: 0.5,1,2,4,7.5,15J Test temp.: -40 - 150℃ Impact speed： 　0.5～4J 　2.9(±5%)m/sec 　7.5J、15J　3.8(±5%)m/sec | 80.0±2×10.0±.02 ×4.0±0.2mmt n=5 | Fracture energy〔J〕 Impact strength〔kJ/m2〕 | JIS K7111-1 (ISO 179-1) JIS K6745 JIS K6911 |
| Izod impact | Hammer capacity: 　1,2.75,5.5J 　40,80,150kg・cm Test temp.: -40 - 150℃ Impact speed: 3.5(±10%)m/sec | 80.0±2×10.0±0.2 ×4.0±0.2mmt 63.5±0.5×12.7±0.1 ×2 - 13mmt n=5 | Fracture energy〔J〕 Impact strength〔J/m、kJ/m2〕 | JIS K7110 (ISO 180) ASTM D256 |

A test specimen having a V-shaped notch is placed on the holder in such position that the notched section is in the center of the holder, and the specimen is broken by striking the back of the notched section with the hammer. The fracture energy is determined from the swing-up angle of the hammer and its swing-down angle. The Charpy impact value (kJ/m2) is calculated by dividing the fracture energy by the cross-section area of the specimen.



A test specimen having a V-shaped notch is fixed vertically, and the specimen is broken by striking it from the same side as that of the notch by the use of the hammer. The fracture energy is determined from the swing-up angle of the hammer and its swing-down angle. The Izod impact value (J/m, kJ/m2) is calculated by dividing the fracture energy by the width of the specimen



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**Results:** Theof given Izod/charpy Impact Strength plastic sample is -------------J/m

**EXPERIMENT NO. - 5**

**Object:** Determination of melt flow index of polymer material.

**Equipment**: M F I Tester

**Specification:** ASTM D 1525.

**Significance:** The melt flow index (MFI) or melt flow rate (MFR) is a measure for the ease

of flow of melted plastics. It is often used in the plastic industry for quality control of

thermoplastics. The method is described in the standards ASTM D1238 and ISO 1133.

The melt flow indexer is the most popular device in the plastic industry to determine

material viscosities and is often used to test batch-to-batch consistency. However, it is also

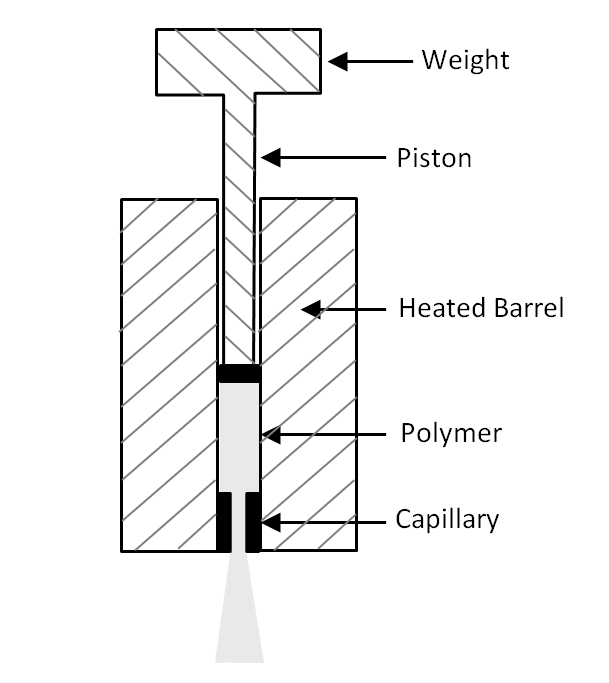
the least accurate method. A small sample of about 5 grams is heated above its melting or

softening point and forced to flow through a capillary using a piston actuated by a specified

weight, usually 2.16 kg or 5 kg. The weight of melt in grams flowing through the capillary

in 10 minutes is the melt flow index.

### MELT FLOW INDEXER



In general, a higher MFI indicates a lower material viscosity, and when comparing

polymers of the same class, a lower melt flow rate corresponds to a higher molecular

weight and/or less branching.

**Procedure:**

* A small amount of the polymer sample (around 4 to 5 grams) is taken in the specially designed MFI apparatus. A [die](https://en.wikipedia.org/wiki/Die_(manufacturing)) with an opening of typically around 2 mm diameter is inserted into the apparatus.
* The material is packed properly inside the barrel to avoid formation of [air](https://en.wikipedia.org/wiki/Air) pockets.
* A piston is introduced which acts as the medium that causes extrusion of the molten polymer.
* The sample is preheated for a specified amount of time: 5 min at 190 °C for [polyethylene](https://en.wikipedia.org/wiki/Polyethylene) and 6 min at 230 °C for [polypropylene](https://en.wikipedia.org/wiki/Polypropylene).
* After the preheating a specified weight is introduced onto the piston. Examples of standard weights are 2.16 kg, 5 kg, etc.
* The weight exerts a force on the molten polymer and it immediately starts flowing through the die.
* A sample of the melt is taken after the desired period of time and is weighed accurately.
* MFI is expressed in grams of polymer per 10 minutes of duration of the test.

**Results:** TheMelt Flow Index of the given polymer sample is -------------0C

**EXPERIMENT NO. - 6**

**Object:** Determination of Shore A Hardness of given Rubber Sheet

**Equipment:** Shore A Durometer

Test Method: ASTM D2240 00, ISO 7619 and ISO 868

**Significance:** The hardness of rubbers is most commonly measured by the Shore A Durometer test. The durometer measures the resistance of rubbers toward indentation and provide an empirical hardness value and is the preferred method for rubbers/elastomers and is also commonly used for 'softer' plastics such as polyolefins, fluoropolymers, and vinyls. The Shore A scale is used for 'softer' rubbers while the Shore D scale is used for 'harder' ones. The hardness value is determined by the penetration of the Durometer indenter foot into the sample. Because of the resilience of rubbers and plastics, the indentation reading my change over time - so the indentation time is sometimes reported along with the hardness number

The results obtained from this test are a useful measure of relative resistance to indentation of various grades of rubbers. However, the Shore Durometer hardness test does not serve well as a predictor of other properties such as strength or resistance to scratches, abrasion, or wear, and should not be used alone for product design specifications. Shore hardness is often used as a proxy for flexibility (flexural modulus) for the specification of elastomers. The correlation between Shore hardness and flexibility holds for similar materials, especially within a series of grades from the same product line, but this is an empirical and not a fundamental relationship.

**Procedure:**

* Take Shore A Hardness Tester and calibrate with standards.
* Place the 5-10 mm. thick rubber sheet sample on a hard platform.
* By applying the force in a consistent manner, without shock, press the apparatus by placing the needle of tester on sample and read the Shore A scale.
* Repeat on several points and take the average.

**Result:** The average Shore A Hardness of the given sample of rubber sheet is -------**.**

**EXPERIMENT NO. - 7**

**Object:** Determination of the percent water absorption in 24 hours of moulded Plastic

samples.

**Equipment Used:** Mettler balance

**Specimen :** Two inch diameter disks, 0.125" or 0.250" thick. 

**Test Method:** ASTM D570

**Scope:** Water absorption is used to determine the amount of water absorbed under specified conditions. Factors affecting water absorption include: type of plastic, additives used, temperature and length of exposure. The data sheds light on the performance of the materials in water or humid environments Some polymers have a natural tendency to absorb water. Indeed, superabsorbent polymers are gaining traction in advanced application in medical, construction etc., however at the same time, absorption capacity of thermoplastics lead to several **changes w.r.t processing and properties.   
  
Moisture/water absorption** is the capacity of a plastic or a polymer to absorb moisture from its environment. Absorbed moisture has been shown to act as a plasticizer, reducing the [glass transition temperature](https://omnexus.specialchem.com/polymer-properties/properties/glass-transition-temperature?src=omproperty) and strength of plastic – which is a reversible effect. However, absorbed water also can lead to irreversible degradation of the polymer structure. Some of the known effects include:Dimensional & mass changes (e.g. swelling) caused by water absorption; extraction of water-soluble components and changes in mechanical (elasticity, tensile strength, impact strength) and electrical performance

**Procedure:**

* For the water absorption test, the specimens are dried in an oven for a specified time and temperature and then placed in a desiccator to cool.
* Immediately upon cooling the specimens are weighed.
* The material is then emerged in water at agreed upon conditions, often 23°C for 24 hours or until equilibrium.
* By finding increase in weight the % water absorption is tested.

**Result:** The percentage of water absorption is ------------

**EXPERIMENT NO. - 8**

**Object:** Determination of Impact Strength of a Polymer Film by Falling Dart Impact Taster

**Test Method:** As per IS-2508-1984, ASTM D1709, ISO 7765-1

**Specimen:** Film samples that can be cut to 230 mm x 230 mm (9" x 9") specimens  
 A minimum of 30 specimens are required for the test

**Scope:** Falling dart impact is a traditional method for evaluating the impact strength or toughness of a plastic film. This test uses a single dart configuration and a single drop height, while varying the weight of the dart. Test results can be used either as a quality control evaluation or for end use comparisons.

**Procedure:**

* Measure the thickness of the film in Microns and decide the Impact Loads as per IS Table.
* Take 10 samples of 10x10 inch sized and clamp the test specimen securely in a pneumatic ring at the base of the drop tower. The mounting bracket is adjusted to the appropriate drop height, and the dart is inserted into the bracket.
* Create the Vacuum and release the dart onto the center of the test specimen from the distance fixed as per IS.
* Observe the sample for failure and repeat the rest 9 samples for same load.
* Calculate the number of failures per 10 samples e.g. 8 pass /2 fail.
* Repeat for another 10 samples for load higher to previous one.
* Report the Load in Grams at which 50 % samples fail.

**Result:** The falling dart impact strength of given plastic film is ---------

**EXPERIMENT NO. - 9**

**Object:** Determination of viscosity of polymer by Brookefield viscometer

**Specimen:** Concentrated polymer solution

**Test Method:** ASTMD2196

**Scope:** Brookfield viscosity usually refers to a viscosity measurement performed with a Brookfield Viscometer. The viscometer measures the resistance to rotation and reports a viscosity value.  Various spindle designs can be employed, depending on the nature of the sample and the requirements

### Dip-in LV Spindles & RV Spindles

Labelled as LV or RV spindle sets, these comprise simple shafts ending in a disk or cylinder. A sample of 400 - 600ml in a suitable container is placed under the viscometer which is then lowered to dip the spindle into the sample up to an immersion mark on the spindle shaft. The dip-in spindle is suitable for comparative testing of the viscosity of free-flowing fluids.

### Small Sample Adapter (SSA)

The Small Sample Adapter is a concentric cylinder measuring system, often know as a coaxial or "cup and bob" system.  In the small sample adapter a small sample (typical only 5 - 10ml) is sheared between the moving inner cylinder and stationary outer cylinder or chamber.  The accessory was, as its name suggests, originally designed for measuring samples of limited quantity however due to its fairly narrow clearances the system operates in a degree of defined shear - a valuable capability of you wish to perform viscosity / shear rate profiles on a sample.

**PROCEDURE :**

* Check the spirit level of the viscometer everytime before use. The level is adjusted by using the two leveling screw on the base. Adjust so that the spirit level on top of the instrument is centered with in the circle.
* Ensure the calibration status is valid and turn the power switch located on the rear panel to the ‘ON’ position.This will result in different screen displays and after a few seconds the screen displays remove spindle, press any key.
* After removing the spindle ( if attached to it ) and pressing any key, the instrument begins its auto zero. The screen will blink “AUTOZEROING”.  
  After a few seconds, the blinking stops and the following is displayed REPLACE SPINDLE, PRESS ANY KEY, pressing any key at this point result is in the display of the instrument default screen.
* Connect the selected spindle to the viscometer by screwing them to the lower shaft. The lower shaft should be hold in one hand and lifted up and the spindle should be screwed to the left.
* Spindles are identified by the number on the side of the spindle nut.  
  The two digit entry code for the selected spindle should be entered by pressing the SELECT SPINDLE key, on pressing it once, the top line of the display will begin to blink.  
  Then press the UP or DOWN arrow keys for increasing or decreasing the number which is to the right of the ‘S’ character while ‘S’ is blinking, until the requirement spindle value is displayed.  
  The two digit entry code for each spindle is given below.  
  SPINDLE NUMBER 1 (LV1)  
  TWO DIGIT CODE 61  
  SPINDLE NUMBER 2 (LV2)  
  TWO DIGIT CODE 62  
  SPINDLE NUMBER 3 (LV3)  
  TWO DIGIT CODE 63  
  SPINDLE NUMBER 4 (LV4)  
  TWO DIGIT CODE 64
* Then press the SELECT SPINDLE key once again after selecting the desired spindle code. This will cause the ‘S’ character to cease blinking and the new spindle code will be displayed and accepted for use.
* Then select the speed for the spindle by first pressing either the UP or DOWN arrow keys, which will cause the area to the right of RPM (on the bottom line) to display the currently selected speed. If the arrow key is pressed just once and then released, the characters RPM will blink for a few seconds and then will cease blinking, resulting in no change to the speed entry.
* Press the UP or DOWN arrow key until the desired speed in displayed and then release it.  
  Then press the AUTO RANGE key, which display the maximum calculated viscosity (full scale reading) which can be measured with the current spindle / speed setting.  
  If the viscosity of the test fluid is greater than the AUTO RANGE displayed, then “ cP EEEE” and ‘%EEEE’ will be displayed when operated with this test fluid.
* Change either the spindle or speed to achieve a maximum accuracy with the auto range.  
  Pressing and holding the AUTO RANGE key during power on will enable the viscosity display tot he read cPs or m.pas. 1 cPs = 1milli pascal.  
  Insert and center spindle in the test material until the fluid level is at the immersion groove in the spindle shaft. Press the MOTOR ON / OFF key once to turn the motor ON.  
  Allow time for the indicated reading to stabilize. For maximum accuracy, readings below 10% torque must not be taken.
* Press the MOTOR ON / OFF key once again to turn the motor OFF when changing a spindle / speed or changing samples.
* Remove the spindle before cleaning and clean it after use.

**Result:** The Brookefield viscocity of given polymer sample is--------

**EXPERIMENT NO. - 10**

**Object:** Determination of polymer yield by microwave synthesizer

**Specimen**: Polar monomers, initiators and modifiers

**Scope:** Microwave ovens operate with electromagnetic nonionizing radiation with frequencies between 300 GHz and 300 MHz. The corresponding wavelengths span a range from 1 mm to 1 m, exhibiting the medial position of microwaves between infrared and radio waves. Most commercial microwave systems, however, utilize an irradiation with a frequency of 2 450 MHz (wavelength l ¼ 0.122 m) in order to avoid interferences with telecommunication devices. The corresponding electric fields oscillate 4.9 109 times per second and consequently subject dipolar species and ionic particles (as well as holes and electrons in semiconductors or metals) to perpetual reorientation cycles. This strong agitation leads to a fast noncontact heating that is (approximately) uniform throughout the radiation chamber.

A large number of reactions, both organic and inorganic, undergo an immense increase in reaction speed under microwave irradiation compared with conventional heating. Apart from this main advantage, significant improvements in yield and selectivity have been observed as a consequence of the fast and direct heating of the reactants themselves. Furthermore, high-pressure synthesis is easily

The main advantages of MW-assisted chemistry are shorter reaction times, higher yields, and a reduction of side reactions compared with syntheses performed under conventional heating. **Procedure:**

* Take the flask of MW reacor
* Add the required quantities of monomers, initiator and moifiers
* Set the required conditions in the reactor and start the reaction
* Stop the reaction after set time period and convert them into dry polymer
* Determine the yield

**Result:** The percent yield of the MW synthesis is ----------------