

# **PREPARATION OF WAX FROM WASTE PLASTIC**

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Submitted by



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**National Institute of Technology**

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**CERTIFICATE**

This is to certify that the report on **“Preparation of Wax from Waste Plastic”** submitted by Arvind Kumar in partial fulfillment of the requirement for the degree of master in technology in the department of chemical engineering at National Institute of technology, Rourkela is an authentic work accomplished by him under my guidance and supervision.

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## ABBREVIATIONS

PP	Polypropylene
TGA	Thermo gravimetric Analysis
DSC	Differential Scanning Colorimetric
FTIR	Fourier Transform Infrared spectroscopy
SEM	Scanning Electron Microscopy
GCMS	Gas Chromatography Mass Spectrometry
NMR	Nuclear Magnetic Resonance
PD	Penetration Degree
CHNS	Carbon Hydrogen Nitrogen Sulphur
GCV	Gross Calorific Value

## ABSTRACT

The aim of the present work address the issue of recycling of plastic by thermal pyrolysis. In this study PP waste has been converted to PP wax. The process was carried out in a semi-batch reactor with various temperature range of 350-450 °C and the different time range of 50-120 min. The waxes from pyrolysis of PP was obtained and the effect of temperature and time on product yield has been studied. The maximum yield of 59% was obtained at optimum conditions of 450 °C and 75 min. Further, the obtained product at optimum conditions has been carried out for its physical and chemical analysis. From the physical analysis studies it observed that the penetration degree of wax ranging from 0.0 – 13.3 mm, and the melting point of PP wax is observed at an optimum condition is 122 °C. As a consequence of the chemical analysis like FTIR of PP wax shows that most of the functional group are aliphatic in nature. Which was also confirmed through GCMS analysis, especially the highest of compounds are aliphatic hydrocarbons and very few of them are carbonyl and aromatics in nature. The utmost 74% of Cyclopentane, 2-propenyl is observed in PP wax. However, from <sup>1</sup>HNMR studies it was proved that the obtained compounds were highly branched hydrocarbons. From the above result it can be proved that the wax obtained from PP pyrolysis can be used for industry and domestic purpose.

**Keywords** – PP, PP wax, DSC, Penetration Degree, FTIR, NMR, and GC – MS.

# CHAPTER 1

## 1. INTRODUCTION

Plastics play a vital role in today's world because of their durability, light weight, strength, chemical resistance, design flexibility and low production cost. The main constituents of plastics are organic (Major elements: carbon, hydrogen, nitrogen) and inorganic (Major elements: Chlorine, Fluorine, Sulphur) molecules. Plastics are used the whole range of manufacturing and local areas. It manufactured on a huge scale worldwide and its production crosses the 150 million tons per year worldwide. In India, almost 8 Million tons plastic products are consumed every year (2008) which is expected to rise 12 million tons by 2012 [1]. The estimation is available in the total generation of plastic waste in the country, however, considering 70% of total plastic consumption is rejected as waste. Thus, approximately 5.6 million tons per annum (TPA) of plastic waste is generated in the country, which is about 15342 tons per day [1].The table shows the Per Capita Consumption of Plastics in Some Countries in the World [2].

Table 1. Per capita Consumption of Plastics in Some Countries in the World.

Sl. No.	Country	1980	2005	2015
1	Japan	50	80	109
2	Nafta	46	105	139
3	Western Europe	40	99	139
4	India	4	7	13
5	Central Europe	9	24	48
6	Latin America	7	21	32
7	Asia(Excluding Japan)	2	20	36
8	Middle East Africa	3	10	16
9	World Total	11	30	45

And only approximately 60% of waste was possible recycled, rest of balanced 40% was not possible to reuse. So gradually it goes on gathering, thereby leading to various serious disposal and environmental problems. Most of the Plastics are derived from petrochemical industries.. Therefore, have naturally high calorific value. The calorific values of some of the different plastic materials along with coal and some of the petroleum products are represented in Table 2 [3].

Table 2. Calorific value of some plastics materials

Material	Kilojoule per kilogram	Btu per Pound
Light fuel oil	44000	18920
Medium fuel oil	43000	18490
Petrol	44,800-46,900	19264-20167
Diesel	46000	19780
Gas oil	46000	19780
Plastics		
Polypropylene	45000	19300
Polyethylene	46500	20000
PVC	19000	8170
PET	21600	9290
Polystyrene	41600	17900

As plastics are non-biodegradable, so the plastic waste creates a lot of both direct and indirect effects on the environment and human welfare [4]. The increase of plastic waste and its harm has concerned the worry of the political and technological circles. Some of the methods adopted for waste management. There are only landfilling, and incineration methods but these are not suitable because of environment problems such as air pollution and soil contamination. In a long term both of the technique are not safe and suitable as the incineration of waste plastic produces harmful or greenhouse gasses e.g.  $SO_x$ ,  $NO_x$ ,  $CO_x$ , etc. Recycling has become an important issue. Recycling can be classified as energy recycling, chemical recycling, and martial recycling [5]. In chemical recycling pyrolysis is a form of thermal behavior where waste materials are heated at high temperatures with the absence of air under high pressure to transform it into solid, liquid and gas products. The solid by-product of PP can be converted into useful domestic products such as wax [6]. PP is a macromolecular hydrocarbon that can be converted into useful products such as oil and waxes but since the molecular chain of PP is composed of  $-CH_2-$ ,  $-CH_3-$ , the freezing point of the fuel oil obtained is very high and the research octane number of gasoline is very low near 88. almost all of polyolefin pyrolysis has involved the conversion of polyolefin to gaseous and/or liquid products; ultra-pyrolysis

(>400°C) is used to maximize the productivity because it allows for reducing time of the products [7]. Thus, it is not a feasible way to convert some of the waste plastics to oil. It is, however, a suitable way to convert some of the plastics waste to wax [6]. The "wax" is generally defined as a diverse class of organic substances that are hydrophobic, malleable solids near ambient temperature but liquids at slightly higher temperatures. The main chemical composition of waxes is complex, but normal alkanes are always present in high proportion and contain a higher molecular weight range. Mostly the waxes are obtained by crude oil. But there are some other sources to produce such as Plants, animals and waste solids [8]. Paraffinic wax and a microcrystalline wax derived from petroleum products. Waxes have some uses in the manufacture of candles, packaging, paper coating, wood polishing, water and chemical resistance and decorative purposes, in polishes, electrical insulators, paper coatings, printing inks, textile finishes, and leather dressings, etc. [9].

## **OBJECTIVE OF THE PROJECT**

- Preparation of wax from waste plastic (PP) by thermal pyrolysis and optimization of the process parameter.
- Study of the physical and chemical characterization of obtained wax and comparison with commercial wax

# CHAPTER 2

## 2. LITERATURE REVIEW

### 2.1. Plastics

Plastics are materials whose basic constituent is manufactured synthetically or semi-synthetically monomer. Plastic consists largely organic molecules (such as carbon-oxygen, nitrogen) and inorganic molecules (chlorine, fluorine, and Sulphur). It formed by polymerization. It may be reshaped under pressure and temperature.

A process in which the small units of similar and different molecules combined to form a high molecular and long branched molecules, called polymerization.

### 2.2. Plastic classification

1. **Thermo Plastics** In such plastics the molecular chains are not cross-linked. So they are softening on heating and then harden again on cooling and remolded over and over again.

Ex- polycarbonate, polyethylene, PET, PVC, PP, PS.

2. **Thermoset Plastics** These plastics have closely meshed cross-linked molecular chains. Due to this type of shape they can no longer be shaped after Harding. They also cannot be melted.

Ex- epoxide, phenol-formaldehyde, polyurethane.

3. **Elastomers Plastics** –These are crosslinking of the knotted molecular chains. Highly stable but still elastically molecules. By applying a load, they become disentangled but again they can get shape after removal of the load.

Ex – rubber

### 2.3. Production of Plastic

The increasing trend in plastic production since 1950 is shown in the underneath table [11]. The production of plastic is continuously increasing.

Table 3. Worldwide plastics production

Sl.no	year	Production in metric tons
1	1950	1.5
2	1976	50
3	1989	100
4	2002	200
5	2008	245
6	2009	250
7	2010	270
8	2011	279
9	2012	288
10	2013	299

### 2.4. Types of waste plastic and their recyclables

The main category of plastics include [8]:

A. Reusable Plastics (Thermoplastics): PET, HDPE, LDPE, PP, PVC, PS, etc.

B. Non-Recyclable Plastics (Thermoset & others): Multilayer & Laminated Plastics,

As per BIS Standard Arrangement, as notified in Rule 8 (b) of the Plastic Waste (Management and Handling) (Amendment) Rules, 2011, they classified plastics in different seven categories [10].

Table 4. Types of waste plastic and their recyclables

Type	Mark	Recyclable	Abbreviation	Description
Type 1		Yes	PET	Polyethylene Terephthalate Beverages.
Type 2		Yes	HPDE	High-Density Polyethylene Oil packets.
Type 3		Yes, but not common	PVC	PVC food packets wrapping, oil bottles
Type 4		Yes	LDPE	Low-Density Polyethylene, Many plastic bags, shrink wraps,
Type 5		Yes	PP	Poly Propylene, Refrigerated containers, some bags, most bottle tops, some carpets.
Type 6		Yes, but not common	PS	Polystyrenes. Through away utensils, meatpacking.
Type 7		Yes	PC,ABS,PBT	Usually layered or mixed plastic.

## 2.5. Advantages and Disadvantages of plastics

### Advantages of Plastics

1. Plastics are light in heaviness.
2. They can be easily reshaped and have excellent finishing.
3. They possess very good strength & toughness, good shock absorption capacity.
4. Corrosion is resistant & chemically inert.
5. Low coefficient of thermal expansion and possess good thermal & electrical insulating property.
6. Used to make ladder bottles, pens, plastic bags, cups, etc.
7. Plastics are a strength, durable in nature, and very cheap to produce.
8. Used to reduce soil and wind erosion [11].

## **Disadvantages of Plastics**

1. It is nonrenewable resources.
2. It causes cancer.
3. Embrittlement at low temperature and deformation under load
4. Low heat resistant & poor ductility.
5. Plastics are combustibility.
6. They produced toxic fumes when it burnt.
7. It is a recycling process but very costly.

### **2.6. Types of Waste Plastics and Generation of Waste**

It is the useless and unwanted solid material generated from combined residential, industrial & commercial activities in a given area.

Waste plastics mainly classified as industrial waste plastic and municipal waste plastics based on their origin. They have different quality and properties.

#### **2.6.1. Industrial Waste Plastics**

Industrial plastic squanders are those mainly produced from the plastics assembling and handling industry. Normally, they are homogeneous or heterogeneous plastic pitches, moderately free of contamination and accessible in genuinely expansive amounts. For mechanical plastic squanders, reshaping and remolding and reusing appear to be simple. However, when plastic squanders are different or comprise of blended saps, they are wrong for recovery. For this situation, pyrolysis is a superior method to change over it into helpful items [10].

#### **2.6.2. Municipal Waste Plastics**

Municipal plastic squanders for the most part gathered as family unit squanders and a part of city strong squanders as they are rejected and. Civil Waste Plastics usually covers around 10% of the aggregate squanders by weight and by volume. For the reusing of metropolitan plastic squanders, detachment is required from other family unit wastes. While the partition procedures have been concentrated broadly, it is still unrealistic to group MSW mechanically and get attractive portions. So squander division at the family is required concerning MSW [12].

Table 5. Generation of plastic waste in top five cities (Tones/day)

Sl.no	Name of city	1992-2000	2004-2005	2010-2012
1	Mumbai	5355	5320	6500
2	Bangalore	2000	1669	3700
3	Kolkata	3692	2653	3670
4	Chennai	3124	3036	4500
5	Hyderabad	1556	2187	4200

Table 6. Top five states in India (Tones/day)

Sl no.	City	1999-2000	2009-12
1	Maharashtra	18198	19204
2	Tamil Nadu	10806	12504
3	Uttar Pradesh	11920	11585
4	West Bengal	9242	12557
5	Kerala	2596	8338

As the data above data, the rate of generation of waste plastics is higher in Mumbai .the rate of generation is decreased in the year 2004-2005. The average rate of generation is approx. 11.8 %. Maharashtra is the highest waste plastics generating a state in India. But in the West Bengal the rate of a generation very rapid.

## 2.7. Disadvantages of plastic waste

Indiscriminate littering and unorganized recycling/reprocessing and non-biodegradability of plastic waste raises the several environment issues [13].

- The release of harmful gasses such as CO<sub>x</sub> , SO<sub>x</sub>, etc. during product manufacturing.
- The landfill is a major disadvantage.
- Land get to be barren because of indiscriminating dumping of waste plastics
- The collection, recycling, and reuse are not easy.

- Contaminated ground & surface water.
- Huge production of plastic waste pose unattractive look and block the drain.
- Health impact due to direct contact with plastics like- skin, a blood infection.
- Damage ecosystem.
- Discourage tourism & other business.

#### Utilization of plastic waste

- In road construction.
- Used as a raw material in plastic waste as alternative fuel and raw material (AFR) in cement kilns and power plants.
- Conversion of waste plastic into liquid oil and wax [14].

### 2.8. Waste management strategic

#### 2.8.1. Recycling treatment

Recycling is the process in which we mainly remove the items from the waste stream that can be further reused as a raw material in the producing new products. By definition the recycling takes place in three parts: in which first, the waste is selected and recyclables collection, the recyclables are used to produce new raw materials. These raw materials are then used as a ram material in the production of new products. Plastic recycling consists chemical, mechanical and energy recycling.

Chemical recycling is an important treatment to convert waste plastics into low molecular hydrocarbons or useful chemicals .Further, these products can be used as a ram materials in various industries. It is also known as feedstock recycling or tertiary recycling, aims to convert waste polymers into original monomers or other valuable chemicals.

#### 2.8.2. Landfill treatment

Landfill treatment is an ancient method for waste management. Landfills are considered to reduce greatly or eliminate the risks of waste disposal which may lead to public health hazards as well as environmental degradation. They are usually located in areas where land structures are not more suitable indicating infertile soil which is fairly water logged because of its tightly packing of particles [15]. Furthermore the different arrangement of landfill, other protective actions are associated with its designs. The base and sides of landfills are lined with layers of earth or plastic to keep the fluid waste i.e., leachate, to remain within the dimension of soil. The leachate is collected and pumped to the surface for treatment. Boreholes or monitoring

wells are dug in the vicinity of the landfill to monitor groundwater quality. In addition to the strategic placement of the landfill, other protective measures are incorporated into its design. The bottom and sides of landfills are lined with layers of clay [11].

### 2.8.3. Incineration

Incineration is a prominent thermal treatment process. It is a controlled high-temperature oxidation process. This is the combustion of waste in the presence of oxygen which results in conversion of waste into carbon dioxide, water vapor, and ash. This method may be used as a means of recovering energy to be used in heating or the supply of electricity. In addition to supplying energy incineration technologies have the advantage reducing the volume of the waste, rendering it harmless, reducing transportation costs and reducing the production of the greenhouse gas methane.

### 2.8.4. Pyrolysis

Pyrolysis, thermal decomposition decomposes organic waste by exposing it to high temperatures and allows no oxygen. These techniques use heat and an oxygen deficit environment to convert biomass into other forms. A mixture of combustible and non-combustible gasses as well as pyrolytic liquid is produced by these processes. All of these products have a high heat value and can be utilized. Gasification is advantageous since it allows for the incineration of waste with energy recovery and mitigating air pollution.

## 2.9. Related Work in This Field

Shuyuan et al. (2002) reported the yield of the polyethylene wax is obtained 49-98.94% at the pyrolysis temperature, and it varies with temperature and pyrolysis time. When an increase in the temperature, yield of PE wax decreased. Because of vaporization of wax and loss of the gaseous product and the penetration degree that is the hardness related to the molecular weight range and deepness of PE wax. At certain temperature penetration degree of PE wax increased as pyrolysis time increased. Low temperature does not affect the penetration degree but at high-temperature penetration degree increases with an increase in pyrolysis temperature. The obtained Pyrolysis PE wax has a melting point of 104-114 °C, and penetration degree is 0.1-0.4 mm. The dyes do not affect melting point and penetration degree, they affect PE wax colors only [1].

Umaru et al. (2014) concluded that the waste polyethylene was pyrolyzed at 200 °C & 250 °C for 1 hour, 2 hours, and 3 hours respectively. As the pyrolysis time and temperature increase the yields of pyrolyzed wax decreased. The yields of pyrolyzed wax are higher than at 200 °C. When temperature increases polyethylene structure break down randomly at any position that

possesses decrease in the molecular weight of the polymer. So the viscosity of polyethylene wax also decreases as the time and temperature of pyrolysis increased. The molecular breakdown carries on with an increase in temperature and time of pyrolysis. The melting point of pyrolyzed waxes was obtained within the range of 8-114 °C [2].

Arabiourrutia et al. (2012) examined that the yield of waxes decreases as temperature is increased and yield also depends on the structure of plastics. At low-temperature cracking of branched chain first and then low cracking in the principal chain for the short residence time. The unbranched paraffin waxes have a melting point range in the 50-70 °C, and melting points for branched paraffin waxes are in the range of 60-91 °C. The heating value of the waxes is lower than the feed. When the temperature increases the heating value of the waxes increased [3].

Ademiluyi et al. (2013) investigated that the polyethylene wax obtained by waste water sachets has yielded over 90% at the optimum pyrolysis temperature range 110-150°C, and optimum pyrolysis time 20-30 minutes. The obtained polyethylene wax has a melting point of 46-74 °C. And penetration degree of wax was 1-40.6 mm. The yield of wax decreases when to increase in the pyrolysis temperature and time. At high pyrolysis temperature, the melting point of obtained wax decreased sharply. When polyethylene waste sachets were pyrolyzed at a temperature range from 110°C to 150°C, the penetration degree of Polyethylene wax obtained varies from 1 to 40.6mm. At certain pyrolysis temperatures, with an increase in pyrolysis time, the penetration degree of polyethylene wax obtained increases. At low temperatures, the penetration degree does not change significantly with an increase in temperature [4].

H. R. Zhang et al. (2015) studied the conversion of PP waste to PP wax at various conditions by low-temperature pyrolysis. And found product yield 53.9%. He observed that the melting point range of PP wax was 121–150°C. From Fourier transform infrared analysis of the polypropylene wax, he proved that it is less branched and that its olefinic content is higher than that of commercial wax [7].

Sunder Lal et al. (2006) investigated the depolymerization of HDPE to wax at different temperature and time and obtained product yield 63.5 %. The melting point of the product changes from 120 – 80 °C and The wax formed, has –COOH and –OH functional groups [30].

Barbara J. Musser et al. (1998) characterize the different types of waxes by included elemental analysis, FTIR spectroscopy, field desorption mass spectroscopy, 1H NMR spectroscopy. And

observed H/C ratios between 1.92 and 2.05. The  $^1\text{H}$  NMR indicated the presence of ketone but negligible alcohol or ether. From FTIR measurement, he found the waxes were gauged to contain between 50 and 80% straight methylene chains.

## 2.10. Types and use of waxes

The term wax defines an organic, natural or synthetic plastic-like material, which is solid at room temperature and turns into a liquid when heated; it is also possible to deform a wax without heating it but simply through pressure. [1]

### 2.10.1. Characteristics common to all types of wax

Waxes are solid near at 20 °C, and its consistency lies from soft and plastic to hard and brittle. They have low thermal and electrical conductivity, Combustible, relatively low viscosity, soluble in an organic solvent like chloroform, hexane and insoluble in water. [2]

### 2.10.2. Classification of waxes

Waxes are classified by their origin and properties and showing different chemical properties.

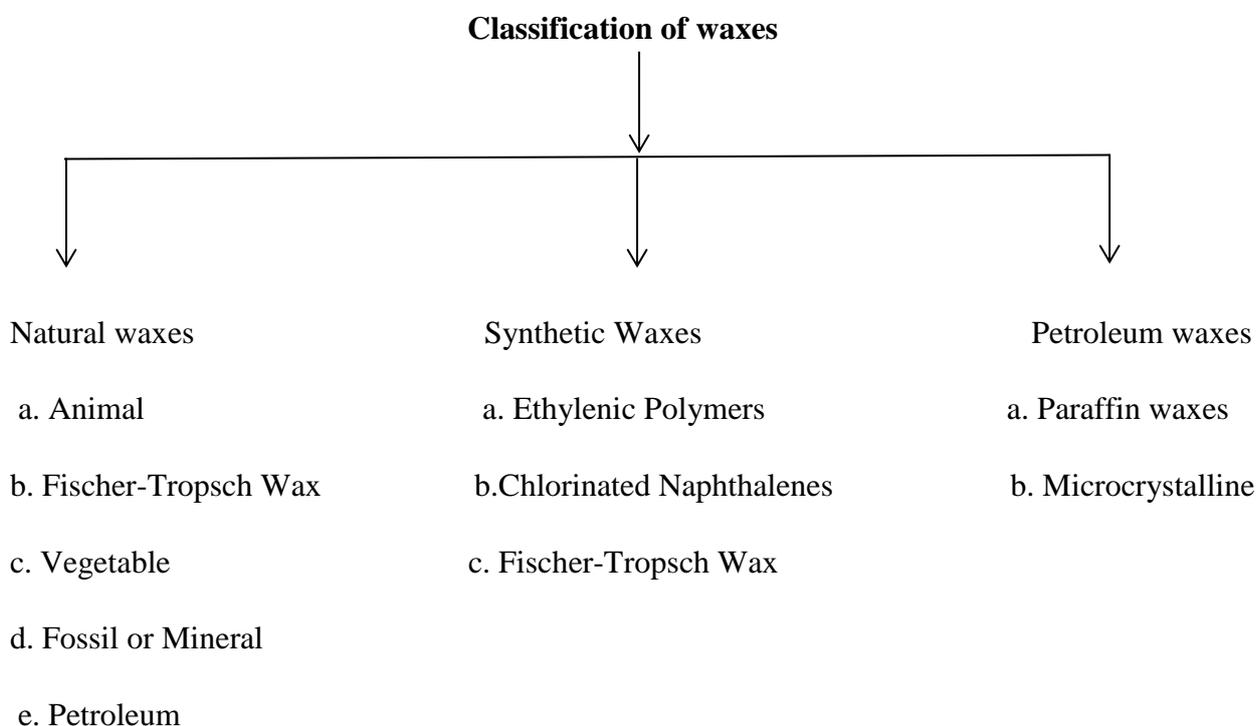


Figure. 1. Classification of waxes

### 2.10.3. Application of waxes

In general, life waxes useful at everywhere in school, office, on the street we are directly or indirectly surrounded from the use of wax. Waxes are used in candles, matches or polishes for various surfaces; it is less clear instead when we refer to pencils, precasts, tyres, pharmaceutical products, cosmetics, electrical cables, packaging, textiles, food packaging, explosives, fireworks, paints, plastics, sinters, chewing gum and so on

# CHAPTER 3

### 3. MATERIALS AND METHODS

#### 3.1. Collecting of Raw materials

The raw materials used in this study is a polypropylene (PP) waste, the disposal PP waste glasses were collected from the premises of NIT Rourkela that were used by students, faculties in different purpose. Before performing experiment the waste raw materials were washed by using tap water then dried at room temperature and then cut into small pieces by the plastic cutting machine.



Figure. 2. Waste plastic materials collected for process



Figure. 3. Chopped waste plastic (PP)

### 3.1.2. Proximate and ultimate analysis of raw material

The compositional analysis of PP Plastics was carried out by proximate and ultimate analysis. Proximate analysis was carried out by the standard procedure described by the ASTM D3172-07a method to identify the moisture, volatile matter, fixed carbon and ash content of the raw material. The percentage of carbon, hydrogen, nitrogen, oxygen and sulfur in the plastic was determined using CHNSO Elemental Analyzer by Variacel CUBE, Germany. The proximate and ultimate analysis of PP sample are shown in Table 4.1.

### 3.1.3. Calorific value

The calorific value of raw materials PP and wax were experimentally calculated through a bomb calorimeter (Model: Parr 6100 EE digital bomb calorimeter). The sample ( $0.6 \pm 0.03$ g) was placed inside the bomb and burned in the presence of oxygen with an increment of  $\pm 0.01$  °C to determine the HHV (higher heating value) as per the ASTM D 4809-95 method.

### 3.1.4. Thermo gravimetric analysis

Thermo gravimetric analysis (TGA) of PP was obtained by using the SHIMADZU model DTG-60/60H. Approximately, (10-12 mg) of the sample was taken in an  $\text{Al}_2\text{O}_3$  crucible and heated up to the final temperature of 500 °C with 10 °C/min heating rate. The inert atmosphere was created by flowing pure nitrogen gas at around 50 mL/min flow rate in replacement of the air present in the pyrolysis zone for avoiding unwanted oxidation of the sample.

### 3.1.5. Differential Scanning Calorimeter

Differential Scanning Calorimeter (DSC) was carried out by using NETZSCH DSC 200F3. Approximately, 12-14 mg of the sample was taken in an Al DSC pan and heated from ambient to final temperature of 200 °C with mention the 5 °C/min heating rate. The inert atmosphere was created by flowing pure nitrogen gas at around 60 mL/min flow rate in replacement of the air present in the pyrolysis zone for avoiding unwanted oxidation of the sample

### 3.1.6. Experimental setup for the production of wax

The reactor used in the present study was made of the stainless steel semi-batch reactor (dimensions: length 16cm, outer diameter 5.2cm, and inner diameter 4.8 cm). The reactor is screwed with two rods and covered with an iron cover. Pyrolysis of waste disposal glasses (PP). 30 gm was filled in the reactor for each experiment and kept in an electrically heated furnace. The reactor system consisted of a PID temperature controller with an adjustable heating rate for the furnace. The pyrolysis reaction was carried out at different temperature and time ranges of

350 °C to 450 °C and 50 min to 200 min. Respectively with a constant heating rate of 10 °Cmin<sup>-1</sup>. The liquid product was transferred into a tray which was getting solid at room temperature and weighed after cooling, and the gas was escaped to the atmosphere. Further, the product yield was calculated from the material balance. Fig 1 shows the schematic diagram of pyrolysis setup.

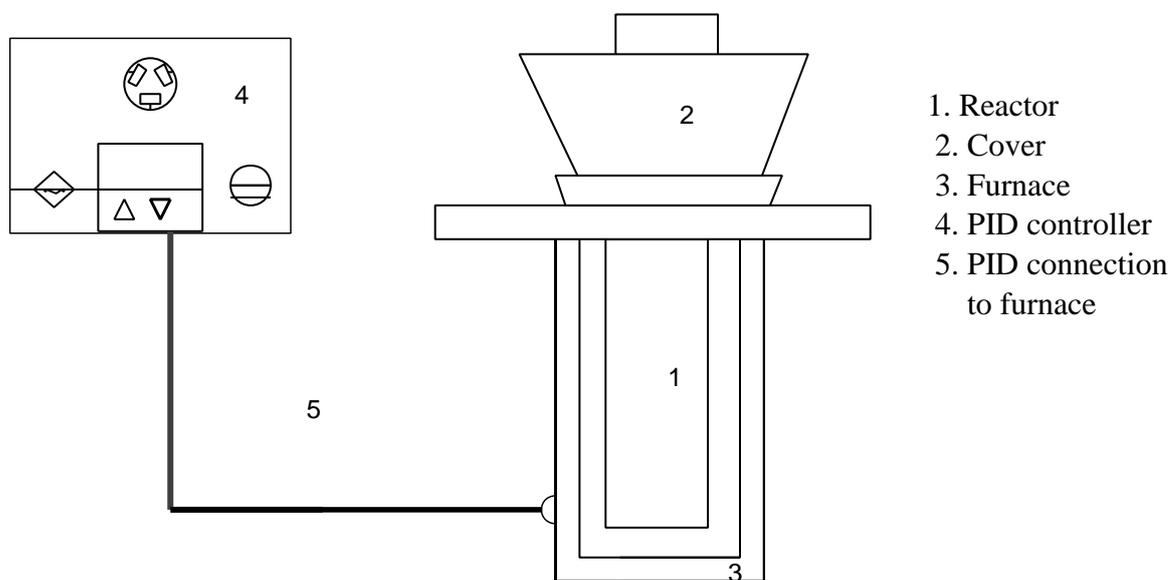


Figure. 4. Schematic diagram of experimental set-up

## 3.2. Characterization of pyrolytic wax

### 3.2.1. Physical characterisation of pp wax

#### 3.2.1.1. Penetration Degree

Penetration degree was analyzed by standard method ASTM D1321. Penetration tests are done on petroleum products to determine consistency and shear stability (lubricating greases) for design, quality control, and identification purposes. A standard needle (did - 3.2mm, 2.5 gm, and hardened stainless steel) is released from a penetrometer (Supplied with 100 gram weight) and allowed to drop freely into the sample for 5 seconds at room temperature. The deepness of penetration of the needle into the sample is measured in tenths of a millimeter by the penetrometer.

#### 3.2.1.2. Melting point

It provides the information on the temperature at which most of a given sample changes from a solid to a liquid. By the melting point, we can determine the quality of wax. It is particularly

applied to petroleum waxes to determine the nature of wax whether it is crystalline or amorphous. To analyze melting point, 14 mg of the sample has taken in an alumina crucible and placed in DSC with a temperature range of 30 °C -200 °C with a constant heating rate of 5 °Cmin<sup>-1</sup>. In nitrogen atmosphere.

#### 3.2.1.3. SEM Analysis

The physical morphology of the pp wax was analyzed using scanning electron microscopy (SEM) with JEOL-JSM-6480LV model at 15 kV accelerating voltage. Before the analysis, the sample was coated with thin layer of platinum to prevent the charging of sample.

#### 3.2.1.4. Oil Content

Oil content in wax was analyzed using Soxhlet apparatus (made in India induswa ) which consists three heaters and voltage supplied 220 vol. 5 gm of sample was taken in a thimble and placed in the condenser. N-hexane used as a solvent and done for 6 recycles. Colour of the solution was changed, and oil was extracted by the rotary extractor.

### 3.3.2. Chemical characterization of wax

#### 3.3.2.1. Fourier Transfer Infrared spectroscopy (FTIR)

The determination of organic functional groups of chemicals present in PP wax was carried out by Perkin-Elmer Fourier transform infrared spectroscopy (FTIR) at 8 cm<sup>-1</sup> resolution in the range of 400–4000 cm<sup>-1</sup> using Najol mull. A small quantity of the wax was mounted on KBr pellet, and the infra-red spectrum scanning of the wax was performed.

#### 3.3.2.2. GC-MS analysis

Gas chromatography and mass spectroscopy (GC–MS) were performed to quantitatively examine the elemental compounds in the wax by using Agilent 7890B Network GC system that was programmed at 70 °C for 3 min and then rises up to 300 °C at 10 °C/min where the total GC run time was 25 min. The DB–5ms column of diameter 0.250 µm and 30 m length was used where .98 gm wax was injected into the column with the carrier gas (helium) at 1.5 mL/min flow rate. Chemical compounds present in the PP wax were ionized at 70 eV ionization energy, 230 °C ion sources temperature, and were analyzed over a mass electron (m/z) range of 40–700. The chromatograms of the chemical compounds at different retention time and respective mass spectra were plotted and compared against the spectral data with the W9N11 MS library.

### 3.3.2.3. $^1\text{H}$ NMR analysis

$^1\text{H}$ -NMR (Proton nuclear magnetic resonance) spectra were recorded by NMR (AV 400 Avance-III 400MHz FT-NMR Spectrometer Bruker Biospin International, Switzerland). The proton NMR shows the proton shift in the structure. PP wax was dissolved in deuterated chloroform to make a homogenous solution for proton

# CHAPTER 4

## 4. RESULTS AND DISCUSSION

### 4.1. Thermal decomposition characteristic of PP

The thermal properties of the raw material were carried out by TGA, which provides the correlation between weight loss with respect temperature in a controlled environment. It also useful to investigate the thermal stability of the material, or to investigate its behavior in different atmospheres (e.g. inert or oxidizing). In this study thermal stability/degradation of PP was used to determine by TGA from ambient temperature to 500 °C with a heating rate of 20 °C/min. Figure # portrayed that the major decomposition of PP was started from 280 °C and about to complete at 430 °C. Hereafter there was no weight loss observed. Thus, 350–450 °C is a suitable pyrolysis zone where the reaction can be taking place. A similar result also observed in previous literature [17].

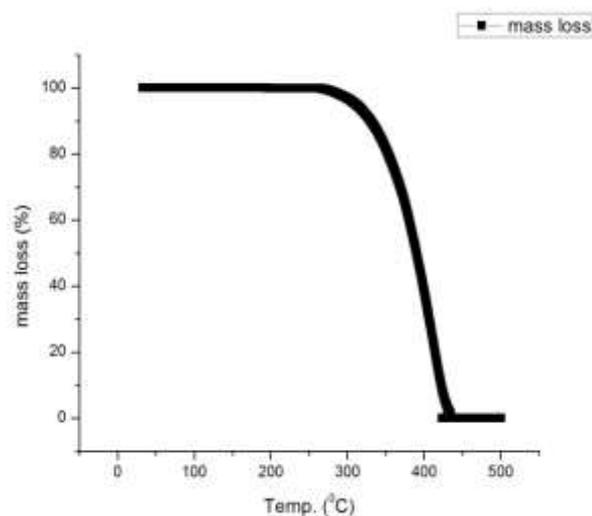


Figure. 5. TGA of waste disposal glasses

### 4.2. Proximate and ultimate analysis of PP

Proximate and ultimate analysis are one of standard composition analysis to characterize a solid fuel. From the table 7, it has shown that the volatile percentage of PP is 100%. Hence, the product can be potentially applicable to the production of energy. Ultimate analysis estimates the carbon, hydrogen, nitrogen, sulfur and oxygen content. The obtained carbon and hydrogen percentage is more than that of nitrogen, sulfur and oxygen. The presence of higher hydrogen and carbon ensure that this can be better used for the production of hydrocarbon. A similar result also observed by other

literature [include references]. The obtained calorific value of wax is 47.64 MJ/Kg which is near about the other conventional fuel. Therefore, this could provide the major impact on energy production.

Table 7. Proximate and ultimate analysis of waste disposal glass

Type of raw material	waste glass (PP)
Proximate Analysis	
Moisture Content	0.00
Volatile matter	100
Ash content	0.00
Fixed carbon	0.00
Ultimate Analysis	
C	82.69
H	13.93
N	0.80
S	0.07
O/Others	2.51
Gross Calorific Value (MJ/Kg)	47.64

#### 4.3. Differential Scanning Calorimeter (DSC)

DSC is used to determine the quantity of energy absorbed or released when the sample being heated or cooled, which also provide the quantitative and qualitative data heat absorption and heat released process. To define the property of plastic and its behavior of plastic mostly DSC is used. Fig 5 shows the melting behavior of PP plastic by using differential scanning calorimeter with respect to temperature and heat flux. From the figure, it was analyzed that the DSC curve of PP plastic shown the melting point at 166.3 °C. Some plastic like HDPE, LDPE has the melting point 204 °C and 105 °C respectively, this difference in melting point for this plastic is the due difference in structure and its molecular weight [18].

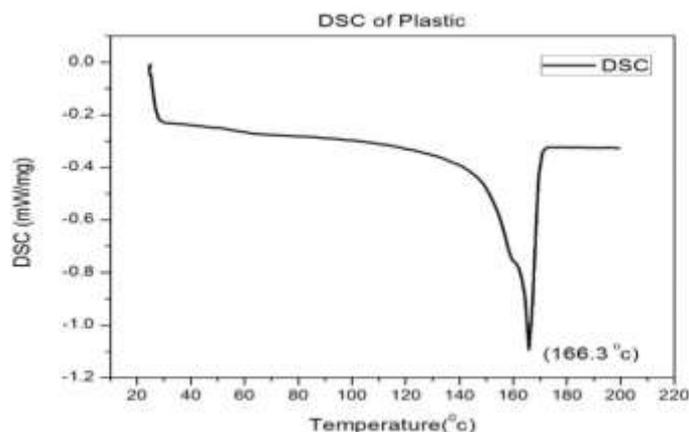


Figure. 6. Thermal analysis of plastic by DSC

#### 4.4. Influence of temperature on product yield and reaction time

Figure 6,7,8 shows the effect of temperature and time on product yield of PP wax, the product yield of PP wax carried out from 350 °C to 450 °C with 50 °C elevation and 50 min to 120 min with 25 min elevation time. From the figures, it was observed that the product yield varies from 20.10–81.94% at 350 °C to 450 °C and 50 min to 120 min respectively. There is no significant change of product yield was observed at low temperature and time. However, at a certain temperature, with an increase in reaction time, the product yield becomes decreases. This is also in agreement with other plastics [9]. At high temperature and high reaction time there is a sharp change of product, yield was observed. This happens due to the vaporization of wax and formation of non-condensable gas [6]. To determine the properties of wax in the plastic deepness of plastic pyrolysis should be measured. At a particular temperature, the reaction time determines the deepness of the pyrolysis an increase of reaction time, the deepness of pyrolysis becomes deeper. However, at low temperature and the short reaction time the obtained product yield becomes behaves like plastic properties. As a consequence, 450 °C at 75 min are the optimum parameters for the production of wax, the maximum wax obtained at this conditions wax 59.2%. From figure 8 it was analyzed that at 450 °C with increase in reaction time the PP waste has been over pyrolyzed, and the quality of the wax cannot meet the requirements of the standard which is also stated by Jixing et al. for PE plastic

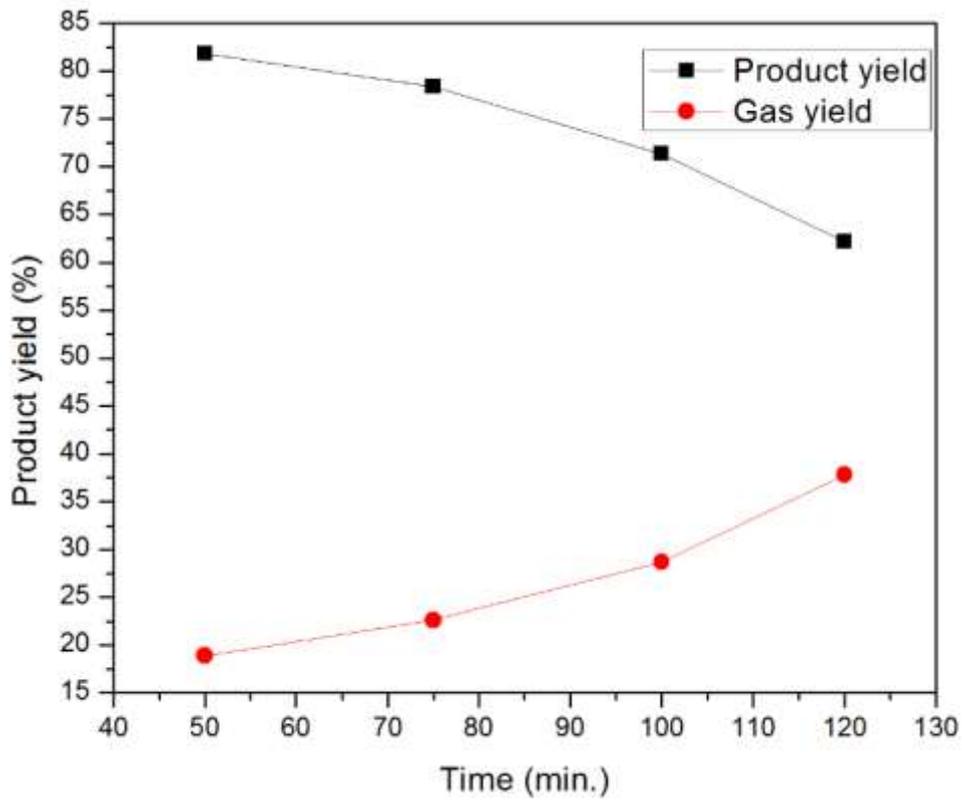


Figure. 7. Product yield at Temperature 350 °C

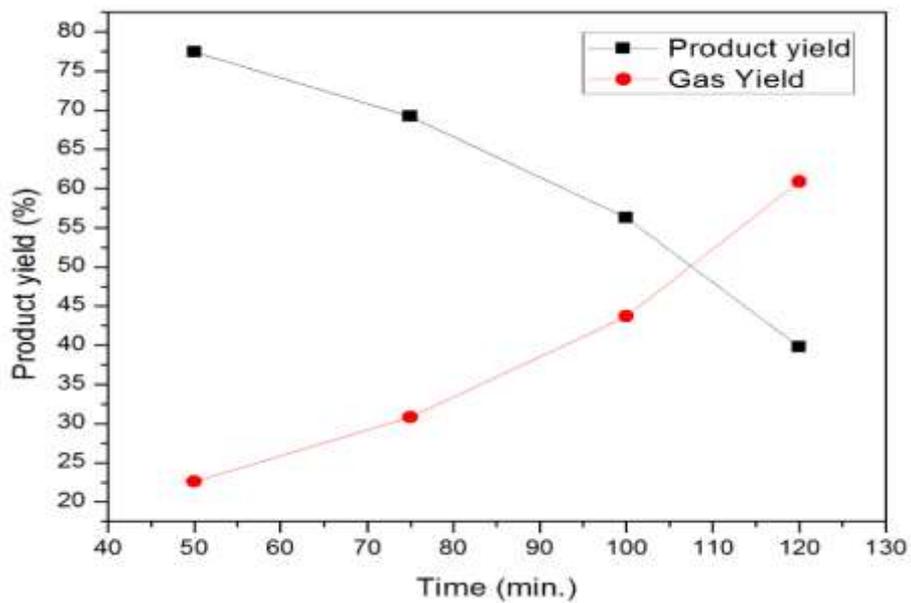


Figure. 8. Product yield at Temperature 400 °C

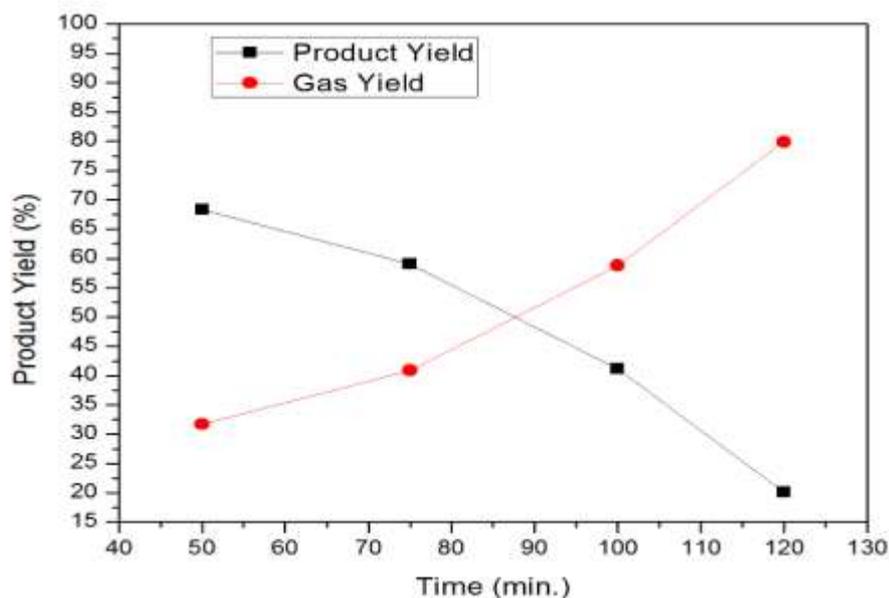


Figure. 9. Product yield at Temperature 450 °C

#### 4.5. Penetration Degree of PP wax

Penetration degree is especially used to measure the hardness of wax; it is related to the pyrolysis deepness of wax material with respect to the molecular weight range. Usually, penetration degree can be determined at room temperature of (25 or 40 °C). Figure 9 shows the PD of PP wax at a various temperature range of 350 to 450 °C and various time range of 50 to 120 min. From the figure, it can be analyzed that at low temperatures there is no major effect occurred in the penetration degree of wax. However with increasing pyrolysis temperature and time hardness of wax decreases and penetration degree increases. But at high temperature and high reaction time, it decreases suddenly. Henceforth, it was considered that 450 °C and 75 min is a suitable condition for wax formation. Furthermore with an increase in reaction time at a constant temperature the PP gets over pyrolysed hence at this condition the quality of obtained wax cannot meet standard property of wax. This similar statement also proved by the Jixing et al. for PE wax [11]. The obtained PD of wax ranging from 0.0 – 13.3 mm. The PD of another plastic wax was previously studied and shown that water sachets wax is having the range of 1 to 40.6mm [9]. Similarly Jixing et al. obtained the PD range of PE wax is 0.1–42.4mm [6].

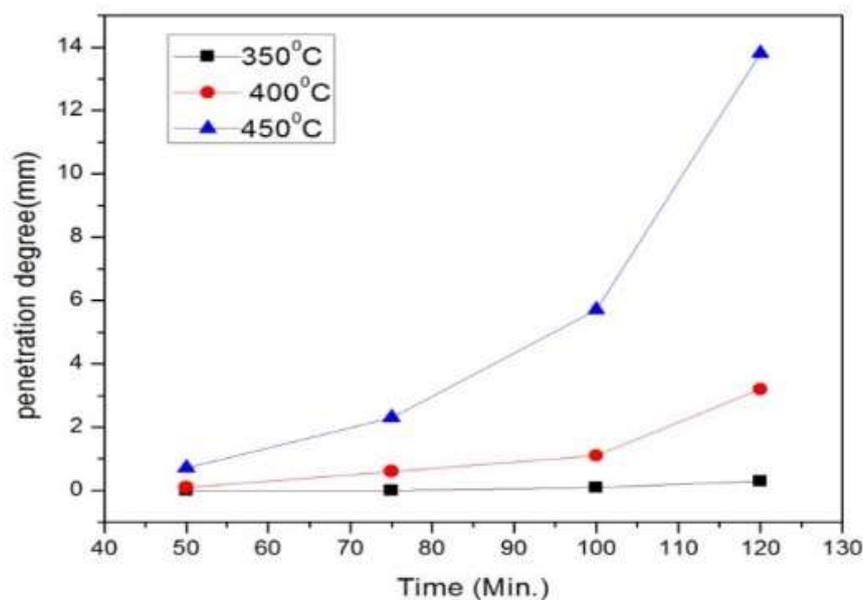


Figure. 10. Penetration degree at different temperature & time

#### 4.6. Melting point of PP wax

The melting point is one of the widely used physical property to study the quality wax. It also measures the molecular weight range of wax and the extent of pyrolysis of raw material. Figure 10 represent the melting point of PP wax at various conditions (temperature and time). The deepness of pyrolysis reduces the melting point of the wax. There are no significant changes was observed for PP wax at a lower temperature and low reaction time. However with the increase in temperature and reaction time, the MP of PP wax sharply decreases. The similar fashion was also observed in previous literature [19]. The obtained melting point of PP wax is 164-103 °C at 350 °C to 450 °C. Jixing et al., investigated pyrolysis of waste PE and obtained the wax with various operating conditions, they mentioned the melting point of PE wax is within the range of 144 °C-104 °C [6]. Ademiluyi et al. noted that the melting point of polyethylene wax varies from 86°C to 142 °C [9]. Similarly Umaru et al. obtained PE wax via pyrolysis of PE at various operating conditions of 200 °C and 250 °C for 1hour, 2hours, and 3hours respectively, and he found the melting point range of PE wax is 84-114 °C [16]. The melting point wax is varied for all the plastic wax this is due the different molecular structure of plastic and different operating conditions. As a consequence from the melting point characteristic, it was determined that 450 °C and

75 min is the effective operating conditions to obtain the suitability of wax. Furthermore, it was found that the obtained PP wax melting point property is matching with standard wax property [20].

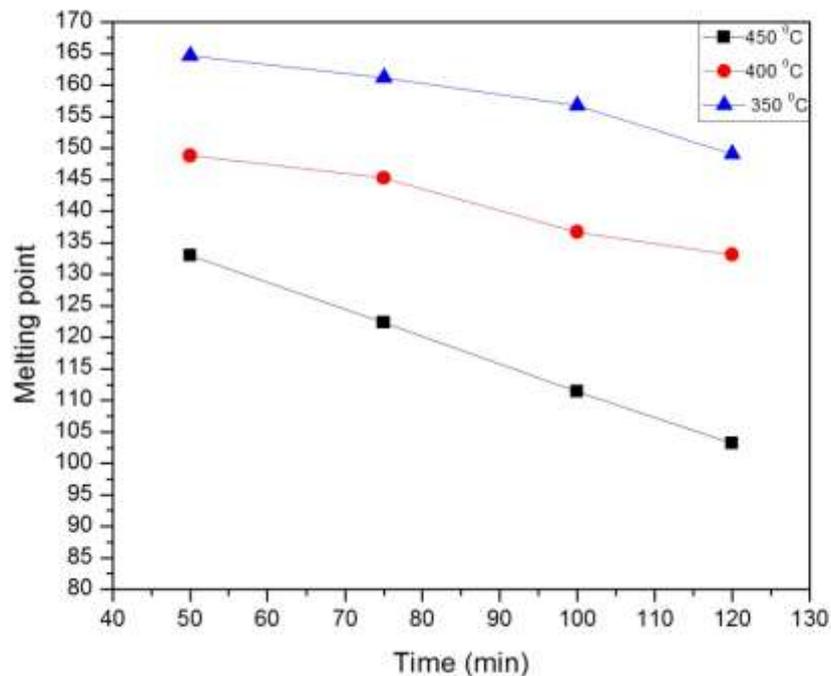


Figure. 11. Melting Point at different temperature & time

#### 4.7. Characterization of wax

##### 4.7.1. Oil content

The oil content of waxes is an indication of the degree of refining. Excess oil is giving a dull appearance and a greasy feel. Such type of wax cannot be suitable for many applications, particularly the manufacture of food wrappings, adverse effect on sealing strength. The oil content of wax may have significant effects on several of its properties, such as strength, hardness, flexibility, scuff resistance, the coefficient of friction, and coefficient of expansion, melting point, and oil straining [21]. However, the oil content of the microcrystalline wax is, 20%. Which is not considerable but in the present study the PP wax of oil content is 6.3% which can be favorable to used as wax in a further application. Waxes containing more than 20% oil would usually be classed as petrolatum, but this type of demarcation has not observed for oil contents of PP wax [22].

#### 4.7.2. Function group analysis

Fourier Transform Infrared (FT-IR) spectroscopy is an important technique which is based on the principle that almost all molecules absorb infrared light, excluding (He, Ne, Ar, etc.) and homopolar diatomic ( $H_2$ ,  $N_2$ ,  $O_2$ , etc.) molecules. On the interaction of infrared light with wax, the chemical bond will stretch, contract, and absorb infrared radiation in a definite wavelength range regardless structure of the rest of the molecules [23]. Based on this principle functional group present in the pyrolytic wax were identified. Figure 11 shows the FTIR spectra of PP wax obtained at optimum conditions of 450 °C and 75 min. Table 8 summarized the various functional group of PP wax corresponding to their wavelength range. The C-H scissoring and bending vibrations at  $2955.18\text{ cm}^{-1}$  and  $2855.7\text{ cm}^{-1}$  represent the presence of alkanes and alkenes. At  $1458.59\text{ cm}^{-1}$  the  $CH_2$  and  $CH_3$  deformation vibration also indicate the presence of alkanes. The peak at  $1376.83\text{ cm}^{-1}$  with O-H bending indicates alcohols and phenols. While the presence of acids and derivatives at O-C band was represented at  $1217.47\text{ cm}^{-1}$ . The obtained functional group of PP waxes also in agreement with the previous literature [7]. Further, the functional group determination was confirmed by GC-MS and  $^1H$ NMR analysis.

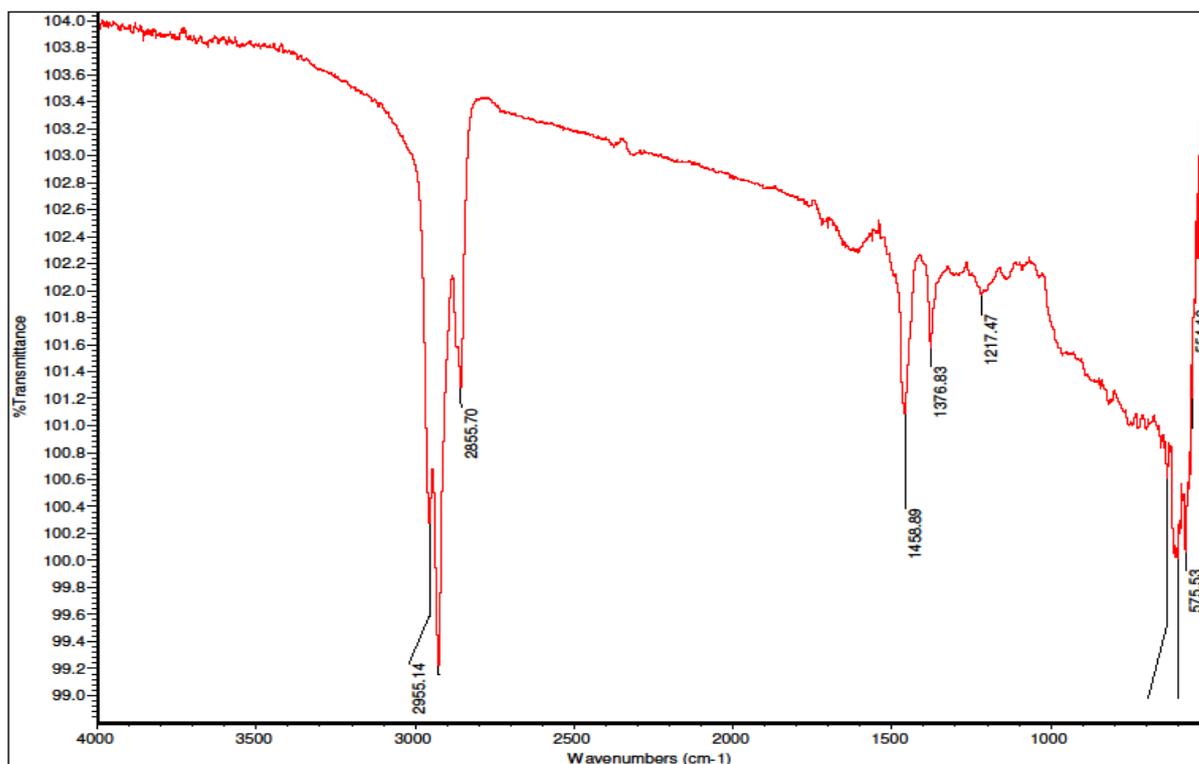


Figure. 12. The FTIR spectra of PP wax

Table 8. FT-IR functional groups of PP wax

Wavelength Range (Cm-1)	Class of compounds	Functional group
2955.14	CH <sub>3</sub> , CH <sub>2</sub> & CH	Alkanes
2855.70	CH <sub>3</sub> , CH <sub>2</sub> & CH	Alkanes
1458.89	CH <sub>2</sub> , CH <sub>3</sub> Deformation	Alkanes
1376.83	O-H bending (in-plane)	Alcohols & Phenols
1217.47	O-C Strech	Ether

#### 4.7.3. Elemental Analysis

The quantitative representation of carbon, hydrogen, nitrogen, sulfur and oxygen in wax is estimated and compared with paraffin wax which is presented in Table 9. From the table, It was established that the presence of carbon and hydrogen is more in PP wax and near about to paraffin wax. Moreover, the sulfur content slightly varies. However previously Barbara j Musser et al. mentioned that the Sulphur high sulfur

content may effect on H/C ratio. They provided the elemental analysis of different wax and shown the sulfur content range of 0.0% to 0.71% which is near to the current study. The H/C ratio of obtained PP wax is 1.96 which is near to paraffin wax. It was mentioned earlier, that H/C ratio is one of the important fuel characteristics to classify the fuel. High H/C ratio also indicates to the quality of fuel. Waxes with the lowest H/C ratios were translucent viscous liquids [24]. The existing empirical formulas for PP wax and paraffin wax  $CH_{1.9}$  and  $CH_{2.05}$  respectively.

Table 9. CHNS analysis of PP wax

Element	PP wax	Paraffin wax
C	85.21	85.25
H	13.98	14.59
N	0.00	0.00
S	0.43	0.26
O (by difference)	0.38	0.46
H/C ratio	1.96	2.05
Empirical formula	$CH_{1.96}$	$CH_{2.05}$
GCV(MJ/Kg)	44.2	46
Oil content	6.34	3-5

#### 4.7.4. GC – MS Analysis

GC-MS is one of the modern analytical techniques, which represent both qualitative and quantitative analysis of chemical compound [25]. GC-MS analyses were done for PP wax which produced at an optimum temperature and time of 450°C and 75 min respectively. The various peak of the chromatogram was figure out and shown in Fig. 12 with respect to the corresponding peak the compound were identified from W9N11 library. More than 20 compounds were identified which present in PP wax by GC-MS analysis, among which the high degree of probability ( $\geq 90\%$ ) and peaks areas around or greater than 0.2% are listed in Table 10 From the table it was shown that the different categories of compound were identified after the thermal decomposition of PP wax. The major compound in PP wax are are Cyclopentane-2-propenyl , Cyclohexene-1-methyl-4-(1-methylethenyl) , Bicyclo[2.2.1]heptane, 7,7-dimethyl-2-

methylene , 2-Nonenal, Ethyl-2 hexene-1,2-decenal, 8-Azabicyclo[3.2.1]octan-1-one. Cyclohexene-1-methyl-4-(1-methylethenyl) is used in flavoring, fragrance cosmetics, Insecticide, Insect and animal repellent, also used in paint/varnish/oil remover, fruit-scented lotions , Oil dispersant. 2-nonenal Used for soap manufacturing.

During the thermal decomposition of the poly olefinic compound, it produces most of aliphatic, compound due to initiation, propagation, termination as stated by umaru et al. The PP wax containing the carbon chain length of C<sub>8</sub>–C<sub>18</sub>. Most of the commercial wax were contains aliphatic and carbonyl compounds. Therefore, the presence of these compound in PP wax also proved that this can be used as a wax.

Table 10. GC-MS analysis pyrolytic wax

R.Time	Area %	Compound Name	Chemical Formula
3.317	0.64	Benzocyclobutene	C <sub>8</sub> H <sub>6</sub>
4.012	0.84	2,6,6-Trimethylbicyclo[3.1.1]hept-2-ene	C <sub>18</sub> H <sub>22</sub>
4.832	0.29	2-Methylcyclobuta[b]pyridine	C <sub>8</sub> H <sub>7</sub> N
4.961	2.28	4-Pyridinamine	C <sub>5</sub> H <sub>5</sub> N <sub>2</sub>
5.724	73.14	Cyclopentane, 2-propenyl	C <sub>8</sub> H <sub>14</sub>
6.196	4.28	Cyclohexene, 1-methyl-4-(1-methylethenyl)	C <sub>10</sub> H <sub>16</sub>
6.710	0.22	3-Pyridinecarboxamide, 1,6-dihydro-6-oxo	C <sub>6</sub> H <sub>6</sub> N <sub>2</sub> O <sub>2</sub>
8.500	0.57	2-Propenamide, N-(1,1-dimethylethyl)	C <sub>7</sub> H <sub>13</sub> NO
11.380	0.40	Hexnal	C <sub>6</sub> H <sub>12</sub> O
12.988	0.21	Formic acid, oct-2-yl ester	C <sub>9</sub> H <sub>18</sub> O <sub>2</sub>
13.865	0.36	1,1,4,7,7-Pentamethyl-4-azaheptane	C <sub>11</sub> H <sub>25</sub> N
15.525	2.42	Bicyclo[2.2.1]heptane, 7,7-dimethyl-2-methylene-	C <sub>10</sub> H <sub>16</sub>

15.697	0.66	L-Methionine	$C_5H_{11}NO_2S$
15.780	0.66	8-Azabicyclo[3.2.1]octan-1-one	$C_7H_{12}NO$
16.091	0.80	3-Fluorobenzoic acid, 4-methoxy-2-methylbutyl ester	$C_{13}H_{17}FO_3$
16.262	1.46	2-Nonenal	$C_9H_{16}O$
19.702	0.50	N,N-Diethyl-1-methyl-1,3-propanediamine	$C_8H_{20}N_2$
21.534	0.39	4-Piperidinecarboxylic acid hydrazide	$C_6H_{13}N_3O$
22.338	0.27	Ethyl-2 hexene-1	$C_8H_{16}$
23.801	3.68	8-Propyl-1,5-Diazabicyclo[3.2.1]-Octane	$C_9H_{18}N_2$
24.772	1.64	Hexa-4,5-diene carboxylic acid	$C_7H_{10}O_2$
25.322	2.01	8-Azabicyclo[3.2.1]octan-1-one	$C_7H_{12}NO$
25.472	0.88	2-decenal	$C_{10}H_{18}O$

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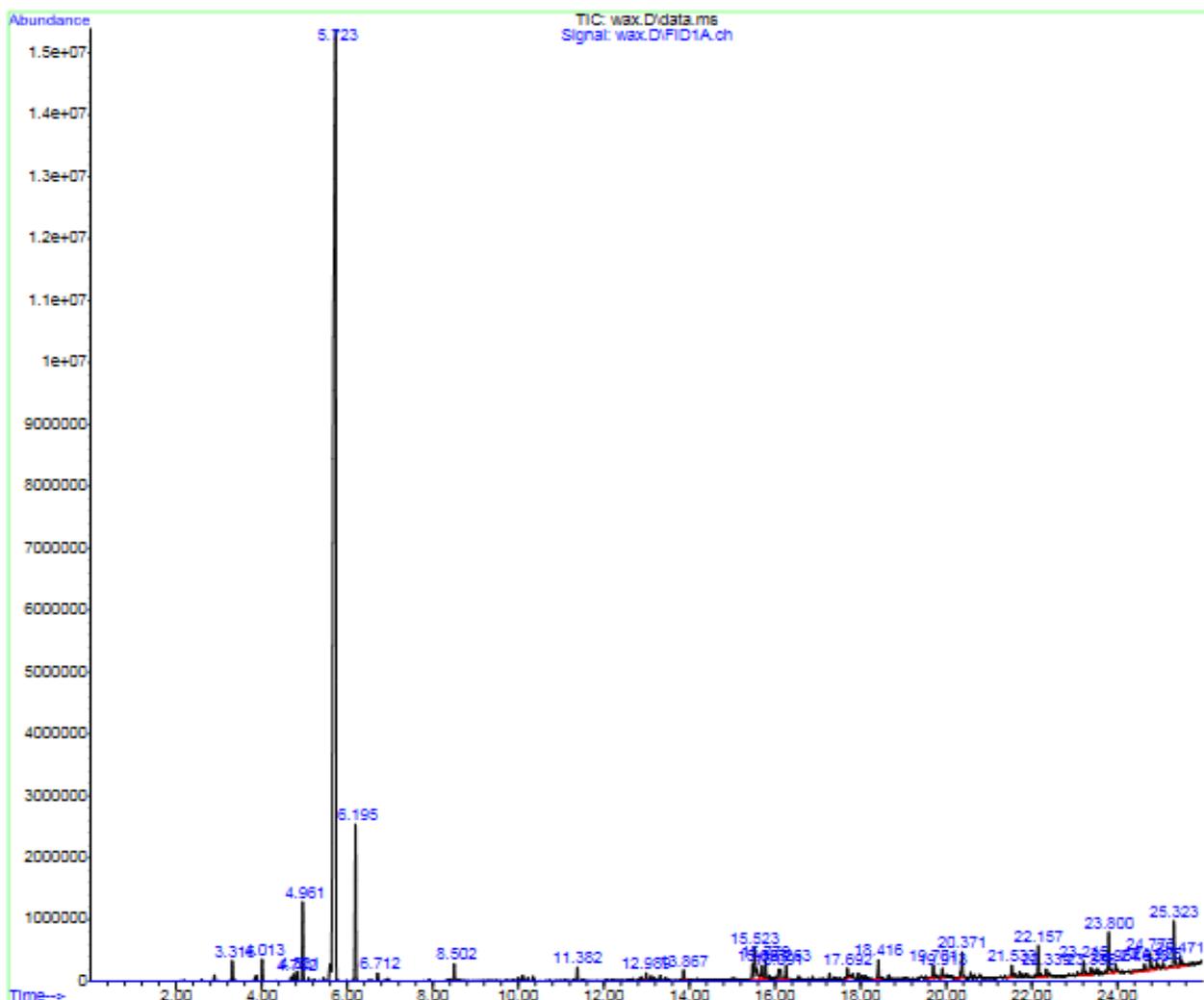


Figure. 13. Mass spectra of PP wax

#### 4.7.5. PROTON NUCLEAR MAGNETIC RESONANCE (NMR)

Nuclear magnetic resonance (NMR) spectroscopy is a best analytical technique for structure analysis of small and large molecules, where standard instruments use superconducting magnets that generate high magnetic fields [26]. However, widespread use of high-field NMR is limited by the cost and complexity of the equipment. From the past, considerable progress has been achieved with compact NMR spectrometers employing permanent magnets, which provide sufficient sensitivity and robustness for applications outdoors and on the factory floor. Percentage hydrogen distribution was computed on the basis of chemical shift values from the  $^1\text{H}$  NMR spectra. Fig. 13

shows the  $^1\text{H}$  NMR spectrum of PP wax which provides detailed information on aromatic, olefinic and aliphatic based on the proton type. From the graph, It observed that the peaks are found between the 0.89 – 2.15 ppm .and this is in the range of paraffin, furthermore peaks are showed between 4-5 ppm this fall in the range of olefins. It can be seen that in the PP wax has a much lower ratio of methylene to methyl carbons this implied a number of branches larger than those the other waxes [27].

Graph 13 shows that a total number of hydrogens are 88% mostly 97.7% of hydrogen are saturated, and 2.3 % hydrogen is olefinic. This has also confirmed through GCMS and FTIR analysis.

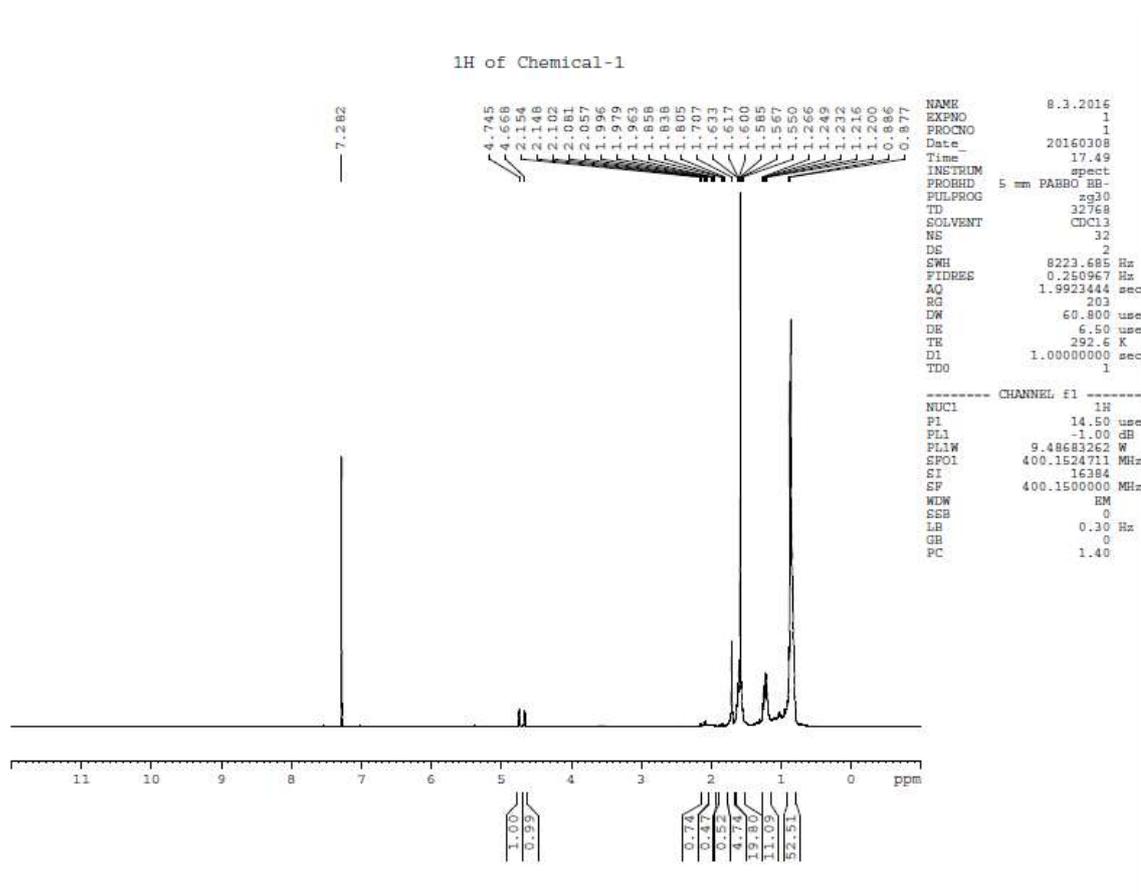


Figure. 14.  $^1\text{H}$  NMR of PP wax

#### 4.7.6. SEM Analysis

Scanning electron microscopy is widely used for physical and chemical characterization of solid materials [28]. Scanning electron microscopy is widely used for surface morphology study [29]. The PP wax obtained at optimum conditions were

analyzed by SEM at three different magnifications of (2000X, 2500X, and 10000X). Figures - to - show the SEM micrographs for PP wax with the three magnifications of 2000X, 2500X, and 10000X respectively. From the SEM graph, it was observed that wax is non-porous, homogeneous distributed, dusty and colloidal in nature.

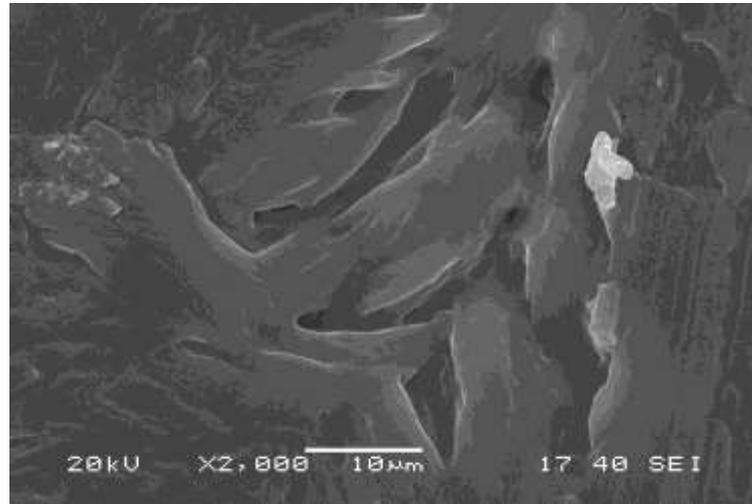


Figure. 15. Micrographs of PP wax at 2000 magnification

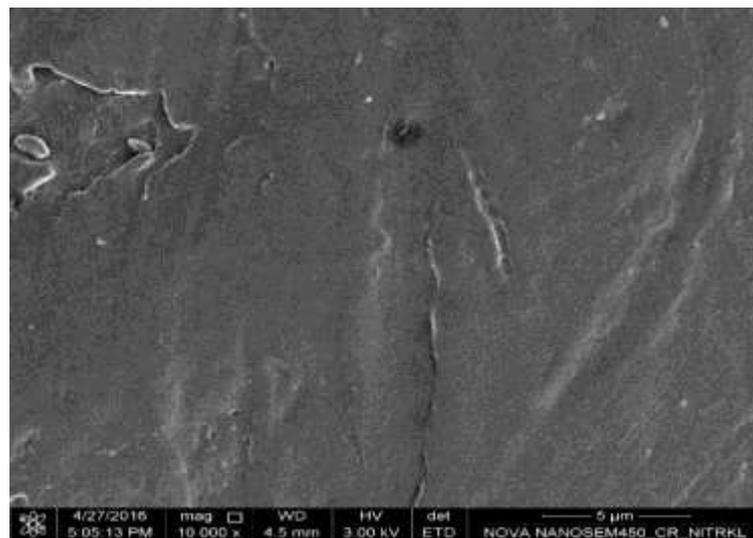


Figure. 16. Micrographs of PP wax at 10000 magnification



Figure. 17. Micrographs of PP wax at 10000 magnification

# CHAPTER 5

## 5. CONCLUSION

Pyrolysis of PP plastic has been attempted in this studies with respect to the various temperature range of 350 to 450 °C and the different time range of 50 to 120 min. The effect of temperature and reaction time on product yield of PP were studied. At low temperature and low time there is no significant change was observed on the product yield, but at a certain temperature with an increase in reaction time, the product yield becomes decreases. However, with the increase in reaction time and temperature, there is a great change of product yield was observed. The maximum 59% of wax obtained at an optimum condition of 450°C at 75 min. Based on the physical and chemical characterization studies the following conclusion are as follows, the physical analysis like penetration degree, melting point analysis, SEM analysis shows that at low temperature there is no major effect occurred in the penetration degree of wax but with increasing in pyrolysis temperature and reaction time hardness of wax decreases and penetration degree increases. The obtained PD of wax ranging from 0.0 – 13.3 mm. The obtained melting point of PP wax is ranging from 164-103 °C at 350°C to 450°C. Whereas the MP of wax at optimum conditions is 122 °C, the oil content of PP wax is 6.3% which is one of the favorable conditions to prove that the wax can be used in petroleum industry. Furthermore, the chemical analysis studies like functional group analysis and GCMS analysis concluded that the abandoned compound is Cyclopentane, 2-propenyl which is aliphatic and few of them are aromatic in nature. The most of the compound contains 97.7% of hydrogen which are saturated, and 2.3 % hydrogen are olefinic was proved through HNMR analysis. The calorific value of wax is 43MJ/kg, which is near about to standard fuels. SEM analysis resulted that PP wax is non-porous, homogeneous distributed, dusty and colloidal in nature. From the above it can be concluded that the recycling process like pyrolysis is one of the effective method for the production of wax which is one of the primary product of pyrolysis it can be useful in chemical industry or can be useful as an alternative feed in the FCC cracking process in refineries. This effective recycling process also reduces the production of waste with that control the environmental pollution.

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