A Comparison between Chemical and Physical Process for LDPE Foams

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Abstract. Foams are created by first dissolving and mixing a gas in the molten polymer, secondly expanding the gas into a lot of small bubbles or cells and finally cooling the expanded polymer and thereby creating the final foam. The foam can be shaped into different semi-finished products such as tubes, profiles, sheets and blocks; expansion results in a substantial reduction of polymer density. Polyethylene (PE), as raw material for the foam, has typically a specific density of 920 kg/m³ and density for PE foams are able to show a weight reduction, expanding the PE approximately 30 times. But not only the weight is changed during the foam process; new properties gained by the PE in its foamed state are responsible for many applications of this product. Two major foaming methodologies are employed for PE foam production: reactive foaming, where final foam products are yielded from a chemical reaction that initially involves a certain amount of gases, from the decomposition of a chemical blowing agent (CBA), and soluble foaming, that involves the dissolution of a physical blowing agent (PBA), in the polymeric melt. As CBA it was used Azodicarbonamide, that evolves 220 cm³/g of gas and leaves a white residue, odorless, nontoxic and nondiscoloring and as PBA, carbon dioxide, an eco-friendly gas, that presents an acceptable solubility and diffusity in polymers. This work aimed to a comparative study between chemical and physical blowing process. From a LDPE exhibiting 2.6 g/10 min Melt Index, samples were gamma-irradiated within a 0 to 30 kGy range and further extruded in a Rheomex 332p, single special screw for foaming, 3:1 L/D and 19/33 compression ratio, under carbon dioxide for soluble foaming. Admixtures containing 2.5, 5.0 and 10.0% of azodicarbonamide (ADCA) were too extruded in the same extruder, keeping the same temperature profile for providing reactive foaming. Cells morphology for foams obtained from both chemical and physical process was investigated by SEM analyses. Other characterizations comprised DSC, TGA, Gel-Fraction, Swelling-ratio and Rheological properties, as Melt Strength and Melt Flow Index.

Introduction

Low-density polyethylene (LDPE) is a commodity polymer used extensively in extrusion operations such as coating, blown film, blow molding, and foaming.

Extrusion of LDPE foams by direct gas injection or so-called Physical Foaming can be separated into 5 distinctive steps, i.e.: melting of the solid LDPE pellets, injection and mixing of the liquid gas in the molten LDPE, cooling and shaping of the melt into the expansion condition, foaming of the melt by the expanding gas and finally cooling of the foam.

Although viscosity and melting/crystallization behavior plays an important role in each or most of these process steps, the gas expansion is the crucial step in the foam process.

In order to obtain a good foam quality (regular fine cell size and high closed cell content), the gas laden melt needs to be cooled down to a temperature close to the crystallization temperature of the semi-crystalline polyolefin to increase the melt viscosity and reduce the time needed for the transition from melt to solid phase. In practice, this means for semi-crystalline LDPE that the temperature of the melt at the die exit is

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always a few degrees centigrade above the crystallization temperature.

When a physical blowing agent is used, such CO_2, an environmentally friendly blowing agent [1], the neat polymer is initially maintained above the melting temperature. Under these conditions, the polymer/blowing agent remains a single phase. As the melt mixture passes through the exit/shaping die the melt rapidly foams and expands, generating foams with small, uniform cell sizes [2]. The rheology of mixtures of a polymeric matrix and a physical foaming agent (PFA), used for the production of polymeric foams, is largely controlled by the plasticizing effect of the dissolved gas or liquid [3].

For the processing and foaming part of the choice it is good to know that most of the viscosity related properties depend on MFI (melt Flow Index) and MWD (Molecular Weight Distribution). An important parameter is the friction heat generation in the melt, which depends on LDPE shear viscosity character and the shear rate in each particular part of the extrusion process. To reduce the friction heat generation, extruders for Physical Foaming always should be run within relatively low screw speeds (10 – 50 rpm).

Long-chain branched LDPE usually has "extensional hardening" characteristics. There is a great deal of long-chains attached to the bone chain of branched LDPE, thereby inducing strong chain entanglement in melt state and enhancing the melt strength, that can improve foamability in foaming applications [4]. The term melt strength can be simply defined as the ultimate force that the polymer melt can withstand while being stretched. In some processes like melt phase thermoforming, the polymer sheet is required to exhibit high melt strength at low shear rates as the sheet is being heated: sag resistance [5][6].

Modification of a polymer using ionizing radiation can be done before or after the polymer has been processed into formed parts. When formed parts made from polymers are modified by radiation, crosslinking is usually the desired reaction, with the purpose to enhance the physical properties of the parts. For radiation modification of polymer resins prior to processing into formed parts, various radiation-induced reactions, including crosslinking, degradation, branching

and grafting, can be taken advantage to add value to the polymer material [7], [8].

In the late 1980s and early 1990s, Himont (which later became Montell) developed ''gel-free'' high melt strength polypropylene resins (PPs) for extrusion coating by radiation-induced long-chain branching [9] [10] reported that the improvement of the melt strength enabled foaming using these novel high melt strength PPs [11]. A similar approach was applied to polyethylene (PE) by Montell to make high melt strength PE in the 1990s [5].

By irradiating PE resins in air at relatively low doses in combination with additional proprietary processing, resins with significant long-chain branching but with insignificant gel content (< 3%) were made and used in various polymer processing methods and applications. The modified PE resins offer significantly improved processibility and end-use properties [11], [12], [13]. High levels of cross-linking would drastically decrease the melt flow of the polymers and high gel contents would make it very difficult or impossible to process the polymers and convert them into parts. The processibility of the polymers is maintained with the low dose modification while significant improvements of practical properties are achieved [14] [15].

When a PBA is used, it dissolves into the polymer melt at a high pressure. In contrast, when a CBA is used, it releases gas at a decomposition temperature. Compared to PBAs, CBAs are easier to use, and in the very low pressure or pressure-free foaming processes, only CBAs can be implemented. CBA procedure utilises a solid residue that incorporates into a matrix. The temperature of the matrix is raised so that the CBA may start to break up and release a gas. This gas is used to blow the plastic which now has a low enough viscosity that bubbles may form and a foam may be produced. Smaller cell size arises from a higher viscosity and the CBA degradation temperature and a stronger polymer melt strength. Cell structure, and especially size of cells is also affected by pressure. A high external pressure on the foaming system will produce a foam with a small cell size. The properties of the foam depend on the structure of the cells and the amount of vapour they contain per volume, as this affects the density and the thermal insulation properties of

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the product [16]. Azodicarbonamide (ADCA) was chosen as the blowing agent because of its relatively high gas yield (230 cc/g of nitrogen, carbon monoxide and carbon dioxide) and low cost [24]. For the chernical blowing agent, it is necessary to consider whether the reaction is exothermic or endothermic. These factors will affect the rate of solidification [18]. In case of ADCA, its decomposition reaction is exothermic [19].

Since blowing agents (CBA) release gases during heating, TGA is able to measure decomposition temperature ranges in a sample and weight loss in function of temperature.

Swelling measurements are used to predict degree of crosslinking in polymer networks [20]. Since crosslinking cannot be seen directly, it must be measured indirectly, by using swelling test; so, the degree of crosslinking should be inversely proportional to the degree of swelling [21].

Experimental

2.1 Materials

Commercially available polyethylene grade exhibiting 2.6 g/10 min Melt Index and supplied by Quattor was selected as base resin for radiation modification. The base resin was gamma irradiated at 10, 15, and 30 kGy doses, ^{60}Co, in air, at a 5 kGy/h rate.

Azodicarbonamide (ADCA) used in 2.5, 5.0 and 10.0 % formulations from 2,6 g/10 min melt index LDPE was supplied by Wenda do Brasil Ltda.

2.2 Premixing procedures

Polymer pellets and the foaming agent (2.5; 5.0 and 10.0 %) were dry-mixed in a Rheomex 332p, single screw, 3:1 L/D and 19/33 compression ratio, at 20 rpm, according to conditions presented in Table 1:

Table 1. Experimental conditions for premixing LDPE and 2.5; 5.0 and 10.0 wt% Chemical Foaming Agent (ADCA):

Screw speed (rpm)	Temperature (^{o} C)			
	Zone 1	Zone 2	Zone 3-4	Die
10	150	155	165	170

2.3 Foaming extrusion

2.3.1 PBA process:

Irradiated LDPE samples (10 to 30 kGy range) were extruded in a Rheomex 332p, single screw special for foaming, 3:1 L/D and 19/33 compression ratio, at 20 rpm, using carbon dioxide as blowing agent, under 20 bar pressure, according to conditions presented in Table 2:

Table 2. Experimental conditions for foam processability of irradiated LDPE samples (10 to 30 kGy range):

Screw speed (rpm)	Temperature (^{o} C)			
	Zone 1	Zone 2	Zone 3-4	Die
20	175	200	210	200

2.3.2.CBA process

LDPE samples (2.5; 5.0 and 10.0 % ADCA) previously prepared and homogenized in extruder was extruded in a Rheomex 332p, single screw special for foaming, 3:1 L/D and 19/33 compression ratio, according to conditions listed in Table 3:

Table 3. Experimental conditions for foam processability of LDPE samples comprising Chemical Blowing Agent (ADCA):

Screw speed (rpm)	Temperature (^{o} C)			
	Zone 1	Zone 2	Zone 3-4	Die
20	170	180	210	220

2.4 DSC/TGA

The thermal behavior of pure and irradiated samples was examined in a DSC Mettler Toledo apparatus, according to ASTM D3418-08 – Standard Test method for Decomposition Kinetics by Thermogravimetry, by using 5 – 9 mg of sample, within a 25 to 300 ^{o}C program, at a 10°C/min, in a nitrogen flow of 50 ml/min.

Crystallinity was calculated according to Eq. 1.

$$X_{DSC}(\%) = (\frac{\Delta H_c}{\Delta H_T}) \; 100 \quad (1)$$

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where ΔH_T is ΔH_C of 100% crystalline PE (68.4 Cal/g or 290 J/g), Heat of fusion (ΔH_c) is the energy involved in the formation and melting of crystalline regions and equal to area under the melting peak [22].

For CBA LDPE samples it was used the same ASTM D3418-08 [23], intended for TGA, by using 5 – 9 mg of sample, within a 25 to 600 ^{o}C program, at a 10°C/min, in a nitrogen flow of 50 ml/min. Decomposition temperature was recorded as the position of the peak associated with the thermal decomposition of the CBA (Azodicarbonamide).

2.5 Swelling measurements-

All LDPE samples were weighed and immersed into xylene, in a fume hood, at room temperature. Within a 15, 30, 45, 60, 90, 120, 150, 240, 420 minutes period, samples were removed from the solvent and inserted in a Quimis centrifuge, for 1 minute, to remove the excess of water adhering to the surface, keeping the same procedure for all samples. Afterwards, samples were weighed to an accuracy of 0.1 mg (using a digital balance) and inserted again in test tubes, up to reach the last period. The degree of swelling was calculated according to the Eq. 2.

$$Q(\%) \ = \ (\ \frac{m-mo}{mo}\ )\ 100 \qquad (2)$$

Where mo is the mass of the unswelled sample, and m is its mass after swelling in solvent.

2.6 Melt Flow Index

According to ASTM D1238-04C – Standard Test method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer [24], samples were evaluated in a Ceast Modular Melt Flow plastometer, at 190° C, 2.16 kg load.

2.7 Gel fraction

Gel fraction was accomplished in 0.3 g of pellets sample, previously wrapped in a stainless steel 120 mesh, immersed in 150 mL of xylene. According to ASTM D2765-01 – Standard Test

Methods for Determination of Gel Content and Swell ratio of Crosslinked Ethylene Plastics [25], this system remained for 24 hours in reflux (round bottom flask, in a heating mantle, 135°C). Non-reticulated fraction remains in the solvent and reticulated fraction is kept in the stainless steel mesh, being this assembling dried in a circulating air oven, at 60° C, for 15 hours, and afterwards maintained in a dissicator, at room temperature, for 1 hour, up to the final weighing. The calculation, in percent, of the extract (gel) follows the Eq. 3:

$$\text{Gel Fraction \%} = \frac{m_f}{m_i}\ x\ 100 \quad (3)$$

Where: m_i is the mass before extraction and m_f is the mass of the dried sample after extraction.

2.8 Melt Strength

In the Rheotens test performed, the tensile force needed for elongation of an extruded filament was measured as a function of the draw ratio, while the polymer melt has been stretched underwent uniaxial extension. The Rheotens test [26] consists of a pair of rollers rotating in opposite directions. The polymer melt strand downwards from a capillary die is drawn by the rotating rollers, whose velocity increases at a constant acceleration rate.

For extrusion of the polymer strand, Haake Rheomex 332p was used in combination with a Rheotens 71.97 apparatus manufactured by Göttfert, and samples were previously extruded at 200° C. The Rheotens test was performed in the following way: at the beginning of the experiment, the take-up speed of the Rheotens wheels was adjusted to 14.7 mm/s, the velocity of the extruded polymer strand for a tensile force zero. Then the experiment was started by slowly increasing the take-up speed of the Rheotens wheels until the polymer filament breaks. Each Melt Strength experiment was repeated five times.

2.9 Morphology of foam

Scanning electron microscopy (**SEM**) observations of the fractured surface of the foamswere made at 25 magnifications. Each foam

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sample was fractured into 1 cm x 1 cm piece after wheels until the polymer filament breaks. Each Melt Strength experiment was repeated five times.

2.9 Morphology of foam

Scanning electron microscopy **(SEM)** observations of the fractured surface of the foams were made at 25 magnifications. Each foam sample was fractured into 1 cm x 1 cm piece after cooling in liquid nitrogen in order to avoid the surface morphology damage and obtain a clean cut. They were coated with a gold-platinum alloy and scanned. A Philips XL30 Scanning Electron Microscope was used in the experiments.

3. Results and Discussion

Foams samples obtained from physical foaming using CO_2 as physical blowing agent and from chemical foaming using Azodicarbonamide (ADCA) as expansion agent were characterized according to the various tests above mentioned and showed following results:

3.1 DSC/TGA

3.1.1 PBA process

A summary of thermal behavior for 0, 10, 15, 20 and 30 kGy foams samples is shown in Fig. 1:
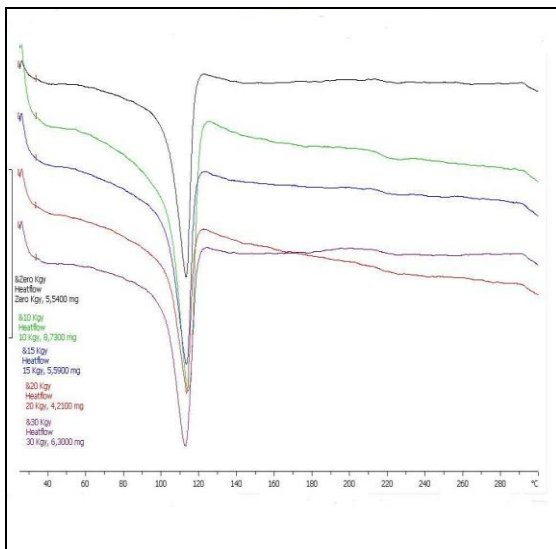

Figure 1.Thermal behavior presented by 0, 10, 15, 20 and 30 kGy foams samples.

In Table 4 are summarized thermal behavior results obtained by each individual foam sample:

Table 4: Thermal behavior shown by 0, 10, 15, 20 and 30 kGy foams samples:

Foam sample	ΔH (mJ/mg)	Tm (^{o}C)	Crystallinity (%)
0 kGy	91.0	110.5	31.4
10 kGy	92.0	111.3	31.7
15 kGy	92.7	110.6	32.0
20 kGy	87.6	110.6	30.2
30 kGy	85.3	109.9	29.4

Using the DSC enthalpy of fusion and taking 290J/g [27] as the value for the 100% crystalline LDPE, results within a 29.4 to 32.0 % range of crystallinity was found.

Low values obtained for crystallinity recommend lower temperatures settings on the extruder die, and it means a relevant parameter for the physical foamability of polyolefins [28].

3.1.2 CBA process

LDPE samples containing 2.5; 5.0 and 10.0 % of ADCA were thermally inspected as per TGA analysis, as shown in Figure 2:
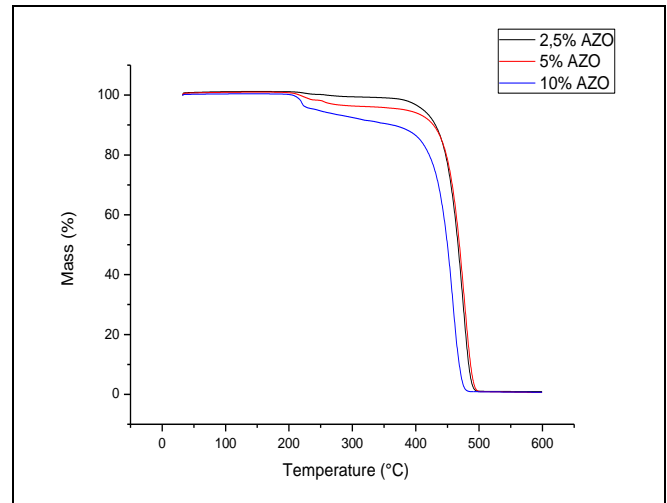

Figure 2. TGA thermograph for ADCA samples: 2.5; 5.0 and 10.0%.

According to Figure 2, decomposition temperature of ADCA is about 220° C. Therefore, 220° C is selected as the optimum temperature for the

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foaming reaction, and foam samples obtained under these conditions had a smooth surface, good appearance and uniform thickness [29].

3.2 Swelling experiments

3.2.1 PBA process

A comparison among foams samples, non-irradiated and irradiated (10, 15, 20 and 30 kGy), in the swollen state, was accomplished, using xylene as solvent. Results gathered and presented in Fig. 3 were practically identical for 0, 10, 15 and 20 kGy, printing a different behavior for 30 kGy foam sample.
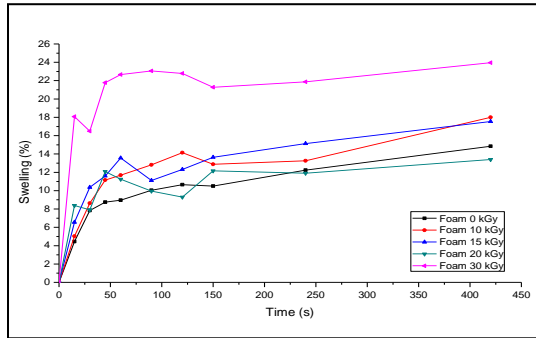

Figure 3: Swelling kinetics for 0, 10, 15, 20 and 30 kGy foams samples, immersed in xylene.

A new experiment, using 30 kGy foam sample, was performed, using besides xylene, cyclohexane, toluene and hexane. Results for immersion in xylene confirmed previous behavior for 30 kGy foam sample and a better swollen xylene capacity when compared to other solvents, as shown in Fig. 4 [30].
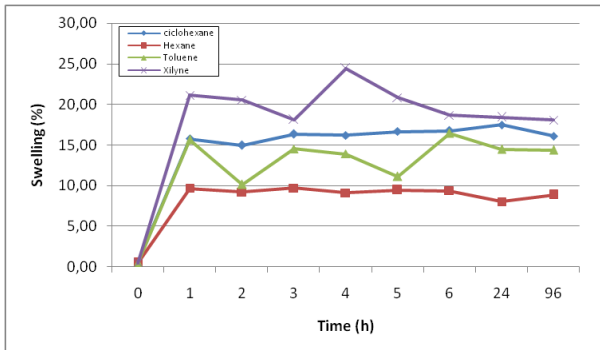

Fig.4. Swelling kinetics for 30 kGy irradiated foam, immersed in: xylene, toluene, hexane and cyclohexane.

This unexpected performance for swelling experiment verified for 30 kGy foam sample was probably due to degradation caused by gamma radiation and further corroborated by its poor Melt Strength result.

3.2.2 CBA process

LDPE samples containing 2.5; 5.0 and 10.0 % ADCA were dipped in xylene within a total 72 hours period.

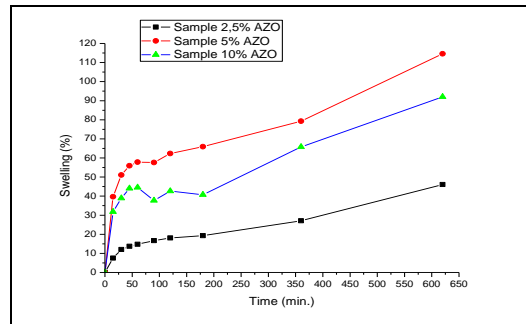In Figure 5 is shown the swelling kinetics obtained for the CBA samples:

Figure 5: Swelling kinetics for LDPE 2.5; 5.0 and 10.0 % ADCA samples, dipped in xylene.

Performance presented by ADCA samples, as per Figure 7, indicated an increase of swelling proportional to an increase in ADCA content.

3.3 Melt Flow Index and Gel fraction

All samples showed low gel content levels: $\leq 2\%$ and the processibility was prejudiced only for 30 kGy sample, according to Melt Flow Index, in Table 5:

Table 5. Gel fraction and Melt Flow Index results for LDPE irradiated and CBA samples

	Gel fraction (%)	Melt Flow Index $(g.10^{-1}\ min^{-1})$
0 kGy	0.47	2.62
10 kGy	0.42	None flow in plastometer
15 kGy	1.08	None flow in plastometer
20 kGy	0.88	None flow in plastometer
30 kGy	2.00	None flow in plastometer
2.5% ADCA	0.66	2.9
5.0% ADCA	1.44	3.0
10.0% ADCA	1.55	5.7

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3.4 Melt Strength

Melt strength increases as the molecular weight distribution (MWD) becomes broader and it is well known that not only the increase in the average molecular weight (MW) of a polymer results in higher melt strength, but also in lower MFI.

The increase in MWD is associated with the absence of flow in plastometer, according to Table 4, and consequently an increase in Melt Strength, according presented for irradiated samples, in Table 6:

Table 6. Melt Strength results for LDPE irradiated and CBA samples

	Melt Strength, 200 ^{o}C (cN)
0 kGy	0.2
10 kGy	5.5
15 kGy	6.0
20 kGy	7.8
30 kGy	Rupture when testing
2.5% ADCA	1.3
5.0% ADCA	0.7
10.0% ADCA	0.5

High melt strength is a critical processing attribute in foam production because it prevents the rupturing of cell walls during the expansion phase of the process allowing the development of a closed cell structure [31].

3.5 Morphology of PBA and CBA foams

Micrographs of specimens were investigated and for a better comparison, the characterization of PBA and CBA samples foamed under the same conditions is presented in Figures 6 and 7:
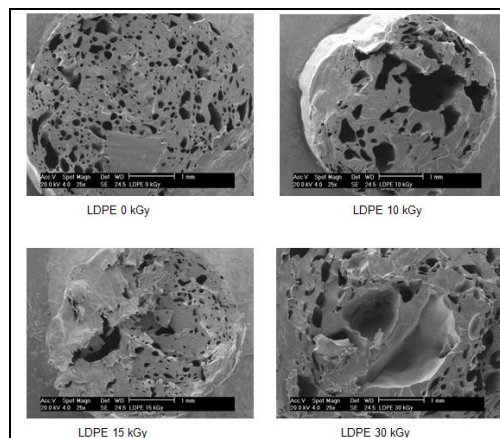

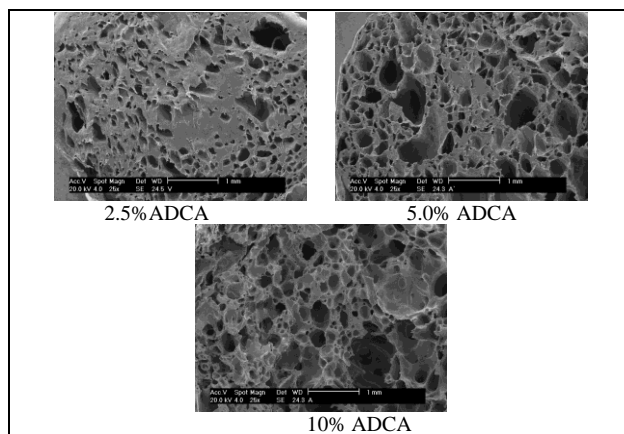**Figure 6:** SEM micrographs for irradiated samples.

Figure 7: SEM micrographs for CBA samples.

All irradiated samples presented difficulties when foaming. Non-irradiated sample and CBA samples showed a better foaming.

Conclusion

Results obtained for melt strength assessments demonstrated that gamma radiation on 0, 10, 15, 20 and 30 kGy foams samples, under ambient conditions, induce long chain branching, in spite of low values for gel fraction: lesser than 2%. The predominance of viscous behavior for irradiated samples was confirmed by melt index evaluations. Swelling experiments showed very close and low results for 0, 10, 15 and 20 kGy foams samples, when immersed in xylene, confirming the theory: the lower the swelling, the higher the crosslink density. However, 30 kGy sample was an exception, showing the highest value for swelling

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test, probably due to degradation caused by gamma radiation, corroborated by its poor Melt Strength result.

SEM micrographs indicated a better foaming for non-irradiated and ADCA LDPE samples, in spite of their poorer melt Strength results, when compared to LDPE irradiated samples. Further investigations will have to be accomplished, in order to assess foam mechanical behavior.

By the present paper it is possible to obtain satisfactory foams from LDPE irradiated at doses lower than 10 kGy, as well LDPE containing 2.5% of Azodicarbonamide (ADCA).

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