Antielectrostatic Agent Addition in Low-Dielectric-Constant Polymerization Media

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ABSTRACT: In the coordinative polymerization of α-olefin by a slurry process, a low-dielectric-constant suspending agent, such as hexane or heptane, must be used. As a result of solvent movement and its friction between system components, electrostatic charges are generated. Because of low electrical conduction of these solvents, the generated charges accumulate in the polymerization medium. Consequently, a repulsive force between same charges effects the growth of the polymer particles and causes them to form fine particles. In this article, we present research results on the effects of antielectrostatic agents on the increase of the electrical conduction of the polymerization medium, particle size distribution, and also the quantity of fine particles in the final polymer. Techniques gained from the fuel industry were applied to modify the subjected polymerization medium. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 125: 1979–1985, 2012

Key words: dielectric properties; emulsion polymerization; particle size distribution; polyolefins

INTRODUCTION

The application of Ziegler–Natta catalysts, based on transition metals, for the polymerization of olefins has been developed over the last 5 decades. Wide application of these catalysts and their polymerization have been reported in several articles. In 1998, the world production of high-density polyethylene (HDPE) and LLDPE amounted to 25.6 million tons, of which over 57% was from slurry processes. Most commercial slurry processes are, however, basically dedicated to HDPE production, with only a small amount dedicated to the production of LLDPE.[1–6]

In the slurry process of production polyolefins, one of the most important criteria is the selection of the proper suspending agent. The polymer should be insoluble to facilitate handling and purification after the final step. It also should be chemically inert to prevent interaction or bonding with reaction components, especially the catalyst. An ease of separation and the fair solubility of the monomer in it are two other main items for this choice. Among the most commonly used organics in industry are hexane, cyclohexane, and heptane. These kinds of fluids have low polarity because of their molecular structures. In a semibatch reactor, a mixer should work continuously during polymerization to supply enough and a steady monomer concentration and prevent particle deposition and mass transfer limit to and within the catalyst particle. The minimum mixer speed is the lowest rate to suspend particles over the reaction time. Mechanical grinding of the polymer particles occurs as the result of high rates.

Because of friction between fluid and other ingredients, particularly polymer particles, electrostatic charges are generated by the continuous working of the mixer in the polymerization medium. Charge generation occurs in the liquid on the molecular level at the interface of any two unlike materials. This phenomenon is called *triboelectrification*.[7,8] The rate of electrostatic charge generation increases with increasing speed of the mixer. These electrostatic charges tend to repel and dissipate toward lower potential according the phenomenon known as *charge relaxation*. The reduction in charge is related to the time elapsed according to eq. (A.1). If the generation rate is greater than the repelling rate, accumulation occurs. As mentioned before, these kind of fluids used as suspending agents have low polarity and dielectric constants; therefore, the low conductivity is the main reason that causes a delay in the movement and expelling of generated electrostatic charges to outside the medium. Consequently, a short time passes just after running charges accumulate in the polymerization reactor. In Table I, some liquids with different polarities and conduction are compared.[9,10]

Effect of electrostatic charges on the particle size distribution (PSD) and fine quantity

The Ziegler–Natta type polymerization with the application of a slurry technique has some negative

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	Polarity	Dielectric constant	Conductivity (pS/m)
Cyclohexane	0.006	2.00	0.005
Hexane	0.009	1.88	0.01
Toluene	0.099	2.38	1
Diethyl ether	0.117	4.34	40
Ethyl acetate	0.228	6.02	100,000
Acetone	0.355	20.70	6,000,000
Ethanol	0.654	24.55	15,000,000

points, despite its several advantages. One of the encountered problems in industrial polymerization is the formation of agglomerates. Agglomerates form in various places, such as the polymerization reactor and the lines of the recycling gas stream or other pipes. Such agglomerates usually have many adverse effects on polymerization. Because of agglomerate formation (fouling), it may be necessary to shut down the reactor. It has been found that agglomerates may be formed as a result of the presence of fine polymer particles in the polymerization medium. In the polyolefin industry, *fine particles* are defined as polymer particles with values of less than 125 μ in diameter. These fine particles are believed to deposit onto and electrostatically adhere to the inner walls of the polymerization reactor and the associated equipment for recycling the gaseous stream, such as heat exchanger. When these agglomerates form within the polymerization reactor, they tend to be in the form of sheets.[11,12]

Electrostatic charges are the most given reason for fine particle generation in such polymerization areas. The polymer chains grow about the catalyst from ethylene insertion at the active sites mainly within the polymer/catalyst mass. As the result of the surface tension, the polymer particles incline toward a lower energy by uniting to form larger particles and a smaller total surface (Fig. 1).

However, as mentioned before, the triboelectric effect and, subsequently, electrostatic charge generation and accumulation, which mostly arise at the boundary of the outer surface of polymer powders and suspending agents, are in contrary opposition to the surface tension because of the repulsion between same charges. According to polyethylene's (PE's) nature, it is proven these charges are negative. These fine particles come into existence by fine catalyst fragments, but the accumulation of electrostatic charges in the reaction medium is the predominant reason. This is confirmed by analysis of molecular weight distribution of the synthesized polymer, and its fine particle portion does not show a significant differences, although it is known that fine catalyst particles produce higher molecular weight macromo-

lecules. The accumulation of electrostatic charges and a slight discharge rate of fluid issued by the low electric conduction of the suspending agent and natural repulsion against surface tension cause fine particle generation.

Several ways for reducing or eliminating electrostatic charge have been suggested; these include (1) installation of grounding devices in the reactor, (2) ionization of the feed gas or particles by electrical discharge to produce ions that neutralize the electrostatic charge on the particles, and (3) use of radioactive sources to produce radiation capable of producing ions that neutralize the electrostatic charge.[13]

Static electricity is one of the most insidious sources of fire and explosions in modern industry.[14,15] Numerous fires and explosions have been caused by static spark ignitions from charges generated in hydrocarbons during discharging.[16] These low-dielectric-constant fuels are so susceptible to explosion because of flammable vapors, which often have a low flashpoint (liquids with a flashpoint at atmospheric pressure between 32 and 55°C are classed as flammable; liquids with a flashpoint below 32°C are classed as highly flammable[17]). Electrostatic charge generation is not avoidable, but the best way is to prevent the accumulation of charges in the medium by an increase in the conduction by the addition of antistatic additive (ASA).

ASAs are some chemicals that are able to increase the conduction in low concentration. Many chemical compounds have been used as ASAs, including olefin–acrylonitrile copolymers, acrylonitrile copolymers/polyamines, olefin–sulfur dioxide copolymers, polyamines alkylphenol–formaldehyde condensates, and vinyl ether–maleic anhydride copolymers.[18] The use of antistatic materials in hydrocarbons as an additive to fuels is common. Some common commercial chemicals used as ASAs are shown in Figure 2.

Through a more deep deliberation of the chemical structure of these materials, it may be understood that resonance existence plays the main role in smoothing the progress of charge transfer. These
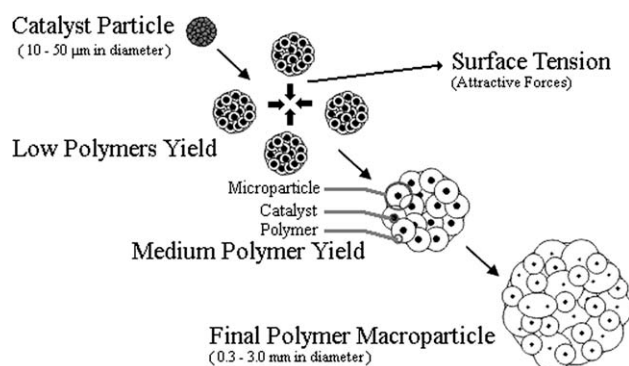
Figure 1 The development of polymer particle growth during polymerization.
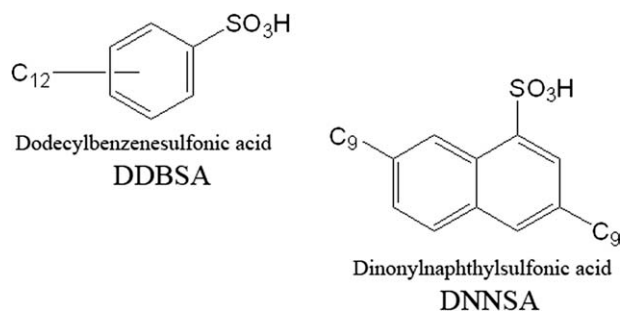
Figure 2 Chemical structure of two common ASA.

ASAs do not have the same efficiency in different media, and some things, such as the viscosity of the liquid and boundary layer effects, can change the functionality considerably.[19]

Ziegler–Natta type catalysts are sensitive to compounds that have oxygen and nitrogen in their molecular configuration.[20,21] It is well known that external donor strongly affects the polymerization.[22,23] As observed, all of these ASAs have a lone pair of electrons in their structure that can easily interact as a Lewis base with Ziegler–Natta catalysts like an external electron donor to influence some essential polymer specifications, including catalyst activity, molecular weight distribution, hydrogen response, melt flow index (MFI), yield, and isotacticity (polyolefins other than PE).

Because the main purpose of using ASA is to decrease the amount of fine particles without any change in the final product properties and with regard to the high sensitivity of these kinds of polymerization, the most suitable compounds as ASAs should be soluble in a suspending agent at first and also have enough power to increase the conductivity with a very low concentration to reduce or remove the probability of undesirable interaction with catalyst and other components because of a negligible content of ASA in the reaction medium.

In this study, sodium dioctyl sulfosuccinate (SDOSS) was used as the ASA. SDOSS's advantage compared to other common ASAs [e.g., dodecyl benzene sulfonic acid (DDBSA) or dinonylnaphthalene sulfonic acid (DNNSA)] is the presence of 2-ethylhexyl group in both sides of the molecule. These alkyl groups cover electron pairs at the center of the ASA molecule and

make them hard to access by the vacant site of titanium atom, which may influence insertion of the C=C bonds into the Ti—C bond at the active center.

EXPERIMENTAL

Materials

Ethylene and nitrogen (purity > 99.99%) were passed through columns packed with moisture and oxygen scavengers, respectively. Hexane (Merck, Darmstadt, Germany) was dehydrated by refluxing through a distillation column with sodium as a dehydrating agent under dry nitrogen. Then, hexane was dried over molecular sieves of 0.4 and 0.5 nm (same portion) for 24 h to reach less than 5 ppm. SDOSS (Sigma-Aldrich, St. Louis, Missouri) was purified three times by a recrystallization technique in dried hexane. Other reagents were used without purification [hydrogen (>99.9%) and triethylaluminum (TEAL; Fluka, Milwaukee, Wisconsin)]

Catalyst

An industrial heterogeneous Ziegler–Natta catalyst [Mg(Cl)$_2$-supported TiCl$_4$, third generation with the formula MgTi$_{0.135}$Cl$_{2.3}$, no internal or external electron donor, total Ti = 40 mmol/L and Ti^{+3} = 32 mmol/L] were supplied from Arak Petrochemical Co. (Arak, Iran).

Polymerization

Ethylene polymerization was carried out in a 1.1-L semibatch steel jacket Büchi autoclave reactor (Uster, Switzerland) fitted with a thermocouple equipped with a mechanical seal stirrer (variable speed, anchor form) in the slurry phase. The most common mixing speed for these kinds of reactions is around 500–600 rpm, but to prevent mechanical grinding of polymer particles and better observation of ASA effects, the mixing speed was set down at 250 rpm. After all of moisture and air were run out by hot nitrogen purging and a cooling reactor was used to reduce the inner temperature to near 50°C (to prevent evaporation of the suspending agent), 500 mL of hexane was added. TEAL (2 mL; 4% v/v in

TABLE II
Reaction Conditions

Experiment number	Duration	Temperature	Cocatalyst	Total Pressure	Al/Ti
1	60 min	80°C	0.29 mmol	8 bars	24
H$_2$O in hexane	Ti residue in PE	Al residue in PE	Hexane vapor pressure at 80°C	Hydrogen partial pressure	Ethylene partial pressure
5 ppm	3 ppm	10 ppm	1069 mmHg[a]	0.25	0.75

[a] *CRC Handbook of Chemistry and Physic, 83rd ed.* Lide, D.R., Ed.; CRC Press: Boca Raton, FL, 2002; p. 6–66.

TABLE III
The Details of SDOSS Addition for Each Batches

Experiment number	Concentration of applied SDOSS (ppm)	Applied SDOSS (mL)	Volume of dried hexane	Final concentration of SDOSS in the reaction medium	Final volume
1	—	0	497.7	0	500[a]
2	100	5	492.7	1	500[a]
3	100	50	447.7	10	500[a]
4	10,000	5	492.7	100	500[a]
5	10,000	50	447.7	1000	500[a]

[a] For all batches, 2.0 mL of cocatalyst and 0.3 mL of catalyst were applied. Also, temperature effects on the hexane volume were ignored.

hexane containing 0.29 mmol of Al) was added, and the mixer was turned on to eliminate any probable contaminations for 5 min, and then, 0.3 mL of catalyst was added with a glass syringe. The catalyst injection valve was closed, and then, the reactor was warmed up to 80°C and loaded with hydrogen to 2 bars. Then, the hydrogen inlet was closed. Ethylene was supplied continuously at 8 bars for 60 min with a Büchi pressure-flow gas controller, and the reaction temperature remained stable at 80°C through the circulation of warm water through an outer jacket surrounding the reactor controlled precisely by a sensitive thermostat. Polymerization was terminated by the injection of ethanol while the mixer was working after the ethylene supply was switched off and the reactor was depressurized to atmosphere pressure. The reactor cooled down to below 50°C within 15 min. Other details about the polymerization condition (experiment 1) are shown in Table II.

After the production of HDPE without any ASA addition, SDOSS was added to the reactor in different concentrations after catalyst injection [order of material addition: (1) hexane, (2) TEAL, (3) catalyst, (4) ASA, (5) hydrogen, and (6) ethylene]. To increase the dosage effectiveness, purified and dried SDOSS was prepared at a concentration of 10,000 ppm; then, a diluted solution (100 ppm) was made from the concentrated solution. To ensure that the final volume of 500 mL in the reactor remained constant and all of the experiments had the same conditions as experiment 1, ASA was introduced to the reaction medium according to Table III (experiments 2, 3, 4, and 5).

Characterization

The total obtained polymer was fully gathered, then washed with ethanol, and dried *in vacuo* at 70°C to a constant weight to calculate the total yield. The moisture content in hexane was quantified by the Karl–Fischer method with a coulometeric instrument (Mettler–Toledo titrator DL39, Zurich, Switzerland).[24] The electric conduction of the reaction medium was measured with a precision meter (Emcee 1154, Venice, Florida).[25] The bulk density data were obtained according ASTM D 1895-96.[26] The melting point and degree of crystallinity were based on the differential scanning calorimetry method (Mettler–Toledo Star[e] system DSC 822).[27] MFI is reported according an extrusion plastometer technique (at 190°C and 2.16 kg load, Gotech GT-7200-MI, Taichung, Taiwan).[28] PSD was calculated according to ASTM E 2651.[29] The amount of titanium in the prepared catalyst and other minerals (Al and Ti) in the final polymer were determined by atomic emission spectroscopy Inductively coupled plasma atomic emission spectroscopy (ICP-AES) (Varian 730-ES, Melbourne, Australia). A microwave digesting process was applied to decomposition (Anton Paar Multiwave 3000, Graz, Austria).[30]

RESULTS AND DISCUSSION

Electric conductivity was determined for all batches after the addition of the reaction components and ASA at 25°C. It was recognized that the electrical

TABLE IV
Electric Conductivity (pS/m) for Different Batches at Different Temperatures

Temperature	SDOSS (ppm)				
	0	1	10	100	1000
25°C (298 K)	0.07[a]	0.62[a]	4.1	51.2	584
35°C (308 K)	0.12[a]	0.90[a]	6.1	74.2	701
45°C (318 K)	0.20[a]	1.7	9.5	86.3	891

[a] Alternating-current measurement was applied. For the rest, the direct-current method was used for sensing.

TABLE V
Estimated Values of the Electrical Conductivity at 80°C

SDOSS (ppm)	Z	Ln A	R^2 [a]	Ln κ at 80°C	κ at 80°C
0	−4951	14.0	1.0000	−0.063	0.94
1	−4877	15.8	0.9667	2.027	7.6
10	−3953	14.7	0.9971	3.474	32.3
100	−2488	12.3	0.9522	5.271	195
1000	−2002	13.1	0.9909	7.406	1646

[a] Ln κ = Ln A − Z/T. R^2, coefficient of determination.

TABLE VI
Total Yield of Polymerization and Fine Particle Percentage at Different Concentrations of SDOSS

SDOSS (ppm)	0	1	10	100	1000
Yield (g)	95	101	98	92	74
Fine particles (%)	11.4	11.2	10.5	5.2	5.8

TABLE VII
PSD (%)

		<125	<250	<355	<500	<710	<1000
SDOSS (ppm)	0	11.4	46.9	25.8	7.4	4.4	2.9
	1	11.2	45.3	26.3	7.6	4.9	2.6
	10	10.5	46.8	26.2	7.6	4.3	2.8
	100	5.2	49.4	28.3	7.4	4.7	3.1
	1000	5.8	49.2	28.1	7.3	4.8	2.7

(Sieve size (μ))

conductivity varied significantly with temperature, and the relation differed for the various types of media.[31,32] Therefore, it was necessary to correct the measured values of conductivity to a particular temperature. With reference to several previous studies of nonconductive liquids, the conductivity was roughly independent of pressure and temperature below about 20°C but increased according to an Arrhenius function at higher temperatures.[33] An ASA is supposed to act in the reaction medium, and conductivity at the reaction temperature should be considered, but the polymerization temperature is supposed to be set at 80°C, and this made it impossible to measure the conductivity of hexane outside the reactor by a conductivity meter at this temperature because of the low boiling point at atmospheric pressure (69°C). To extrapolate the conductivity at this temperature, an alternative expression of the Arrhenius equation was used by the application of different temperatures to each sample (Table IV).

The conductivity of hexane was measured after the addition of cocatalyst, catalyst, and ASA (except for blank) at three different temperatures (25, 35, and 45°C) to calculate the estimated value at 80°C from the obtained plot with the linear regression technique [eq. (4), Appendix B]. Table V shows the estimated conductivity values at the polymerization temperature for each batch. The electrical conduction increased linearly with increasing ASA concentration at 25°C like in dilute solutions of inorganic electrolytes, but it increased with higher rates at elevated temperatures. This indicated that the temperature was very effective in ASA efficiency and should be fully analyzed for any different kinds of ASAs at many concentrations. It would be more preferable to design and set up an interior electrode to get the online conductivity of the reaction medium like a temperature or pressure sensor.

It can be seen from the data that fine particles were reduced considerably at concentrations near and higher than 100 ppm. The experimental results show that at low concentrations of ASA, the total yield was not influenced but reduced at a high concentration (Table VI). This fall in polymer yield was due to the competition of ethylene and ASA to interact with the transition metal; hence, at a higher concentration, it had enough power to act in response as an external donor.

Particle size analysis showed that the fine particle amount diminished when the electric conduction increased as a result of an increase in the SDOSS concentration in hexane (Tables VI and VII). The experimental data collected from physical properties, including the density, melting point, MFI, and percentage crystallinity, showed that no significant change occurred (Table VIII). These results demonstrate that each antistatic agent had an optimum range of application. In these series of experiments, around 100 ppm SDOSS was acquired as optimal because a maximum decrease in fine particles was obtained without a remarkable reduction in the polymer yield.

The bulk density of polymer powder was determined, and a small reduction was observed at higher concentrations of ASA (Table IX). This change could be interpreted in two ways: (1) the effect of ASA as an external donor on the polymer morphology or (2) the reduction of fin particle content and change in PSD.

Thus, this is worth it to emphasize that the fundamental properties were found to have no significant influence on the final polymer by ASA addition, especially the density and MFI (two main factors from an industrial point of view), despite the major decrease in fine particle percentage. Almost all of the proposed ASAs could interact as external electron donors in effective concentrations and could, thus, bring about wide changes in the product specification. However, the application of a very low and

TABLE VIII
Physical Results

SDOSS (ppm)	0	1	10	100	1000
Density (g/cm^3)	0.954	0.956	0.958	0.952	0.952
Melting point (°C)	134.2	134.3	134.0	134.2	134.4
MFI	0.64	0.65	0.64	0.63	0.64
Crystallinity (%)	70	71	69	70	72

TABLE IX
Bulk Density of the Polymer Powder

SDOSS (ppm)	0	1	10	100	1000
Bulk Density (g/cm³)	0.33	0.33	0.32	0.31	0.30

effective concentration of ASA could be a benefit in an industrial process without any changes in the product specifications.

CONCLUSIONS

In slurry polymerization, one essential factor that must be seriously considered is the electric conduction of the medium. In low-dielectric-constant polymerization media, electrostatic charge generation and accumulation occurs, and it influences PSD and increased the amount of fine particles. It can be abated with the use of ASA agents to enhance the electrical conduction and accelerate repelling charges. The best materials for the coordinated polymerization of α-olefins via Ziegler–Natta catalysts are high-molecular-weight compounds, especially those that have a steric hindrance around functional groups having a lone pair of electrons, which have no ability to interact with transition metals and influence the nature of the slurry polymerization. Each ASA has an optimum range of operation and should be used at the lowest effective concentration to prevent any effects on the chemical and engineering properties of the final product. In this research work, a concentration of 100 ppm SDOSS was found to be optimum in the polymerization medium. It may preferable for an industrial unit to devise an online monitoring and programmed system for these kinds of polymerization media to inject sufficient ASA automatically according to electrical conduction.

APPENDIX A[34]

Mathematic relations

For liquids with a conductivity greater than 1 pS/m, charge relaxation proceeds by exponential or ohmic decay. This so-called ohmic theory of charge relaxation has been experimentally confirmed for this category of hydrocarbon liquids, and exponential charge relaxation is described by the following equation:

$$Q = Q_0 e^{\frac{-t\sigma}{\varepsilon}} \tag{A.1}$$

where Q is the charge density (C/m³), Q_0 is the initial charge density (C/m³), e is the base of Naperian natural logarithms (ca. 2.718), t is the time (s), ε is the dielectric permittivity (F/m), and σ is the liquid conductivity (S/m).

Hence, for liquids that follow ohmic relaxation, the relaxation rate depends strongly on the conductivity. The lower the conductivity is, the slower the relaxation is. The ratio of dielectric permittivity to the liquid conductivity is referred to as the *relaxation time constant*. The relaxation time constant is the time for a charge to dissipate to e^{-1} (ca. 37%) of the original value if the charge relaxation follows exponential decay. It gives an indication of the electrostatic accumulation relaxation time constant of typical liquids.

Liquids with a conductivity of less than 1 pS/m do not, in practice, relax charges as slowly as ohmic relaxation would suggest. When such liquids are highly charged, the usual relationship described by Ohm's law does not apply; instead, for these liquids, charge relaxation proceeds by hyperbolic decay. The hyperbolic theory of charge relaxation has been experimentally confirmed for low-conductivity hydrocarbon liquids, both in small-scale laboratory experiments and in full-scale tests. Hyperbolic charge relaxation is described by the following equation:

$$Q = \frac{Q_0}{\left(1 + \mu Q_0^{\frac{t}{\varepsilon}}\right)} \tag{A.2}$$

where μ is the ion mobility (m² V⁻¹ s⁻¹).

For low-conductivity liquids, charge relaxation is dependent only on the initial charge density and ion mobility. The conductivity of the uncharged liquid is not a factor. In addition, charge decay is not very sensitive to initial charge density when the initial charge density is greater than about 100 μC/m³.

APPENDIX B[35]

The Arrhenius-type behavior of hydrocarbon conduction can be expressed as follows:

$$\kappa = A \exp(-E_a/RT) \tag{B.1}$$

where κ is conductivity, A is constant or pre-exponential factor, E_a is Arrhenius activation energy, R is molar gas constant, and T is temperature (K).

This alternatively can be written as

$$\text{Ln}\,\kappa = \text{Ln}\,A - Z/T \tag{B.2}$$

where $Z = E_a/R$, which is calculated from a plot of the natural log of conductivity against $1/T$.

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