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Investigation on Physical and Mechanical Properties of High Density Polyethylene (PE100) Using Novel Catalyst

M. A. Nikoohemmat[a], H. Mazaheri[a], A. H. Joshaghani*[a], E. Joudaki[b]

[a] *Department of Chemical Engineering, Arak Branch, Islamic Azad University, Arak, Iran*
[b] *Department of Chemical Engineering, Faculty of Engineering, Arak University, Arak, Iran*

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A B S T R A C T

Novel catalyst called super active catalyst for the production of high density polyethylene (HDPE) pipes grade 100 was prepared and localized for the first time in Iran. The purpose of this paper is to evaluate the performance of the new catalyst and compare it with Finix-112 catalyst which is a commercial catalyst for the production of HEPE grade 100. Extensive experiments were performed on the physical and mechanical properties of the product using both catalysts including melt flow index, particle size distribution, volatility, density, bulk density, scanning electron microscope-energy dispersive x-ray analysis, charpy impact strength. Comparison of the results showed that the hydrogen responsibility performance of the product with super active catalyst is 19%, charpy impact is 16.7% and pressure condition in first reactor about 28% was better than product with Finix-112 catalyst. Home-made super active catalyst can be a good alternative to imported finix-112 catalyst and save a considerable amount of foreign currency.

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NOMENCLATURE

SAC-510	Super activa catalyst	PE100	Polyethylene pipe grade of 100
PSD	Particle size distribution	g	Gravity (m/s^2)
MFI	Melt flow index	d	Diameter
GPC	Gel permeation chromatography	µ	Micron
HDPE	High density polyethylene		

1. INTRODUCTION

High density polyethylene (HDPE), because of its high tensile strength, is the most widely used plastic for the production of water pipes, packaging, butter tubs, garbage containers and oil and gas distribution network in the form of homopolymer and composite [1, 2]. Balanceing between mechanical properties and processablility of polyethylene is one of the key issues for better performance of the polymer [3]. However, HDPE production using Ziegler-Natta catalyst did not have the desired quality and longevity [4]. Many efforts have been done to modify this heterogeneous catalyst which plays an important role in the polymer morphology [5-10]. Upgrading the structure of the catalyst and changing some of its manufacturing instructions will increase the quality of the final products [11]. The effectiveness of quality improvement is achieved by measuring the physical and mechanical properties of pipe raw materials. Measurements of mechanical and physical properties can be obtained by performing some tests such as MFI, GPC, charpy impact and density. HDPE-PE100 is the best choice for replacement with concrete and steel pipes in terms of performance; it has unique properties that can withstand more pressure with more density and less weight and thickness [12]. The impact of the usage of new and advanced catalysts can be examined in two main factors: 1. Hydrogen responsibility, 2. Butylene

*Corresponding Author Institutional Email: *a-hasani@iau-arak.ac.ir*
(A. Hassani Joshaghani)

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responsibility [13, 14]. The effect of the mentioned factors prevents the loss of consumption of valuable materials of hydrogen and butylene, reduces the by-product of wax and the costs of the production process [15].

Production of PE100 pipe grade raw materials in Iran's petrochemical industry is based on slurry, bimodal and under Hostalen technology. Finix-112 catalyst is classified as Ziegler-Natta catalysts and is used in these industries to produce PE100, which is imported and it is too expensive. Some problems using finix-112 includes difficult process control; high hydrogen injection and high operating pressure in reactors, which leads to high hydrogen emissions and costly production. To overcome this problem, super active catalyst with a unique formulation and nature was made to produce PE100 pipe grade for the first time in Iran. Then the results of product at laboratory scale and pilot plant were compared with both catalysts. The purpose of this paper is to investigate the physical and mechanical parameters of PE100 pipe grade using SAC-510 catalyst with improved structure, as well as removing barriers to production, including hydrogen responsibility, ease of production process and economic saving while maintaining product quality.

2. MATERIALS AND METHODS

2. 1. Sieve Analysis in HDPE The determination of the particle size distribution by sieve analysis is a more accurate and informative method compared to the bulk density method. The particle size distribution is determined by sieve analysis under an air jet. Equipments include balance, precision (500 g, 0.01 g), air jet sieve (0.063 – 0.500 mm), and ultra sonic cleaning device. About 25 g of powder is weighed on each sieve and the remaining powder is determined, after machining and suction [16].

2. 2. Melt Flow Index (MFI) of HDPE The melt flow rate depends on the polymer molecular weight and hence specifies individual product types. The melt flow rate can be used as a criterion for a first evaluation of their flow properties. Alternatively, the volume flow rate may be indicated. Determinations according to ISO 1133 are obligatory, in particular, for the final inspection of product batches destined for sale, and render binding results for delivery specifications, works certificates, etc. Equipment includes 50XC9001A-D (Melt viscosity test instrument), 50XC9002 (Balance, analytical (200 g, 0.1 mg), 50XC9024 (Muffle furane). The pressure container with the reactor sample delivered from the polymerization plant is carefully depressurized in a hood by respecting the safety regulations. The total content of the container will be emptied into the plastic beaker. The small crucible is filled half with the hot suspension while stirring continuously. After applying a vacuum the filter cake on the crucible is sucked dry for 5 minutes. Two Petri dishes are filled with the pre-dried powder to a height of 1 cm and dried for 15 minutes at 80 °C and 100 mbar in the vacuum dryer. When the melting time is over, the piston with a weight will be loaded. Then, pre-run time and the actual automatic measurement of piston travel starts until the lower ring mark has reached the upper edge of the cylinder. During the test period, at least 3 intermediate values for the piston travel at regular intervals is recorded. The test is finished when the upper ring mark has reached the upper edge of the cylinder or at least 25 minutes after the beginning of the melting time [16].

2. 3. Volatiles in HDPE This test is for the volatile concentration calculation (e.g. residual hexane or low boiling wax fractions) in HDPE powder or pellets by weighing. Equipment includes infrared or Halogen moisture analyzer with accessories and analytical balance (with accuracy of 0.1 mg). The analysis is performed on a representative sample at 105 °C until constant weight for 60 minutes. Sample amount should be 10.0 ± 0.1 g. For further information refer to the instruction manual of the instrument [16].

2. 4. Mechanical Tests Charpy Impact Strength was done at 23°C according to DIN EN ISO 179/1eA. Test specimen is made from a molded sheet of 4 mm and a diameter of 120 mm. The specimen is sawed to size and the notch is planned on it. The test is carried out on a pole of 80 mm x 10 mm x 4 mm, which is notched in the center and is tested in a lying position by a pendulum hammer impact on the narrow side opposite to the notch. The Charpy impact strength is the impact energy attained at the breaking of the test specimen.

Note the measured values for impact energy at break of test specimen and break type [16].

2. 5. Bulk Density of HDPE The bulk density is determined according to DIN EN ISO 60 by weighing out special calibrated beakers. Equipment's includes Bulk density measurement, laboratory balance (1,000 g, 0.1 g). Testing powder the empty beaker is weighed. The bulk density measurement consists of a 200 ml funnel and a 100 ml beaker. The funnel bottom is equipped with a flap from where the powder flows into the beaker. 110 – 120 ml of powder are gently transferred into the funnel and then the flap is opened. The accumulated heap of powder exceeding the beaker rim is levelled off with a cardboard at an angle of 45°. The filled beaker is weighed [16].

2. 6. Density of HDPE According to ISO 1183 the density is one of the two characteristic properties of

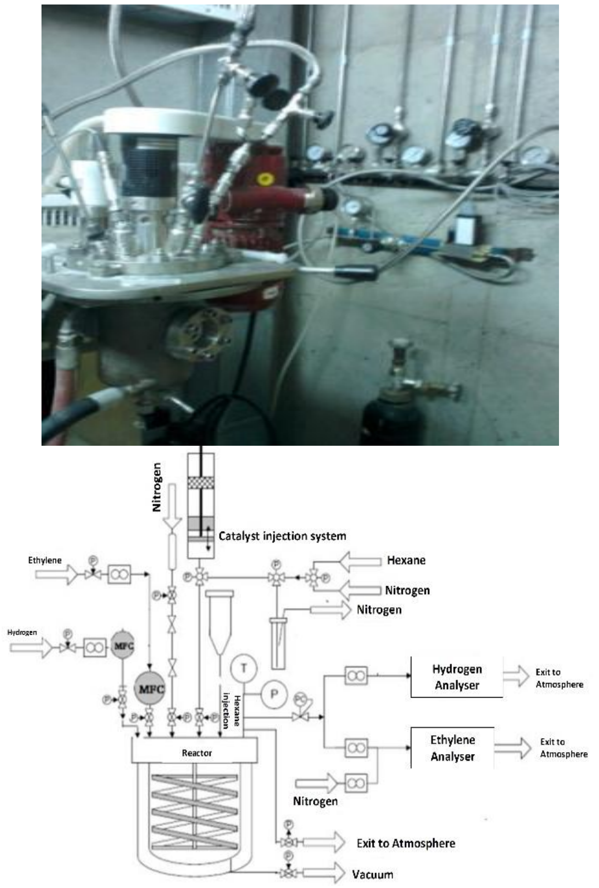

Figure 1. The experimental set up for copolymerization of ethylene/1-butylene and its scheme

polyethylene. The density results from the different buoyancies of the specimen in air and in butyl acetate.

2. 7. Polymerization Figure 1 shows the experimental equipment of ethylene slurry polymerization. The jacketed stainless steel reactor with a volume of 1 liter and Buchi model is used. The volume of the reactor jacket is 300 ml, the maximum pressure allowed on the reactor is 60 bar, for the jacket this amount is 20 bar and the reactor test pressure is 10 bar. The temperature range of the reactor and its jacket is from -10°C to 250°C. The setup has the ability to inject catalyst under nitrogen atmosphere by operator, and co-catalyst injection is also done from the same route. The catalyst injection system consists of a pipe with valves at both ends, one end of the pipe is connected to the reactor and the other end is connected to nitrogen. Injection is done by pressurizing the catalyst with nitrogen into the reactor. The reactor temperature is determined using a sensor (Keller druck messtechnik). Ethylene and hydrogen enter the reactor through the mass flow controller. 400 ml of dry hexane stored in a molecular sieve and a sodium strip are injected into the reactor at room temperature. Pressure and temperature are controlled throughout the polymerization. The reactor is equipped with a temperature jacket that keeps the polymerization temperature constant at the desired point. To keep the pressure constant during polymerization, a control system is used which enters the monomer into the reactor at the same rate of consumption. Prior to polymerization, the reactor is degassed by a stream of nitrogen gas. Degassing is repeated for 30 minutes and about 20 times. Finally, the reactor is saturated with ethylene gas. The mixing speed of the reactor was set at 2800 rpm. The flow of nitrogen, hydrogen and ethylene gas is free of any impurities by passing through a column consisting of molecular sieve. Antifouling, co-catalyst and catalyst injections are performed in the presence of ethylene pressure. In the copolymerization test, 4g of 1-butene co-monomer is injected into the container as a density controller after injecting other components. In all experiments, the amount of hydrogen is introduced into the polymerization container with a partial pressure of 4 bar as a chain transfer agent. The polymerization is kept constant at 80°C and ethylene is continuously fed into the reactor vessel. The ethylene pressure is kept constant by the continuous entry of ethylene monomer in the amount of 6.4 bar for one hour of the test. At the end of the reaction, the monomer flow is stopped and the polymerization temperature is brought to ambient temperature and the reaction is stopped [16].

3. RESULTS AND DISCUSSION

3. 1. Particle Size Distribution (PSD) Figures 2 and 3 show the particle size distribution for Finix-112 and SAC-150 catalysts, respectively. Table 1 summarized the comparison between particle size information for both catalysts. The average particle diameter is the same for both catalysts. However, the SAC-150 catalyst showed 10% of particles were smaller than 128 µm, which is smaller than commercial one. Coarse particles with a very small amount of fine particles showed a lower

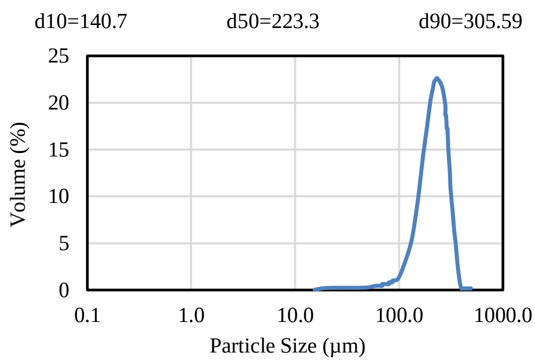

Figure 2. PSD for HDPE powder using Finix-112 catalyst

viscosity compared to the suspension containing course particles and suspension with fine particles containing some course particles. They did not show much changes compared to the suspension containing fine particles.

The presence of fine particles between large particles reduces the collision of large particles and fine particles act as a lubricant and facilitate the rotation of the particles and reduce the relative viscosity.

3. 2. Sieve Analysis for HDPE Powder by Super Active and Finix-112 Catalysts

Table 2 shows the molecular sieving results of the HDPE powder product from the two catalysts. Also, bulk density and HDPE powder density are presented in this table. The results show that due to the higher bulk density condition, the product powder of SAC-510 catalyst is better than the product powder of Finix-112 catalyst. The low density of powder super active catalyst indicates its higher strength than the product powder with Finix-112 catalyst.

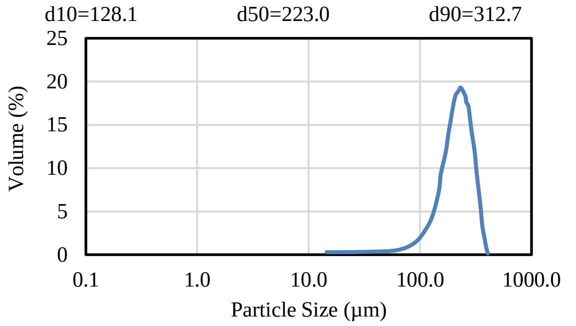

Figure 3. PSD for HDPE powder using SAC-150 catalyst

TABLE 1. Comparison of PSD

Catalyst	SAC-510	FINIX-112
Sample Date	25 October 2021	25 October 2021
d(0.1)	128.1	140.7
d(0.5)	223	223.3
d(0.9)	312.7	305.59
Fine<63µm	2.57	1.61
Fine<125µm	9.33	6.46

TABLE 2. Sieve Analysis results for HDPE powder

	SAC-510		Finix-112	
Sample	NO.1	NO.2	NO.1	NO.2
Bulk Density (g/cm^3)	0.43	0.43	0.4	0.4
Sieve (%<500)	99	98	96	95
Sieve (%<400)	96	96	92	94
Sieve (%<315)	93	92	87	90
Sieve (%<250)	79	78	73	72
Sieve (%<200)	57	57	51	49
Sieve (%<160)	32	27	19	22
Sieve (%<125)	16	15	6	18
Sieve (%<63)	2	2	0	0
Sieve(µd50)	181	184.7	201	194
Density (g/cm^3)	0.947	0.948	0.949	0.95

3. 3. Gel Permeation Chromatography (GPC)

Figure 4 shows the GPS diagram for both catalysts. The aim of the GPC test is to measure the molecular mass of the chains and to distribute the mean molecular weight of PE100. The nature of Ziegler-Natta catalyst slurry systems for the production of PE100 is such that the length of polymer chains is not the same [17].

When a GPC is taken from a product, its distribution graph is wide. This width is due to the nature of the catalyst as well as the bimodal property and the polymerization reaction in the two reactors. The purpose of GPC test was to evaluate the compatibility of PE100 product with SAC-510 catalyst with Finix-112 product, which shows good compatibility.

As can be seen in Table 3, the Mw/Mn parameter of the product with both catalysts is higher than 20, which indicates the wide molecular distribution and the parameter M_{z+1} and M_{z+2} of the product powder with catalyst SAC-510 is more than the product powder with catalyst Finix-112. This parameter indicates the number of long molecular chains and causes more polymer entanglement. However, the amount of co-monomer bulk in the parameter product powder with catalyst SAC-510 is less than Finix-112.

3. 4. MFI Changes in SAC-510 and Finix-112

The melt flow rate is mainly controlled by the amount of hydrogen in the reactor. To be more precisely, it is the ratio of the partial pressures of hydrogen and ethylene in the gaseous phase which - at a given partial pressure of ethylene-exactly determines the average molecular weight. The partial pressures of hydrogen and ethylene are measured by analyzing the concentrations in the gaseous phase of the reactor. The concentrations of H_2

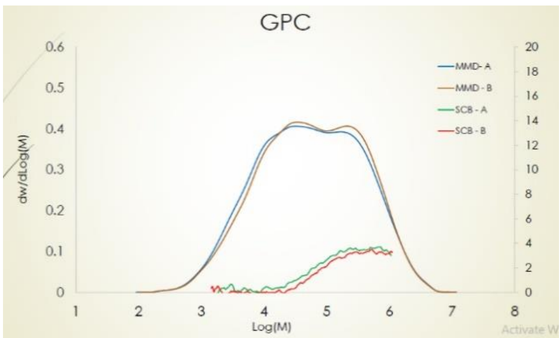

Figure 4. Comparison of GPC diagrams of PE100 product with both catalysts

TABLE 3. GPC results for HDPE-PE100 using SAC-510 and Finix-112 catalyst

HDPE Powder		
	A (Finix)	**B (SAC 150)**
Mw (g/mol)	233372	241203
Mn (g/mol)	10546	11430
Mw/Mn	22.13	21.1
Mz (g/mol)	1185062	1178261
Mz+1 (g/mol)	2245649	2363459
Mz+2 (g/mol)	3035200	3501861
Mv (g/mol)	168265	176333
Bulk CH_3/1000 C	4.317	3.764
Corrected Bulk CH_3/1000 C	1.7	1.3
Bulk comonomer wt%	0.66	0.53
Calculation method version	2	2

and C_2H_4 in the hexane diluent are proportional to the composition of the gaseous phase.

Keep in mind that MFI control via hydrogen feed will not work properly if other process data like temperature, gas feed, and activator concentration are fluctuating.

It is clear that by increasing the melting flow index (MFI) in the first reactor, the length of the monomer chain is short and, in other words, the optimal crystalline is formed in the first reactor and by decreasing MFI in the second reactor; the fabrication of product strength and desirable product is achieved. As seen in the Figures 5 and 6, in a 24 hour period in the first reactor for every two hours, the MFI test was taken with a weight of 1.2 kg and in the second reactor every two hours of MFI test was taken with 5kg weight. As can be seen in Figure 5 the amount of MFI in both reactors is logical match using SAC-510 catalyst than the Finix-112 and showed the quality, strength and density of new catalyst did not drop, and even in some cases, there was a relative superiority to Finix-112 catalyst.

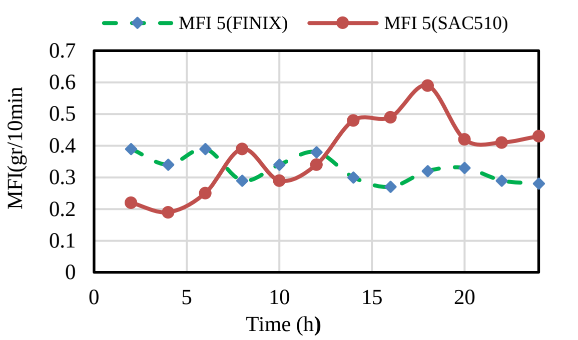

Figure 5. MFI of PE-100 in second reactor at 190°C and 5kg

According to Figure 6, it is observed that in the second reactor MFI with SAC-510 catalyst had a better performance than Finix-112 catalyst. This is indicating the optimal state of amorphous product using SAC-510 catalyst than Finix-112 catalyst.

3. 5. Comparison of the Effect of Catalyst on Process Hydrogen Responsibility

Hydrogen is injected into the first reactor to shorten the polymer chain to control the density and crystallinity of the product. Hydrogen as a valuable substance the less it is consumed, the better it is economically. The injection of excess hydrogen into the first reactor increases the pressure of the reactor and subsequently pours larger amounts of the gas mixture, which also contains hydrogen. Therefore, it is desirable that the H_2/C_2 ratio, i.e. the ratio of hydrogen to ethylene, be a minimum value while maintaining the desired density and properties of the product. According to Figure 7, it is clear that the product hydrogen responsibility with SAC-510 had a better performance and was able to produce the desired product with less hydrogen.

3. 6. Charpy Impact

Figure 8 shows the changes in charpy impact of pipe produced with SAC-510 catalyst and comparison with pipes produced with Finix-112 catalysts. The x- axis shows the time in terms of day and

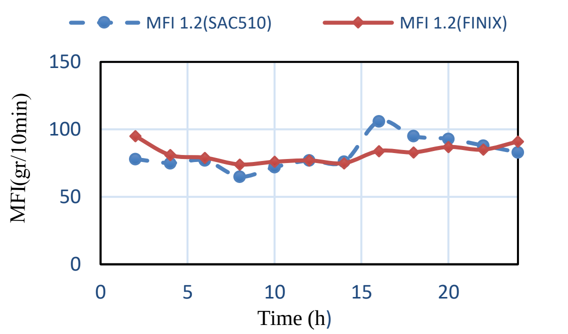

Figure 6. MFI of PE-100 in first reactor at 190C° and 1.2kg

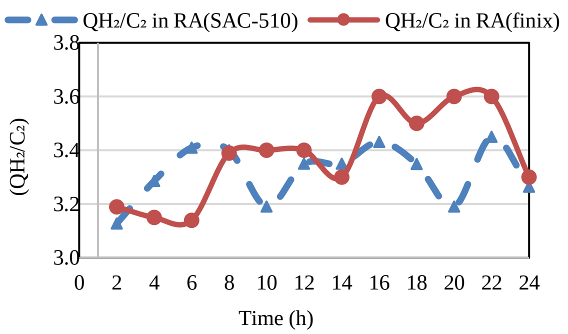

Figure 7. Comparison of product hydrogen responsibility SAC-510 catalyst with product with Finix-112 catalyst

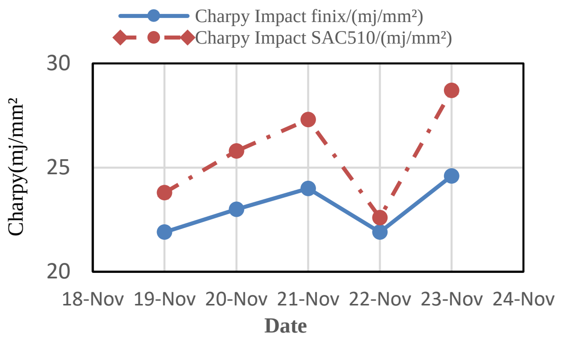

Figure 8. PE100 Charpy Impact using SAC-510 and Finix-112 catalysts

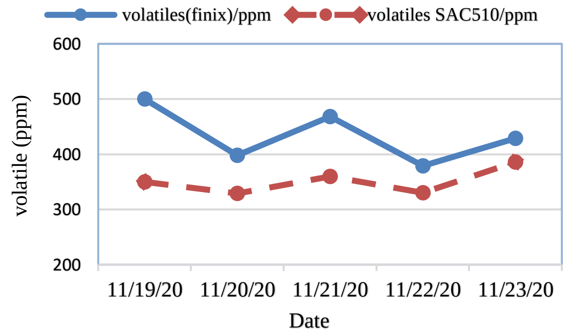

Figure 9. Comparison of volatile changes of PE-100 pipe grade using SAC-510 and Finix-112 catalyst

the y-axis charpy impact. The higher the, the stronger the pipe.

As shown in Figure 8, the average charpy impact in the diagram of a PE pipe grade100 made with a SAC-510 catalyst is greater than that of a pipe made with a Finix-112 catalyst. As a result, a pipe made with a SAC-510 catalyst has more strength and durability than a pipe made with a Finix-112 catalyst.

Charpy impact value obtained from the production of PE-100 with SAC-510 catalyst were also compared with the values of PE-100 produced in the most up-to-date articles [3].

Table 4 shows that the strength and durability of PE-100 with SAC-510 catalyst are greater than the strength of PE-100 mentioned in thisarticle.

3. 7. Comparison of Volatile of Produced using SAC-510 and Finix-112 Catalysts Figure 9 shows the volatile changes over time for the PE-100 product using the SAC-510 and Finix-112 catalysts. As it is proved in the previous section, the lower the volatile in the product, the better the quality of the catalyst used in the product. Therefore, according to Figure 9, the product using SAC-510 catalyst showed less volatile and this indicates better than SAC-510 catalyst.

Some effective mechanical parameters in PE-100 such as carbon black content, MFI and density for the product using SAC-510 and Finix-112 and all 3 catalysts are listed in Table 5.

TABLE 4. Charpy Impact for PE100 using SAC-510 and Finix-112 catalyst

Sample Code	Charpy (KJ/m²)
N-100J2^{a*}	28.12±0.8
B1-100J2^{a*}	26.17±1.3
B2-100J2^{a*}	27.03±0.6
PE100ARMCRP$_{finix}$	24.6±0.6
PE100ARMCRP$_{SAC510}$	28.7±0.8

a*: Refer to literature [3]

TABLE 5. Quantitative analysis of contaminants and residual fillers for various HDPE samples

Sample code	Density (g/cm³)	MFI1 (g/10min) 5kg, 190°C	Measured according MFI	MFI2 (g/10min) 2.16kg, 190°C
PE100$_{N-100j1}$^{a*}	0.952	0.22	ISO 1133:1997	6.9
PE100 $_{N-100j2}$^{a*}	0.95	0.18	ISO 1133:1997	6.2
PE100$_{N-100jM}$^{a*}	0.951	0.21	ISO 1133:1997	5.8
PE100ARMC RP$_{finix}$	0.949	0.24	ASTM D1238-ISO 1133	6.3
PE100ARMC RP$_{SAC510}$	0.947	0.17	ASTM D1238-ISO 1133	6.1

a*: Refer to literature [3]

The amount of carbon in the product with SAC-510 catalyst is less than the product used with Finix-112 catalyst, which is due to the better quality of SAC-510 catalyst compared to Finix-112 catalyst. But this amount is slightly higher than the other three products in the article.

4. CONCLUSIONS

The Ziegler-Natta spherical super active catalyst was prepared for the polymerization of the ethylene slurry phase in the presence of titanium chloride using magnesium ethoxide substrate and internal coal catalysts and halocarbon. Appropriate particle size distribution and specific surface area and spherical catalyst morphology observed in scanning electron microscopy have resulted in high catalyst activity. These results can be achieved by comparing the product of PE100 polyethylene pipe using two catalysts SAC-510 and Finix-112: Hydrogen responsibility has improved compared to catalyst Finix-112 Charpy impact PE100 with catalyst SAC is better than PE100 with catalyst Finix-112. Melt flow index values indicate that the powder and pellet conversion gap decreased in the product with SAC-510 catalyst and so we will have an easier process in production. Finally, due to the production of this valuable super active catalyst in Iran, we may not need to import expensive catalyst like Finix-112 and at a much lower cost than importing catalyst Finix-112. In fact, with the synthesized and experimented catalyst in this work, we have successfully achieved the production of HDPE PE100 pipe.

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Persian Abstract

چکیده

کاتالیست جدید بنام کاتالیست فوق فعال برای تولید لوله پلی اتیلن با دانسیته بالا گرید ۱۰۰ برای اولین بار در ایران ساخته و بومی سازی شد. هدف از ارائه این مقاله ارزشیابی عملکرد کاتالیست جدید در مقایسه با کاتالیست Finix-112 که کاتالیستی متداول در تولید گرید لوله فوق الذکر است. با استفاده از آزمایشات متعدد بر روی خواص فیزیکی و شیمیایی محصول، عملکرد هر دو کاتالیست شامل توزیع اندازه ذرات، آنالیز پراکندگی اشعه ایکس، میکروسکوب اسکن الکترونی، فراریت، دانسیته، دانسیته بالک و ضربه چارپی ارزیابی گردید. نتایج نشان می‌دهد که عملکرد پاسخ‌پذیری هیدروژن محصول با کاتالیست جدید ۱۹، ضربه چارپی ۱۶.۷ و فشار عملیاتی در راکتور اول با کاتالیست جدید ۲۸ درصد بهبود یافته است. کاتالیست ساخت داخل می تواند جایگزین خوبی برای کاتالیست وارداتی finix-112 باشد و صرفه جویی ارزی قابل ملاحظه‌ای به ارمغان آورده است.