PROJECT REPORT

On

"SYNTHESIS AND CHARACTERISATION OF POLYSTYRENE FILM USING BULK POLYMERISATION METHOD"

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



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B.Sc. CHEMISTRY PROJECT REPORT

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Year of Work: 2018-2019This is to certify that the project "SYNTHESIS ANDCHARACTERISATION OF POLYSTYRENE FILM USING BULKPOLYMERIZATION METHOD" is the work done by PUSHPA C,REESHAL ROSE XAVIER, ARDHRA GEORGE, SREE LAKSHMIT.P.Dr. Ushamani MDr. Annu RajuHead of the DepartmentStaff-member in charge

DECLARATION

We hereby declare that the project work entitled "SYNTHESIS AND CHARACTERISATION OF POLYSTYRENE FILM USING BULK POLYMERIZATION METHOD" 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 Dr. Annu Raju, 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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Introduction

1.1POLYSTYRENE

Polystyrene is a synthetic aromatic hydrocarbon polymer made from the monomer styrene. Polystyrene can be solid or foamed. General purpose polystyrene is clear, hard and rather brittle. It is an inexpensive resin per unit weight. It is a rather poor barrier to oxygen and water vapour and has a relatively low melting point. Polystyrene is one of the most widely used plastics, the scale of its production being several million tons per year polystyrene can be naturally transparent, but can be colored with colorants [1].Uses include protective packing (such as packing pea nuts and CD and DVD cases), containers (such as clamshells), lids, bottles, trays, tumblers, disposable cutlery and in the making of models. The other name of polystyrene is thermocol.

1.1.1CHARACTERISTICS OF POLYSTYRENE

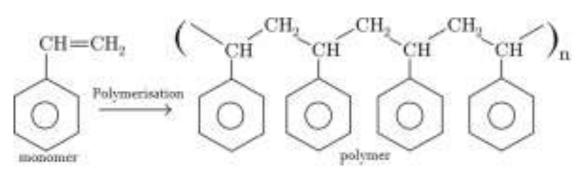
As a thermoplastic polymer, polystyrene is a solid state at room temperature but flows if heated above about 100 degree Celsius, its glass transition temperature. It becomes rigid again when cooled. This temperature behavior is exploited for extraction and also for molding and vacuum forming, since it can be cast into molds with fine details. Polystyrene is slow to biodegrade and is therefore a focus of controversy among environmentalists .It is increasingly abundant as a form of litter in the outdoor environment, particularly along shores and waterways, especially in its foam form, and also in increasing quantities in the pacific oceans [1].

In chemical terms, polystyrene is a long chain hydrocarbon wherein alternating carbon centers are attached to phenyl groups (a derivative of benzene). Polystyrene's chemical formula is (C8H8)n; it contains the chemical elements carbon and hydrogen. The material's properties are determined by short range Vander Waals attractions between polymers chains. Since the molecules consist of thousands of atoms, the cumulative attractive force between the molecules is large [2].

1.1.2POLYMERISATION

Polystyrene results when styrene monomers interconnect. In the polymerization, the carbon- carbon sigma bond is formed, attaching to the carbon of another styrene monomer to the chain. The newly formed sigma bond is stronger than pi bond that was broken, thus it is difficult to depolymerized polystyrene, giving a molecular weight of 100,000 - 400,000[2].

Introduction



1.2 TYPES OF POLYSTYRENE

1.2.1 Isotactic polystyrene

Each carbon of the backbone has tetrahedral geometry and those carbons that have a phenyl group attached are steroid genic. If the backbone were to be laid as a flat elongated zigzag chain each phenyl group would be titled forward or backward compared to the plane of the chain [2] .The relative steriochemical relationship of consecutive phenyl groups determines the tacticity , which has an effect on various physical properties of the materials . The diastereomers were the entire phenyl group on the same side is called isotactic polystyrene, which is not produced commercially synthesized.

1.2.2 Atactic polystyrene

The only commercially important form of polystyrene is atactic, in which the phenyl groups are randomly distributed on both sides of the polymer chain. This randomly positioning prevents the chains from aligning with sufficient regularity to achieve any crystallinity. The plastic has a glass transition temperature TG of approximate 90^oC.Polymerization is initiated with free radicals [2].

1.2.3Syndiotactic polystyrene

Ziegler–Natta polymerization can produce ordered syndiotactic polystyrene with the phenyl groups positioned on alternating sides of the hydrocarbon backbone. This form is highly crystalline with a Tm of 270 ^oC (518F) syndiotactic polystyrene resin is currently produced under the trade name XAREC by idemitsu corporation, who use a metallocene catalyst for the polymerization reaction [2].

1.3 DIFFERENT TYPES OF 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. The initiator should dissolve in the monomer [3]. The reaction is initiated by heating or exposing to radiation. As the reaction is exothermic and wide range of molecular masses are produced.

Bulk polymerization is carried out in the absence of any solvent or dispersant and is thus the simplest in terms of formulation. It is used for the most step growth polymer. In the case of chain growth reactions, which are generally exothermic, the heat evolved may cause the reaction to become too vigorous and difficult to control unless efficient cooling is used [3].

Advantages

- The system is simple and require thermal insulation
- The polymer obtained is pure
- 4

- Large casting may be prepared directly
- Molecular weight distribution can be easily changed
- The product obtained has optical activity

Disadvantages

- Heat transfer and mixing become difficult as the viscosity of reaction mass increases
- The problem of heat transfer is compounded by the highly exothermic of free radical addition polymerization
- The polymerization is obtained with a broad molecular weight distribution due to the high viscosity and lack of good heat transfer
- Very low molecular weights obtained
- Gel effect

For reducing the disadvantages of bulk polymerization, the process can be carried out in a solution. This is known as solution polymerization.

1.3.2 Emulsion polymerization

It is a type of radical polymerization that usually starts with an emulsion incorporating water, monomer, and sufficient. The most common types of emulsion polymerization is an oil in water emulsion, in which droplets of monomer (the oil) are emulsified (with surfactants) in a continuous phase of water. Water soluble polymers such as certain poly vinyl alcohols or hydroxyethyl celluloses can also be used to act as emulsifier / stabilizers. The name "emulsion polymerization "is a misnomer that arises from a historical misconception [1]. Emulsion polymerization is used to manufacture several commercially important polymers. Many of these polymers are used as solid materials and must be isolated from the aqueous dispersion after polymerization. In other cases the dispersion itself is the end product. A dispersion resulting from emulsion polymerization is often called latex or an emulsion. The emulsions find applications in adhesives, paints, paper coating and textile coating. They are often preferred over solvent based products in these applications due to the absence of VOCs (volatile organic compounds) in them [1].

Advantages

- High molecular weight polymers can be made at fast polymerization rates. By contrast, in bulk and solution free radical polymerization there is a tradeoff between molecular weight and polymerization rate.
- The continuous water phase is an excellent conductor of heat, enabling fast polymerization rates without loss of temperature control [4].

The final product can be used as is and does not generally need to be altered or proceed.

Disadvantages

- Surfactants and other polymerization advantage in the polymer or are difficult to remove.
- Cannot be used for condensation, ionic or Ziegler Natta polymerization although some exceptions are known.
- For dry polymers water removal is an energy intensive purpose.

1.3.3 Suspension polymerization

It is a heterogeneous radical polymerization process that uses mechanical agitation to mix a monomers or mixture of monomers in a liquid phase, such as water, while the monomers polymerizes, forming spheres of polymer. This process is used in the production of many commercial resins, including polyvinyl chloride (PVC) a widely used plastic, styrene resins including polystyrene, expanded polystyrene and high impact polystyrene [5].

1.4 APPLICATION OF POLYSTYRENE

Polystyrene is seriously versatile and can be fully recyclable. So it has wide range of applications in various fields.

Air is one of the most natural lying abundant elements on the planet, so the fact that Expanded Polystyrene (EPS) is 98 percentage air illustrates just how resource efficient it is actually. To this Day EPS remains the lightest packaging material around, although its application far exceeds just Packaging as it is utilized for temperature control, weight saving and impact protection [2].

Expanded Polystyrene has an array of exceptional properties.

- Durability
- Versatility
- Shock absorption
- Insulation

The application of polystyrene are follows

1.4.1 Automotive parts

Automotive components made up of polystyrene offers outstanding vehicle light weighting Potential allowing manufacturers to take out significant weight kilo by kilo, all the automotive parts are manufactured in line with TS16949.

HVAC-RCOMPONENTS

(HVAC – R =Heating, Ventilation, Air-conditioning and Refrigerator)

Utilizing the exceptional thermal properties of polystyrene house essential heating and Ventilation systems can be used .These can reduce heat loss and improve the durability and efficiency of heat exchangers, water heaters, pumps, filters motors .It can be used in

- A) Thermal covers of heating system components
- b) Air conditioning system components
- c) Heat exchanger casings
- D) Fridge component housing
- e) Thermal ducting solution for ventilation system
- f) Heat interface units

1.4.2 in construction sectors

Polystyrene offers the perfect combination of mechanical and physical properties which may it ideally suited for the construction industry .Polystyrene provides developers and architects with

Excellent thermal performance and compressive strength .It also offers longevity and sustainable Green credentials. It can be used in

1) Structural insulated panels

- 2) Insulated concrete forms
- 3) Exterior insulation and finish system
- 4) Insulating door inserts
- 5) Road construction
- 6) Swimming pool in fill
- 7) Building facades
- 8) Sound deadening

1.4.3 Pharmaceutical and medicines

Offering tight tolerances, temperature controlled capabilities as well as shock absorption to enable safe damage free transits; polystyrene are affordable and have sterile option for use.

Throughout the pharmaceutical and medicinal industry. In medical field it is used in

- Components for medical devices
- Medical supports
- Human organ transportation
- Packaging for essential medicines
- Chemical bottle packs

1.4.4 Packaging of consumer products

Plastics packaging strategists are focused on designing new packaging tailored to meet Sustainable supply

chain needs .It is reusable and recyclable by using polystyrene it is possible to Make packaging materials for

- Television packaging
- Cooker packaging
- Boiler packaging
- White goods appliances packaging 1.4.5 <u>Food</u>

. Polystyrene has an excellent thermal performance. Catering companies, fish processors and butchers take advantage of Polystyrene to keep their products fresh and spoil during storage and transit. Durability and Softening properties help to protect more threatened goods ^[4]

It is used as:

- Temperature controlled packing
- Ice cream transport
- Cool boxes
- Thermal box to deliver pet food
- Insulated shipping containers and boxes
- Fish boxes
- Egg boxes
- Cake rings
- Synthetic corks

1.4.6 <u>Horti culture</u>

Germinating seedling in multi cell seed trays is the use of polystyrene in horticulture. Polystyrene insulates the seed and encourage their growth.

- Seed trays
- Plant pots
- Raised beds

1.4.7 Seating

It is used for seating applications and desegregated with other components and materials. Polystyrene help to reduce weight, increase strength improve the comfort. Other materials can also provide additional strength where required [4].

- Automotive seating
- Lightweight aircraft seating
- Designer seating
- Wheel chair seating components
- Aircraft seat tray in fills

Application in other fields

- Water sports
- Helmet liners
- Model planes
- Display and exhibition models
- Sculpting Marquette
- Remote controlled vehicles
- Large 3D lettering

- Road construction
- Swimming pool infill
- Hot tub covers
- Insulated and protective cases with living hinges
- Mug packs
- Beanbag bead filling
- Buoyancy aids

1.5 HARMFUL EFFECTS OF POLYSTYRENE

Polystyrene is an expensive and hard plastic that can be seeing everywhere around us. It has a wide application in day to day life. In the same way it also causes a lot of environmental side effects.

- Polystyrene takes at least 500 years to decompose when polystyrene is sent to the landfill, this process take away the water and oxygen which would normally help it breakdown.
- Most of the disposable packing material made of polystyrene takes around 500 years to decompose
 ^[5].

1.5.1 <u>in food</u>

Polystyrene reacts with hot food or drink and make it toxic Polystyrene contains most toxic substances styrene and benzene which is carcinogens and neurotoxins that are hazardous to humans. The hot food and liquid cause the partial breakdown of polystyrene, which causes some toxins to be absorbed to our blood stream and tissue. Polystyrene food containers can soluble the toxin styrene, when they came into contact with hot food or drink, oils, acidic food which causes human contamination and set health risk to humans. Avoid drinking tea or coffee, alcoholic beverages fruit juice and wine in polystyrene cup. Red wine will immediately dissolve the styrene monomer. The package foods that contain vitamin A with the addition of heat, vitamin decompose and produce m-xylene, toluene and 2, 6dimethyl naphthalene. Toluene will readily dissolve polystyrene .This cause polystyrene as an unstable material for packing [3].

1.5.2 Marine pollution

Polystyrene foam has unique issues because of its light weight nature, floatability. The lightweight and floating polystyrene can travel easily through gutters and eventually reach the ocean .The large source of marine pollution across the world is due to plastic from urban run-off. Pollution of water negatively affects tourism and quality of life. When polystyrene travels down water ways and reaches the ocean, it breakdown into smaller non-biodegradable pieces that are ingested by wild life and marine life that harm or kill them. In one Californian study reveals that at least 162 marine species including most sea birds eaten plastics [4].

As a result of the impacts on marine pollution and adverse Effects to marine wildlife, several coastal cities

across the United States, have banned the use of polystyrene food Packaging altogether.

1.5.3 Health effects

The fact that styrene can adversely effects human in a number of ways raises serious public health and safety questions regarding its build up in human issues. Although there is evidence that styrene causes cancer in animals, it has not yet been proven to cause cancer in humans. Styrene primarily exhibits it's toxicity to humans as a neurotoxin by attacking the central and peripheral nervous system. The accumulation of these highly lipid soluble material in the lipid rich tissues of the brain, spinal cord, and peripheral nerves is co-related with acute or chronic functional impairment of the nervous system [4].

1.5.4 Public contamination

Polystyrene products are so common, many people assume they are safe, and the government agencies such as the food and drug administration would not allow a health threatening product to be marketed to the public. But styrene is used to make polystyrene foam packages, but migration of Styrene is nothing new .It was first documented in 1972 and then again 1976 .A survey in1988 published by the foundation for advancement in science and education also found styrene in human fatty tissue with frequency of 100% at level from 8 to 350 ng/g .The 350ng/g level is one third of level known to cause neurotoxic symptoms [5].

Materials and Methods

2.1 BULK POLYMERISATION OF POLYSTYRENE

PROCEDURE

2.1.1 MATERIALS REQUIRED

• NaOH - 2g

2g NaoH is accurately weighed and dissolved in distilled water taken in a 100 ml standard flask

- Styrene
- Benzoyl peroxide
- Separating funnel

All the chemicals are purchased from Sigma Aldrich U S Chemicals.

2.2 PREPARATION

2ml of styrene is taken in a separating funnel and it is washed with 1ml of distilled water for 2 times. Allow it to stand still in a tripod stand. The water is removed by opening the pipe of stop cock. After that it is washed with 1ml 2 percentages NaoH for 3 times .It is also removed by opening the pipe of stopcock .Then the styrene is introduced in to a small beaker. 0.1 g of benzoyl peroxide is added at a temperature of 60 degree Celsius with continuous stirring. After sometime it condenses .It is dissolved in toluene and precipitated in alcohol. It is repeated for 2times .Allow it to dry. And this sample is designated as PS1.

In the same way by changing the amount of benzoyl peroxide we can prepare polystyrene of different percentage .We want to prepare polystyrene by using 0.2g, 0.3g benzoyl peroxide. And these samples are designated as PS2 and PS3 respectively.





Materials and Methods

Results and discussion

3.1 FT-IR spectroscopy

Spectroscopic studies back up the successful polymerization of styrene monomer to polystyrene. The FTIR spectra of polystyrene with 0.1 wt. %, 0.2 wt. % and 0.3 wt. % initiator (BPO) concentrations were given below. All the three Polystyrene materials showed intense characteristic peaks at 2850 cm⁻¹ and 1450 cm⁻¹due to the symmetric stretching vibration for – CH₂ bands and bending vibration for –CH₂ bands, respectively [Fig 3.1, 3.2 and 3.3]. The characteristic peak for C=C symmetric stretching was at 1680 cm⁻¹ and the absence of this peak in the spectra of PS1, PS2 and PS3 indicates the complete polymerization of styrene monomer to polystyrene [7].

This confirms the formation of polystyrene chains.

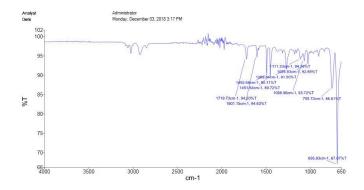


Fig 3.1: FTIR- spectra of PS1

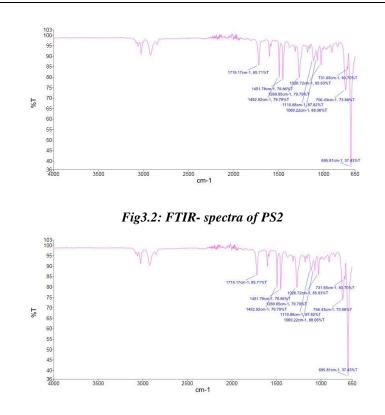


Fig 3.3: FTIR- spectra of PS3

3.2 Thermo Gravimetric Analysis

TGA has been used to study the thermal decomposition behaviour of Polystyrene, and were plotted in the figures below [Fig3.4, 3.5 & 3.6].

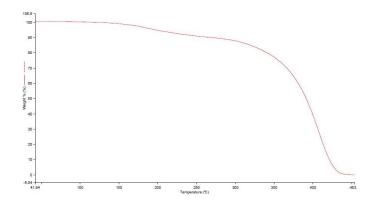


Fig 3.4: TGA curve of of PS1

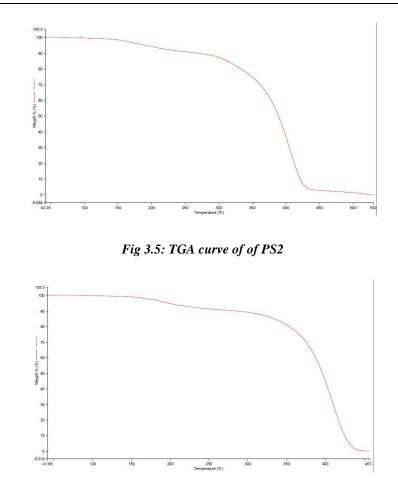


Fig 3.6: TGA curve of of PS3

Above figure shows typical thermogram of mass loss as a function of temperature for PS materials. From the TGA curve it's clear that the organic constituents start to decompose at around ~200°C and ending at ~450°C, which might correspond to the various stage of structural decomposition of the polystyrene chains [9]. The relative thermal stability of the samples is compared in Table 3.1 and the onset degradation temperature (T_d - temperature at 10% mass loss) of PS1, PS2 and PS3 are given. By the increasing the weight percentage of initiator (BPO) in polystyrene synthesis, it is evident that the onset

thermal decomposition were shifted significantly towards the lower temperature range than that of PS1 (340°C) and was minimum for PS3(302.4°C). Around 460°C for all the polystyrene materials the curves became flat, indicating the complete decomposition of polystyrene chains.

Sample	Thermal Properties		
	$T_d^{\ a}(10\%)$	T _d ^a (50%) (°C)	T ^b (°C)
	(° C)	(°C)	(°C)
PS1	340	402	187
PS2	313	395	183
PS3	302	391	165

Table 3.1 Thermal property measurement of Polystyrene

3.3 Differential Scanning Calorimetry

Thermal phase transition changes (e.g., glass transition temperature, T_g) of neat PS materials were studied by recording DSC at a heating rate of 10°C/min in a nitrogen atmosphere. Figure 3.7, 3.8 and 3.9 shows the DSC scan of the samples, in which an endothermic shift in the baseline was observed[12]. The T_g of the PS materials showed a decrease as the initiator concentration increase (Table 3.1). PS1 has glass transition temperature of 187°C, for PS2-183°C and PS3-165°C. These results clearly indicates that, as the initiator concentration increases to 3 wt.% molecular weight of the PS chain segments deceases as a result it tends to retard the segmental motion of the PS matrix and results in a T_g decrease.

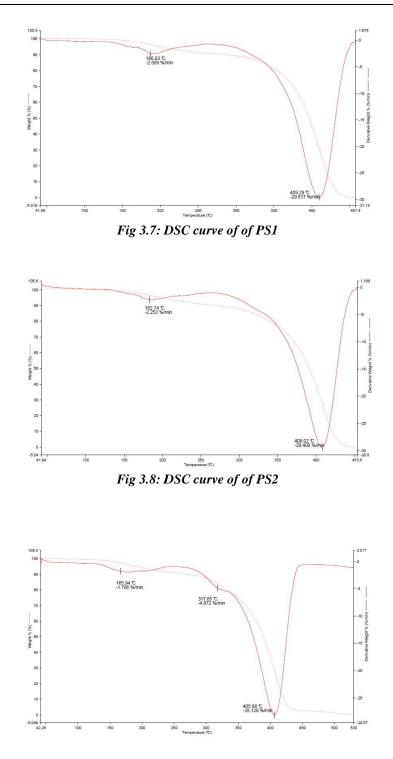


Fig 3.9: DTG curve of of PS3

3.4 Gas Permeation Chromatography

Molecular weights of the various polystyrene samples are obtained by GPC analyses. The molecular weights of the THF soluble bulk PS, were shown in Table 3.2. The number average molecular weights of PS3 are found to be lower significantly than PS2 and PS1, implying the structural restriction of long chain polymer due to increased formation of reactive molecule. i.e. The rate of polymerization reaction will be high as we increase the initiator loading from 0.1 to 0.3 wt.%, which results in decrease M_n and also results in increase in polydispersity[16].

 Table 3.2 Molecular weights of bulk PS

Sample	M _w	M _n	Polydispersity
PS1	3600	3122	1.30
PS2	3592	2865	1.25
PS3	2391	2144	1.12

^a As determined from GPC measurements.

Results and Discussion

Results and Discussion

Conclusions

The polystyrene film is prepared by bulk polymerization method using different initiator concentration. The weight percentage of benzoyl peroxide is varied from 0.1 wt. Percentage, 0.2 wt percentage to 0.3 wt percentage . Its effects are studied by using FT-IR spectroscopy , TGA , DSC and GPC analysis. FT-IR spectroscopy confirmed the polymerization of styrene monomer to polystyrene. The characteristic peak of carbon –carbon double bond stretching vibration is absent in FT-IR of all the three PS1, PS2 and PS3 films. The onset degradation temperature of all the three polystyrene is studied by using TGA. And it is found that PS1 has higher decomposition temperature than PS2 and than PS3. The glass transition temperature is studied by using DSC. The glass transition temperature increases in the order PS3<PS2<PS1.

From GPC is it confirmed that all the three polystyrene films have polydispersity index greater than one. And the number average weight of film increases in the order of PS3<PS2<PS1. This results indicate that PS1 with 0.1 percentage initiator have more number of polymer chains with high molecular mass due to less number of reactive species in propagation step.

Conclusions

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