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Study of Properties of Local Polypropylene Compared to International Standard Properties

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ABSTRACT

This study was conducted in Khartoum refinery in Algylee to study the properties of polypropylene (grade113 &114) which are produced by addition technique; they used refined propylene from Liquefied Petroleum Gas and Ziegler-Natta catalyst. The samples were taken from samples which are produced in (1-15)/08/2010 in three features (powder, granulates and specimens). Both grades are homopolymer and isotactic polypropylene. They checked the component of liquefied petroleum gas by using a gas chromatography instrument and determination of thermal, mechanical, and physical properties. From the result, both grades differed in their properties according to molecular weight. Grade 113 has a large molecular weight which is suitable for extrusion process while grade 114 has a small molecular weight which is suitable for injection molding process. Each grade of polypropylene is within the standard range but they contain a slight amount (<0.5ppm) of sulfur and moisture (49.1ppm) which are affecting product quality. However, if there is no separation unit the product will become yellowish and toxic.

Keywords: Refined propylene, Polypropylene, Ziegler-Natta catalyst, Liquefied petroleum gases, and KPC.

INTRODUCTION:

As early as 1969 propylene was polymerized by Berthelot which did not exhibit interesting properties for industrial application? In 1955 polypropylene was first polymerized by G. Natta following the work of K. Zeigler which resulted semi-crystalline with has strong mechanical properties (Karian, 2003; Gowariker, 1986). Polypropylene is linear hydrocarbon polymer, the steric arrangement of the methyl group attached to every second carbon atom in the chain may vary produce three different kinds of polypropylene; isotactic poly-propylene (methyl group on the same side), syndiotactic polypropylene (pendant methyl group in alternating manner), atactic polypropylene (pendant methyl group in a random manner). Polypropylene is produced two commercially types depending on the properties desired; polypropylene homopolymer contains propylene monomer and polypropylene copolymer contains two or more different types monomer (Karger-Kocsis, 1995; Tripathi, 2002). Polypropylene was prepared from propylene monomer which produced by cracking of petroleum product by using fractional distillation

and Ziegler-Natta catalyst. A typical catalyst system prepared by reacting titanium trichloride ($TiCl_3$) with aluminum triethyl, aluminum tributyl or diethyl mono-chloride in naphtha under nitrogen form slurry. There is new catalyst system which contained magnesium compound will give appreciable improvement in the yield of isotactic material. Both those system were including liquid and gas phases which must need separation unit to remove catalyst residues and atactic material (Brydson, 1999; Cowie, 2007; Lahcene, 2021).

The mechanical and thermal properties of polypropylene dependant on the isotacticity, molecular weight and its distribution, crystallinity. Good balances of properties can be tailored to large fabrication methods and application and low cost make it useful in various1 industries such as automotive, medical application appliance application, textile and nonwovens, packageing, construction (3, 4). Polypropylene is already recycled from a numbers of commercial sources; crates, trays, battery case etc. The recovered material is reused within the automotive industry and for a proprietary rang of furniture (Brydson, 1999; Maier, 1998; Ahmad *et al*., 2018). In this study the material is was polypropylene which was produced in Sudan by Khartoum Petrochemical Company (KPC).

They were used Ziegler-Natta catalyst and propylene as monomer which was obtained from refining liquefied petroleum gas (LPG) by using fractional distillation. There are four units are used to produce polypropylene in (KPC).

1) Gas distillation unit contained four distillation columns, firstly components above C4 were removed, secondly C3, thirdly propane and propylene and fourthly refined propylene and sulfur and moisture are removed.
2) Polymerization unit includes (1) raw material refinery process where propylene refined and removed non hydrocarbon impurities such as water, sulfur and oxygen. All refinery processes are involved alkali dehydration, desulfurization, re dehydration, gas phase de carbon monoxide, de oxygenation and deep dehydration. (2) propylene polymerization in which refinery propylene is transferred via two different ways to polymerization kettle, one from the activator pipe the other from catalyst feed. The reaction was occurred under temperature 74°, pressure 3.5 MPa for 3 - 4 hours till the kettle becomes dry then collected propylene no reacted.
3) Flash vapourization and deactivation unit after the gas in the polymerization kettle is recycled by force of the remained pressure then refined nitrogen gas to displace the air in the flash vapourization kettle then feeding. To discharge the powder from the flash vapourization used rotary valve.
4) Pressing and granulating unit stabilizer materials were added to polypropylene powder and the mixture was pressed into pelletization machine and cut in granulates form. Fourth packing and delivering unit where (poplypropylene granulates) is packed in certain weight and storage.

MATERIALS AND METHODS:

Samples

Liquefied petroleum gas, crude propylene gas, refinery propylene gas, two grades of local polypropylene (grade 113 & grade 114) in three forms (semi product (polypropylene powder), final product (polypropylene granulates) and specimens).

The analysis Method

For analysis was used ASTM standard method.

Determination of liquefied petroleum gases

This test used to determine the composition of liquefied petroleum gases (LPG) and it is applicable to analysis of propane, propylene, butane and sulfur

Apparatus

Gas chromatography instrument with thermal conductivity detector, a strip-chart recorder.

Procedure

The GC instrument was operated (used nitrogen gas as carrier gas LPG sample, crude and refiner propylene and argon gas to determine sulfur). Then the sample was injected and recorded the result (ASTM, D 2163-9)

Melt flow rate of polypropylene

This test measure the rate of extrusion of molten resins through a die of specific length and diameter under prescribed conditions of temperature, load and piston position in the barrel as timed measurement is made by

using a manual cut off operation based on time used for materials having flow rates that fall.

Apparatus

Melt flow index device consist of a dead - weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a die at the lower end and a weighted piston, funnel, tool for cutting, sensitive balance and loading weight.

Procedure

6.0g of samples 2 (powder, granule) was weighed , 5 cm3 of heating stabilizing solution were added and the mixture was in oven at 100°C for 15 min. the cylinder was charge with the sample, reinsert the piston and add the appropriate weight. Few minutes waited until the material was soften and begin to melt then was purged to a position, the purge was completed at least 2 min prior then for material was started. For all tests collecting a time extrudate (t) was started when requirement for the position are met. Once the extrudate is cool, it was weighed (G). For final product (granule) applied the same procedure but without adding the heating stabilizing the melt flow rate was calculated in grams per 10 min

MFR= (G×600)/t …….. (1)

Where

MFR ≡ melt flow rate, G ≡ weigh of extrudate, t ≡ cutting interval time. (ASTM, D 1238-04c)

Deflection temperature of polypropylene

This test determined the temperature at which an arbitrary deformation occurs when specimens are subjected to an arbitrary set of testing conditions.

Apparatus

Deflection Temperature Instrument which consists of specimen supports, immersion silicon oil bath, deflection measurement device, weight, temperature measurement system.

Procedure

To2 determine the temperature at which an arbitrary deformation occurs when the specimens are subject to an arbitrary set of testing conditions. Specimens were prepared in plate form by injection molding with thickness 3.2mm, width 13mm and length 127mm. The apparatus was prepared which arranged to shut off the heat automatically with sound an alarm or record the temperature when the deflection has been reached. The test condition was checked; at room temperature, immersion medium silicon oil heating rate 2°C/ min temperature at the start of the test 35°C, preheating 300s. The test specimens edgewise were positioned in the apparatus and ensure that they are properly aligned on the supports. The road rod was applied to the specimen and lowers the assembly into the bath and the machine was stared. The temperature (heat-transfer medium) was recorded when the specimen has deflected specified amount at the specified fiber stress. (ASTM, D648-98c)

Density and specific gravity of polypropylene

Apparatus

Analytical balance is equipped with stationary support for the immersion vessel above it, sample holder and sinker. Procedure: The mass of specimen was weighed in air (m1). The immersion vessel was put on the support and completed by distilled water and the specimen was weight after immersed (m2) then the specific gravity was calculated

$$\mathbf{Dg/dm^3 = m^1/m^2 \times 997.5} \quad \text{…….. (2)}$$

Where D ≡ specific gravity, m1≡ weigh of specimen in air, m2 ≡ weigh of specimen completed immersion (ASTM, D792-08).

Impact resistance of polypropylene

This test determined the impact resistance which based on the polymer to standardized pendulum-type hammers, mounted in standardized machines in breaking standard specimens with one pendulum swing

Apparatus

Cantilever beam (Izod-type) impact machine, engine lathe and a notch-depth micrometer.

Procedure

The molded specimens were prepared using injection molding with width between 3.0 – 12.7 mm, thickness is between 3.0 – 12.7 mm, and length is 62 mm. The specimens were notched by using engine lathe. The included angle of notch should be 45° with a radius of curvature at the apex of 0.25 mm, the depth of specimen under the notch should be 10.20 mm. The test condition was checked; temperature 23°C, and pendulum hammer energy is 0.5 J. The specimen positioned

precisely clamped in the vise, operated the machine and selected a pendulum of suitable energy. The pendulum was released and recorded the excess energy remaining in the pendulum after breaking the specimen (E), and the average Izod impact resistance (I) of the group of the specimens was calculated

I = (E J ×1000)/w …….. (3)

Where

I ≡ impact resistance J/mm, E ≡ break energy J, W ≡ width of specimen mm. (ASTM, D256 – 97)

Tensile property of polypropylene

This test determined the tensile properties in form standard dumbbell shaped test specimen under condition pretreatment, temperature, humidity and testing machine speed.

Apparatus

Testing machine of constant rate of cross head movement type comprising fixed member, movable member, grips, drive machine, load indicator, crosshead extension indicator.

Procedure

Specimen were prepared in plate form by injection molding with thickness 3.2mm, width of narrow section 13mm, length of narrow section 27mm. width over all 19 mm, length over all165 mm. the test condition checked; at room temperature, load range 2000 N gauge length 50 mm, test speed 50 mm/min. the specimen was place in the grips of the test machine and the test machine was started then the result was recorded. (ASTM, D 638 – 08)

Flexural property of polypropylene

This test determined the flexural properties in form standard dumbbell shaped test specimen under condition pretreatment, temperature, humidity and testing machine speed

Apparatus

Testing machine of constant rate of cross head movement type comprising fixed member, movable member, grips, drive machine, load indicator, crosshead extension indicator.

Procedure

Specimen were prepared in plate from injection molding with thickness 3.2 mm, width 13 mm and length 127 mm. the test condition was checked ; at room temperature, load range 100 N, extension range 10% and test speed 1.3 mm/min, span 50 mm and end point 9 mm. the specimen was placed in the grips of the machine

RESULTS AND DISCUSSION:

Liquefied petroleum gas

Table 1: Components of Liquefied Petroleum Gas Sample.

Component Formula	CH_4	C_2H_4	C_2H_6	C_3H_8	$C_yC_3H_6$	C_3H_6	C_4H_{10}	C_4H_6	C_4H_8	C_5H_{12}
ASTM Standard Range %	0.1-0.3	0.00-0.85	0.01-1.64	2.88-11.58	0.01-0.01	17.8-46.11	21.64-36.57	0.01-0.42	18.64-44.9	0.01-2.24
Test Result %	0.1	0.1	0.16	8.95	0.01	37.57	26.68	0.19	28.04	0.66

From the **Table 1** it was found that LPG contains percentage of 8.95% propane, 37.57% propylene, 26.68% butane, 28.04% isobutylene which may be monomers for manufacture of polymer.

Table 2: Components of Sample Outlet Firstly Distillation Column.

Component Formula	C_3H_8	$C_yC_3H_6$	C_3H_6	C_4H_{10}	C_4H_6	C_4H_8	C_5H_{12}
ASTM Standard Range %	0.04-0.40	0.01-0.27	0.01-0.12	18.55-62.70	37.08-81.09	18.64-44.9	0.01-4.15
Test Result %	0.15	0.04	37.57	42.74	56.46	28.04	0.66

Table 3: Components of Sample Outlet Secondly Distillation Column.

Component Formula	C_2H_4	C_2H_6	C_3H_8	$C_yC_3H_6$	C_3H_6	C_4H_8
ASTM Standard Range %	0.00-0.00	0.01-4.61	10.76-34.25	0.01-0.02	65.53-88.62	0.01-0.80
Test Result %	0.1	0.34	20.47	0.32	78.98	0.02

Table 4: Components of Sample Outlet Thirdly Distillation Column.

Component Formula	C_2H_6	C_3H_8	$C_yC_3H_6$	C_3H_6	C_4H_8
ASTM Standard Range %	0.01-0.49	18.52-32.25	0.01-0.01	67.72-81.10	0.01-0.19
Test Result%	0.03	20.41	0.01	79.56	0.01

Table 5: Components of Sample Outlet Fourthly Distillation Column.

Component Formula	C_3H_8	$C_yC_3H_6$	C_3H_6	C_4H_{10}	C_4H_8	C_5H_{12}	Other
ASTM Standard Range %	61.79-87.84	0.04-0.09	11.98-36.57	0.01-12.44	0.01-8.32	0.02-0.15	0.01-0.32
Test Result %	72.33	0.06	27.35	0.15	0.2	0.11	0.10

From **Table (2, 3, 4, 5)** it is noticed the sample contained 42.74% butane and 56.46 isobutylene which are isolated in the first column and recycled. The likely amount of isobutylene may be used to prepare polyisobutylene ortance forthat impmanufacture of inner tubes for truck and bicycles.

Table 6: Components of Crude Propylene.

Component formula	≤C_2H_6 ppm	C_3H_8 ppm	C_3H_6 %	C_4H_8 ppm
ASTM Standard Range %	0.00-1096	18-9310	99.07-100	4-27
Test Result %	161	1588	99.85	16

Table 7: Components of Refined Propylene.

Component Formula	C_3H_4 ppm	C_3H_8 ppm	C_3H_6 %
ASTM Standard Range %	0.00-409	422-3597	100
Test Result %	1582	16	99.68

From **Table 7** the amount of propyne in crude propylene and the refined propylene is over the rate but didn't effect because is very reactive and can be convert to propylene in the reactor when present of hydrogen gas.

Table (8, 9) showed that refined propylene contained < 0.5ppm of sulfur and 49.1ppm moisture content that effect on polypropylene properties such as melt flow rate, strength and on life time of the specimens.

Table 8: Sulfur Content.

Component formula	LPG ppm	Crude propylene ppm	Refined propylene ppm
ASTM Standard Range %	4.0- 63.0	0.6-55.3	0.00
Test Result %	32.7	7.9	< 0.5

Table 9: Moisture Content.

Component Formula	Crude propylene ppm	Refined propylene ppm
ASTM Standard Range %	91.9-993	1.9-13.2
Test Result %	3.5	49.1

Properties of Polypropylene

Melt Flow Rate

Table 10: Malt Flow Rate of Polypropylene Grade (113&114).

Kind of Polypropylene		Malt Flow Rate	
		ASTM Standard Range	Test Result
Grade 113	Powder	2-5	2.707
	Granules		2.883
Grade 114	Powder	10-12	9.948
	Granules		10.258

Table 10 shows that melt flow rate each powder and granules of polypropylene grade 114 shows greater than polypropylene grade 113. The lower melt flow rate is indicated greater viscosity and higher molecular weight which effect on the processes thus extrusion processes which required high molecular weight must be used polypropylene grade 113 and injection processes which required low molecular weight must be used polypropylene grade 114.

Table 11: Heat Deflection Temperature of Polypropylene Grade (113 &114).

Kind of polypropylene	Heat deflection temperature	
	Test result °C	ASTM standard range °C
Grade 113	80.38	85-90
Grade 114	84.57	

However heat deflection temperature dependent on the molding process thus from **Table 11** it can be seen that heat deflection temperature for two grades of polypropylene are less than standard value due to injection molding specimens. This test is suited to control and development work.

Table 12: Specific Gravity of Polypropylene Grade (113 &114).

Kind of Polypropylene	Specific Gravity at 23°C Kg/cm^3	
	Test Result	ASTM Standard Range
Grade 113	901.2482	900-907
Grade 114	897.7839	

Specific gravity is property useful to identify material, to follow physical changes in the sample, to indicate the degree of uniformity among different sampling units and calculating strength weight and cost weight ratios. **Table 12** shows the different values of specific gravity for two grades of polypropylene due to the difference in crystallinity properties of the polymer.

Table 13: Impact Resistance of Polypropylene Grade (113 &114).

Kind of polypropylene	Break energy (E) J	Impact resistance (I) J/m
Grade 113	0.152	47.57
Grade 114	0.1389	43.407
ASTM standard range	0.150	27-117

Table 13 shows that polypropylene grade 113 has higher impact resistance than polypropylene grade 114 due to the increase in molecular weight.

Table 14: Tensile Property of Polypropylene Grade (113 &114).

Kind of polypropylene	Tensile strength Yield %	Percent elongation yield %	Tensile strength at Break MPa	Percent elongation at Break MPa
Grade 113	36.54	22.44	13.456	895.8
Grade 114	33.689	27.66	21.426	1041.3
ASTM standard range	12-43	1-23	19.7-80	1500

Table 15: Flexural Modulus of Polypropylene Grade (113 & 114).

Kind of Polypropylene	Flexural Modulus
Grade 113	1347.1
Grade 114	1333
ASTM standard range	1261.5

Table (14, 15) show that polypropylene grade 113 has higher value of tensile property and flexural modulus than polypropylene grade 114 due the different in molecular weight, crystallinity and orientation.

Table 16: Comparison Polypropylene Grade (113 &114) with other Polymers (3).

Property	Standard method	Test Result		Other Materials		
		PP Grade 113	PP Grade 114	LDPE	HDPE	PVC
Specific gravity Kg/cm^3	ASTM D792-08	901.2482	897.7839	0.92	0.96	1.4
Impact resistance at 23°C J/m	ASTM D256-97	47.57	43.407	1065	1500	200
Tensile strength MPa	ASTM D638-08	36.54	33.689	10	32	51
Flexural modulus MPa	ASTM D256-97	1357.1	1333	300	1300	3000
Deflection temperature °C	ASTM D648-98c	80.38	84.57	50	75	70

It can see from the table above polypropylene in both grades (113, 114) advantages as higher strength than high density polyethylene (HDPE) and low density polyethylene but lower than poly vinyl chloride (PVC) also they offer resistance to higher temperature and low impact resistance thus polypropylene is good toughness and is referred to use in many application such as transfer liquids, furniture, domestic appliance and automotive (Mostari *et al*., 2020; Awadala *et al*., 2020).

CONCLUSION AND RECOMMENDATIONS:

Polypropylene was produced in Sudan is homopolymer as recycling process of petroleum waste. They are product two grades of polypropylene grade 113 and grade 114, they are different in length of polymer chain; molecular weight. LPG contains enough amounts of gases which can be used as monomers to produce various polymers such as polypropylene, polyethylene and poly-isobutylene. Also it contains a large percentage of propylene but contain sulfur and moisture which affect in production quality. Both grade of polypropylene (113 & 114) offer good thermal, mechanical and physical properties from the result of MFR of both grades concluded that grade 113 has higher molecular weight than grade 114. Both grades may be oriented either in the melt phase or by stretching when it is solid due to high tensile strength also low impact resistance leads to brittle failure. They have low density so are light therefore offer the advantage of being able to manufacture more items for a given weight of polymer. They are good thermal resistance (high heat deflection temperature) then they are useful application which needs high temperature as microwave. Since there is no separation unit (to separate the catalyst residue and byproduct "atactic polypropylene") so it may be toxic. Constructor many factor to produce the polymer from refinery of LPG, constructor unit to treat a large amount of propane in the refined propylene, increased activity of column to remove sulfur and moisture to improve quality of product and added separation unit.

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CONFLICTS OF INTEREST:

The authors declare that they have no conflict of interests.

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