

PROJECT REPORT

On

“SYNTHESIS AND CHARACTERIZATION OF PMMA ”

Submitted by

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*In partial fulfillment for the award of the
BSc Degree in Chemistry*



**POST GRADUATE AND RESEARCH
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**ST. TERESA'S COLLEGE (AUTONOMOUS)
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DECLARATION

We hereby declare that the project work entitled “SYNTHESIS AND CHARACTERIZATION OF PMMA” submitted to Department of Chemistry, St. Teresa’s College (Autonomous) affiliated to Mahatma Gandhi University, Kottayam, is a record of an original work done by me under the guidance of MARY LINCY K A, ASSISTANT PROFESSOR, Department of Chemistry, St. Teresa’s College (Autonomous), Ernakulam and this project work is submitted in the partial fulfillment of the requirements for the award of the degree of Bachelor of Science in Chemistry.

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Chapter 1

Introduction

GENERAL INTRODUCTION

1.1 POLYMERS

Polymer is a long chain molecule which is composed of large number of repeating units called monomers. Certain polymers such as proteins, cellulose, and silk are found in nature, while many others including polystyrene, polyethylene and nylon are produced only by synthetic routes. They find many applications such as in the commercial production of plastics, man-made fibers, adhesives, surface coatings, elastomeric polymers etc. The process by which polymers are formed from monomers is called polymerization [1].

1.2. CLASSIFICATION

Polymers are classified on the basis of source, structure, mode of polymerization and molecular forces.

1.2.1 Based on source

(i) Natural polymers

Natural polymers are polymers which occur in nature and in natural sources like plants and animals. Some common examples are Proteins, Cellulose, Starch, or Rubber (which we harvest from the latex of a tropical plant).

(ii) Synthetic polymers

These are polymers which can be artificially synthesized in lab. They are commercially produced by industries for human necessities. Some commonly produced polymers which we use day to day are Polyethylene or Nylon Fibers.

(iii) Semi-Synthetic polymers

Semi-Synthetic polymers are polymers obtained by making modification in natural polymers artificially in a lab. These polymers are formed by chemical reaction (in a controlled environment) and are of commercial importance. Example: Vulcanized Rubber, Cellulose acetate (rayon) etc.

1.2.2 Based on Structure of Polymers

(i) Linear polymers:

These polymers are similar in structure to a long straight chain which identical links connected to each other. The monomers in these are linked together to form a long chain. These polymers have high melting points and are of higher density. A common example of this is PVC (Poly-vinyl chloride). This polymer is largely used for making electric cables and pipes.

(ii) Branched chain polymers:

Monomers join together to form a long straight chain with some branched chains of different lengths. As a result of these branches, the polymers are not closely packed together. They are of low density having low melting points. Low-density polythene (LDPE) used in plastic bags and general purpose containers is a common example.

(iii) Cross linked or Network polymers:

In this type of polymers, monomers are linked together to form a three-dimensional network. The monomers contain strong covalent bonds as they are composed of bi-functional and tri-functional in nature. These polymers

are brittle and hard. Bakelite and melamine are common examples of network polymers [2].

1.2.3 Based on Mode of Polymerization

i) Addition polymers:

These type of polymers are formed by the repeated addition of monomer molecules without elimination of any small molecules like water, alcohol etc. Formation of polythene from ethane is an example for addition polymerization.

ii) Condensation polymers:

These polymers are formed by the combination of monomers, with the elimination of small molecules like water, alcohol etc. The monomers in these types of condensation reactions are bi-functional or tri-functional in nature. A common example is the polymerization of Hexamethylenediamine and atopic acid to give Nylon – 66.

1.2.4 Based on Molecular Forces

i) Elastomers

Elastomers are rubber-like solid polymers that are elastic in nature. The polymer chains are held by the weakest intermolecular forces, hence allowing the polymer to be stretched.

ii) Thermoplastics

Thermoplastic polymers are long-chain polymers in which the intermolecular forces (van der Waal's forces) hold the polymer chains together. These polymers when heated are softened and hardened when they are allowed to cool down, forming a hard mass. They do not contain any cross bond and can easily be shaped by heating and using moulds. A common example is Polystyrene or PVC (which is used in making pipes).

iii) Thermosetting plastics

Thermosetting plastics are polymers which are semi-fluid in nature with low molecular masses. When heated, they start cross-linking between polymer chains, hence becoming hard and infusible. They form a three-dimensional structure on the application of heat. This reaction is irreversible in nature.

The most common example of a thermosetting polymer is, Bakelite, which is used in making electrical insulation.

iv) Fibers

Fibers are a class of polymers which are a thread like in nature, and can easily be woven. They have strong inter-molecular forces between the chains giving them less elasticity and high tensile strength. The intermolecular forces may be hydrogen bonds or dipole-dipole interaction. Fibers have sharp and high melting points. Example: Nylon 6,6 [3].

1.3. SYNTHESIS OF POLYMERS

Polymerization may be carried out with monomer alone (bulk polymerization), in a solvent (solution polymerization), as an emulsion in water (emulsion polymerization), or as droplets, each one comprising an individual bulk polymerization, suspended in water (suspension polymerization). These methods are commercially applied to radical initiated chain polymers such as polystyrene, PMMA [4]. Gas phase polymerization

and interfacial condensation are special techniques which are mentioned under solution polymerization.

1.3.1 BULK POLYMERIZATION

Bulk polymerization or mass polymerization is carried out by adding a soluble radical initiator to pure monomer in liquid state. This reaction is initiated by heating or exposing to radiation. It is an exothermic reaction and wide range of molecular masses is produced. Depending on the solubility of the growing polymer chains in the monomer the bulk reactivity of step growth polymers favors bulk polymerization.

1.3.2 SOLUTION POLYMERIZATION

Here the monomer is dissolved in suitable solvent along with chain transfer agents and initiators. Catalyst involved may be ionic or coordination catalyst which can either dissolved or suspended. It offers easier temperature control because of added heat capacity of solvent and lower viscosity. The product obtained is in liquid form and can be directly used for applications such as coating and adhesives.

1.3.3. EMULSION POLYMERIZATION

This technique is well suited for addition polymerization. Apart from dispersed and dispersion media several other components are added. The essentials of an emulsion polymerization system are monomer, a surface active agent (surfactant), initiator and water. Temperature control is more convenient in this polymerization, since the viscosity changes very little with conversion and also the thermal conductivity and specific heat of water are higher than those for organic solvents.

1.3.4. SUSPENSION POLYMERIZATION

This polymerization is essentially a bulk polymerization carried out in droplets. It is employed to overcome some of the disadvantages of emulsion polymerization like the tendency of small polymer particles to absorb impurities. The size of the monomer absorbed is greater when compared to emulsion polymerization. Polymer recovery is simple and usually leads to lower impurity level than with emulsion polymers [5].

1.4 CHARACTERIZATION OF POLYMERS

Polymer characterization involves measuring any kind of property of a polymer material. It includes both molecular characterization and

macroscopic property measurement. Some of the characterization techniques are:

1.4.1. Thermo-gravimetric analysis (TGA)

This is a method of thermal analysis which measures the amount and rate of change in the mass of a sample as a function of temperature or time in a controlled atmosphere. It is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation, or loss of volatiles. TGA can provide information about physical phenomena such as phase transitions, absorption, and desorption as well as chemical phenomena including chemisorptions, desolvation, decomposition and solid gas reactions. The basic principle of TGA is that as a sample is heated, its mass changes, hence it can be used to determine the thermal stability up to 1000°C. It is especially useful for the study of polymeric materials such as thermoplastic, thermosets, elastomers, films, fibers, composites, coatings and paints [6].

1.4.2. Fourier Transform Infrared Spectroscopy (FTIR)

IR spectroscopy is a technique mainly for the qualitative analysis. The information regarding the structure of the molecule is obtained frequently by its absorption spectrum. The intensity of the absorption spectrum thus

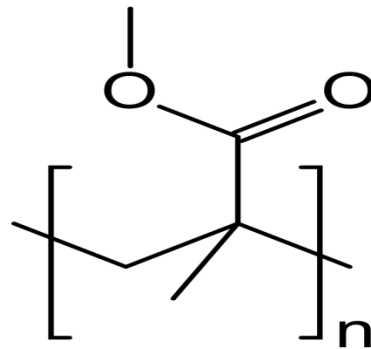
obtained depends upon the size of the dipole moment change associated with the vibration [7]. The intensity can also depend on the concentration of the sample under observation. The IR spectrometry has got many advantages like high resolution, more accurate frequency wavelength measurement etc.

1.4.3. Gel Permeation Chromatography (GPC)

GPC is an analytical technique that separates dissolved macromolecules by size based on their elution from columns filled with a porous gel.

1.5 POLYMETHYL METHACRYLATE

Poly(methyl methacrylate) PMMA which is chemically the synthetic polymer of methyl methacrylate is a transparent thermoplastic that are being used in the form of sheet as light weight or shatter resistant alternative to glass. It is generally classified as a hard, rigid but brittle material that has a glass transition temperature of 105°C [8]. It has got many excellent properties like weather resistance, light weight, impact and shelter resistance. It is always used as an alternative to glass due to its transparency.



It can be molded in number of ways which helps for the easy and low cost manufacture of complex optics. It is considered as an ideal one for its application in the field of optical equipments, building, and household electric appliances and also it has many profound and wide uses that affect our day to day life. PMMA plays an important role in every aspect as it posses tremendous properties like:

- Excellent transparency and gloss.
- Greater surface hardness among plastic components.
- It is a polar material and has high dielectric constant.
- PMMA is an amorphous solid thus it is soluble in solvents with similar parameters such as benzene, toluene, ethyl acetate.
- It absorbs a very little visible light and gives a reflection of 4% for every polymer for normal incident light.

1.5.1 SYNTHESIS OF PMMA

PMMA is commonly produced by the bulk polymerization, solution polymerization, and emulsion polymerization. Anionic polymerization and radical polymerization are generally employed. PMMA that are synthesized from radical polymerization is atactic and fully amorphous. Here we prefer the bulk polymerization method.

- Bulk polymerization

This method is generally employed to obtain the purest form of polymer. The highest yield of polymer per unit volume can also be obtained through this process. A single monomer molecule, initiator and a chain transfer agent are involved in this method. Casting objects of various shapes may be achieved using this polymerization method. It is used for most step-growth polymers and many types of chain-growth polymers. Bulk polymerization have got many advantages:

- Simple and involves thermal isolation
- Polymer obtained is pure
- Changes can be made in the molecular weight distribution
- The product obtained has high optical clarity.

1.5.2. APPLICATIONS OF PMMA

1. TRANSPARENT GLASS SUBSTITUTE

- Used for constructing aquarium.
- Used for viewing ports and pressure hulls of submarines.
- Used in the lenses of exterior lights of automobiles.
- In ice hockey rinks, spectator protection is made from PMMA.
- Used in the designing of aircraft windows.
- For riot control, police vehicles have the regular glass replaced with PMMA to protect the occupants from thrown objects.
- Used in the making of certain lighthouse lenses [9].

2. DAYLIGHT REDIRECTION

- Laser cut acrylic panels can be used to redirect the sunlight into a light pipe (tubular) and from there, to spread it into a room.
- Acrylic broadband light guides are dedicated to decorative use.
- Pairs of acrylic sheets with a layer of micro replicated prisms between the sheets have both reflective and refractive properties that allow them to redirect the part of incoming sunlight in

dependence on its angle of incidence. Such panels can act as mini light shelves which can be used as a window such that light descending from the sky is directed to the ceiling or into the room rather than to the floor [10].

3. MEDICAL TECHNOLOGIES AND IMPLANTS

- PMMA has a good affinity with human tissue and is used in the manufacture of rigid intraocular lenses which can be implanted in the eye when the original lens has been removed during the treatment of cataract [11].
- In orthopedic surgery, PMMA bone cement is used to fix implants and to remodel lost bone [12].
- In cosmetic surgery, tiny PMMA microspheres suspended in some biological fluid are injected as a soft tissue filler under the skin to reduce wrinkles or scars permanently.
- Biotechnology and biomedical research uses PMMA to create micro fluidic lab-on-a-chip devices.
- Bioprocess chromatography columns use cast acrylic tube as a substitute to glass and stainless steel. These are pressure rated and satisfy firm requirements of materials for biocompatibility,

toxicity.

- Due to its biocompatibility, PMMA is used as a material in modern dentistry, particularly in the fabrication of dental prosthetics, artificial teeth and orthodontic appliances [13].

1.5.3 SIDE EFFECTS OF PMMA

PMMA have more toxic effects [14]. It also takes longer to feel these effects, so people may take another pill in the mistaken belief that the first has not worked, sometimes resulting in overdose.

PMMA affect everyone differently, but effects may include

- Feeling alert and excited
- Seeing colours and shapes
- Heightened senses like sight , hearing and touch
- Dry mouth
- Teeth grinding
- Increased sweating
- Difficulty in breathing
- Irregular eye movement
- Muscle spasms
- Kidney failure
- Extremely high body temperature

- Vomiting
- Coma

Chapter 2

Materials and Methods

Introduction

This chapter comprehends on the materials and methods used for the synthesis of PMMA. The various studies done on the PMMA is also summarized.

2.1 PREPARATION OF PMMA

2.1.1 Materials

Reagents for the preparation of PMMA such as:

- MMA,
- 2% NaOH
- Benzoyl peroxide
- Toluene
- Alcohol

2.2 Method

2.2.1 Preparation of PMMA through bulk polymerization

About 20 ml of methyl methacrylate (MMA) was washed thrice with 10 ml of 2% NaOH and then thrice with 20 ml of distilled water. It is then

transferred into 250 ml beaker. To this about 1.2 g of benzoyl peroxide was added and then heated with stirring at 60°C to initiate polymerization. The reaction mixture is stirred until it becomes highly viscous. It is then allowed to dry at room temperature. After this the PMMA hence obtained was dissolved completely in toluene. It was recrystallized with alcohol [15].

2.3 Characterization of PMMA

2.3.1 Thermo gravimetric analysis (TGA)

TGA is an analytical technique used to determine a material's thermal stability and its fraction of volatile components by monitoring the weight change that occurs as a sample is heated at constant rate [16].

2.3.2 Gel permeation chromatography (GPC)

GPC is a type of size exclusion chromatography (SEC) that separates analytes on the basis of size. It is the most convenient technique for characterizing the complete molecular weight distribution of a polymer. GPC can determine several important parameters like number average molecular weight (M_n), weight average molecular weight (M_w) [17].

2.3.3 Fourier-Transform Infrared Spectroscopy

FTIR is a technique used to obtain an infrared spectrum of absorption or emission of solid, liquid or gas. It relies on the fact that the most molecule absorb light in the infrared region of the electromagnetic spectrum. This

absorption corresponds specifically to the bonds present in the molecule. The frequency ranges are measured as wave numbers typically over the range 4000-600 cm^{-1} [17].

Chapter 3

Results and discussion

This chapter comprehends on the results obtained from the current studies on the analysis of PMMA. Many methods can be employed for the preparation of PMMA. It was prepared by the addition of benzoyl peroxide as the initiator.

3.1 TGA Analysis of PMMA

The influence of organic initiator on thermal stability of PMMA was studied by the thermogravimetric analysis. Thermogravimetric measurements provide a qualitative means of estimating the thermal stability of polymeric materials [16]. Thermal stability is defined as the onset temperature of degradation and the rate of degradation of the respective materials.

TGA graph of PMMA is represented in Fig.1. Poly Methyl Methacrylate shows stability up to 100°C. At 167°C PMMA shows weight loss by 8%. At 280°C PMMA shows weight loss by 25%. At 310°C PMMA shows weight loss by 52%. It is observed that as temperature increases the rate of decomposition of PMMA increases and at 410°C PMMA completely burns. The thermal degradation of PMMA also leads to the production of

char, which was produced by the elimination of methoxycarbonyl side chains. The amount of char produced increased with increasing temperature.

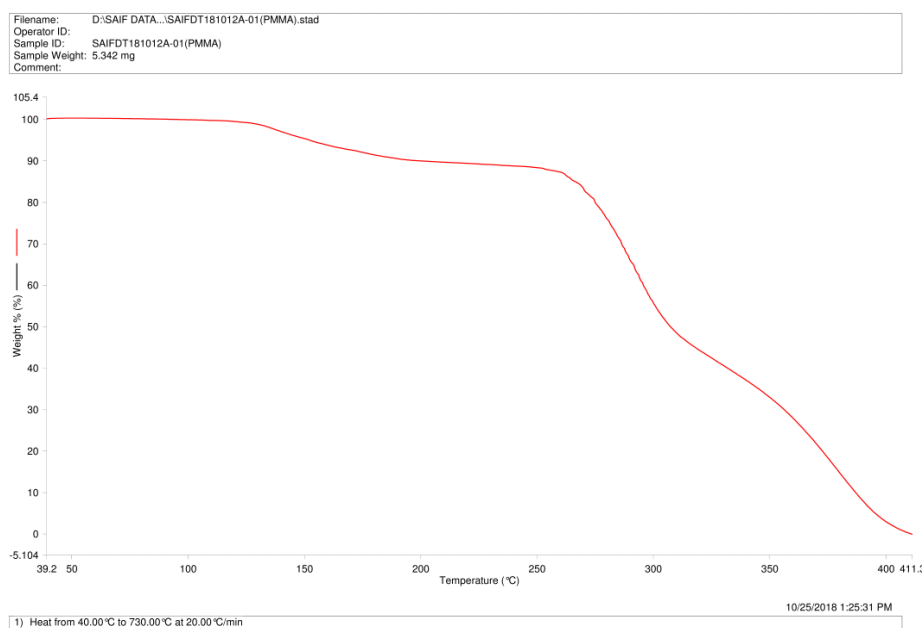


Fig .1

3.2 FTIR Analysis of PMMA

Infrared spectroscopy has been used for qualitative determination of the products of degradation. Since the products of degradation of PMMA are known to consist principally of monomer, the determination of C=C group can be an indication of the degree of degradation or the degree of reverse polymerization [17]. FTIR spectra of PMMA are represented in Fig .2. The main peaks for PMMA can be assigned as follows: alcoholic OH

stretching vibration of PMMA is observed at 3434.72cm^{-1} , the CH_3 symmetric stretch is observed at 2930.58cm^{-1} , the peak at 1727.69cm^{-1} is the strong α - β unsaturated ester bond. The peak at 1630.08cm^{-1} is due to the $\text{C}=\text{O}$ stretching vibration. The peak observed at 1389.84cm^{-1} is the bending vibration of CH_3 . The peak at 1115.16cm^{-1} is due to the $\text{C}-\text{O}$ stretching vibration.

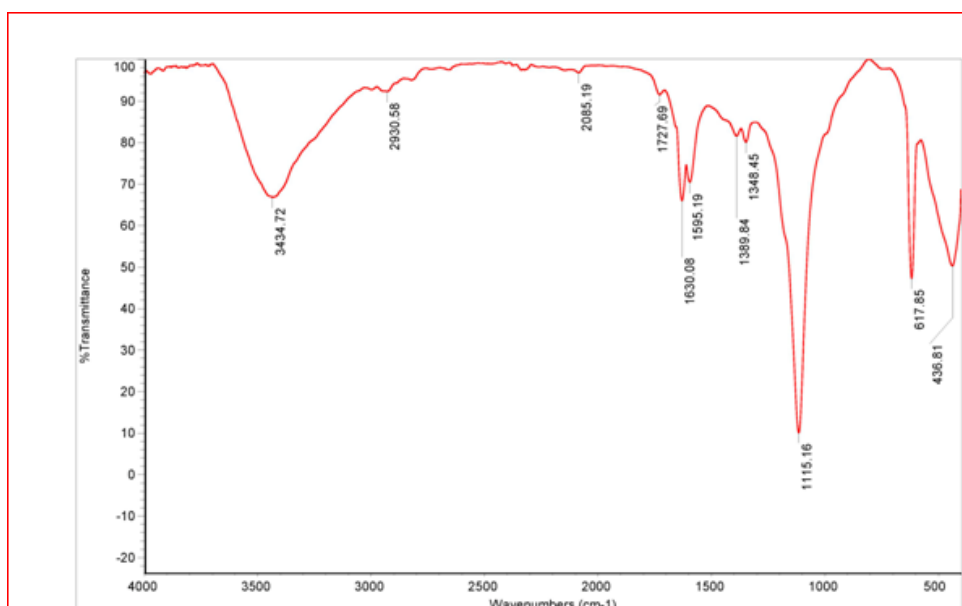


Fig .2

3.3 GPC Analysis of PMMA

The GPC method is used widely to measure molecular weights of linear polymers. High- quality GPC data contains detailed information on many aspects of the polymer's molecular weight distribution [17]. From the GPC analysis of PMMA it was found that, the number average molecular

weight (M_n) is 23166 and the weight average molecular weight (M_w) is 26070. Thus the polydispersity index (M_w/M_n) of PMMA is equal to 1.12535.

3.4 Visible Observation



Bulk polymerized PMMA

Chapter 4

Conclusions

PMMA is a material which has wide applications in many areas such as medical, industrial and analytical field of Chemistry. In our project PMMA was polymerized successfully through bulk polymerization with Methyl methacrylate as the monomer and benzoyl peroxide as the initiator. The PMMA thus obtained was characterized successfully through FTIR, TGA and GPC techniques. PMMA is a material which has wide applications in many areas such as medical, industrial and analytical field of Chemistry.

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