PRODUCTION, CHARACTERIZATION AND ANALYSIS OF NANO GEL OF POLYPROPYLENE WITH HIGH MELTING STRENGTH AND EXTENSIBILITY

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The goal of this work was to prepare and to characterize the morphology of gels prepared from isotactic polypropylene (iPP) of different molecular weights and further treated by gamma irradiation at 30 kGy with different concentration of TAIC. The results reveals that gels surface roughness and strength increases as the irradiation and incorporation of an additive is performed. The melt index increases with increasing dose of irradiation. The mechanical measurements show that the stiffness of iPP is greatly enhanced by electron beam irradiation and concentration of the additives (TAIC).

Introduction

The properties of iPP processed via nanogel formation induced by gamma irradiation, in the presence of acetylene and other additives have been investigated. The precise characterization of those iPP nanogels was crucial for the understanding of the nucleation processes [Af 07], [Fe 95] and to elucidate the correlation between the molecular structures [Ho 98], [Ku 96] and mechanical properties [Su 05] of the resulting materials [Za 02].

In this work, different samples of iPP gels were prepared and treated by gamma irradiation and the addition of an additive, TAIC (Triallyl isocyanurate). The morphology of the resulting materials was then investigated [Wf 07]

By different microscopy techniques, namely optical microscopy, SEM [Re 07] and AFM. Gels surface roughness was also evaluated by profilometry and elipsometry [Za 95], [Za 04].

Experimental

Gels of iPP were prepared from polymer samples of different molecular weights supplied by BRASKEM, types iPP-603 and iPP-210. The i-PP is supplied as granulated (with Irganox 1076) and spheres (without antioxidants). Additionally, some gels samples were modified by gamma irradiation at 30 kGy, without and with an additive, TAIC. The gel preparation was as follows. iPP was dissolved in xylol at 135 ºC. The resulting solutions were then cooled down to 3 different temperature values, namely 30, 25, and 20^{o}C. Only samples were the gel was completely formed were analyzed. The resulting gels were isolated by filtering.

Samples of gels were characterized by different microscopy techniques, namely optical microscopy, SEM coupled with EDX, and AFM. Besides, gels surface roughness was investigated by elipsometry and profilometry.
Results and Discussion (Times New Roman 10, bold)

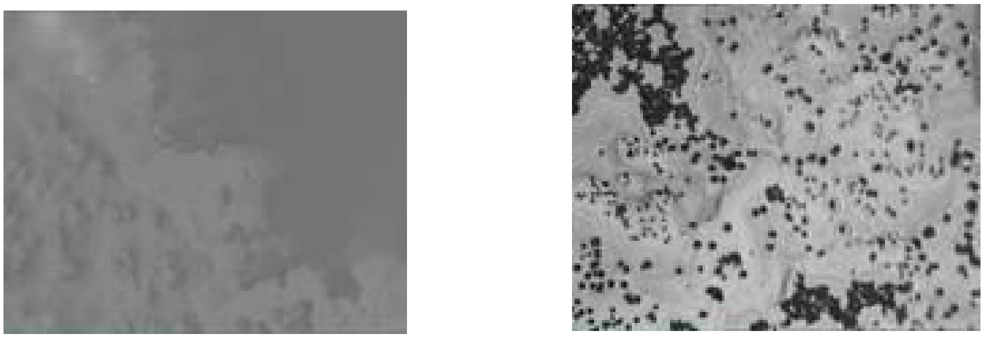
Results and Discussion

The optical microscopy photos are shown in Figure 1 revealing the nucleation of iPP nanogel. These nuclei correspond to the sites in iPP of higher energy where the polymer chains start to concentrate and form a high density material.

SEM micrographs are presented in Figure 2 showing the topographic of iPP gel surface during its formation. Samples containing the additive and treated under gamma irradiation display a higher surface roughness when compared to control (untreated iPP gel). AFM images, Figure 3, and profilometry results shown in Table 3, corroborate the results from SEM, indicating that iPP surface of gels becomes rougher as TAIC concentration and irradiation dose are increased.

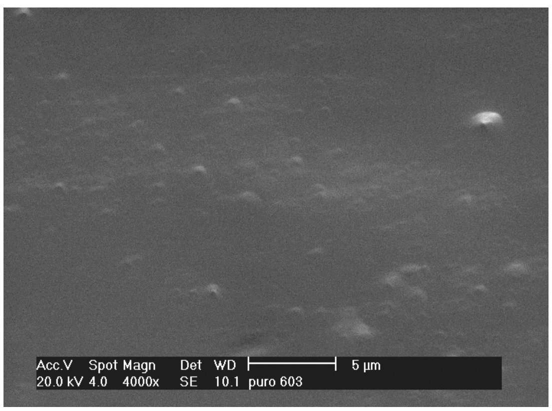
Elipsometry data, presented in Table 4, reveals that besides the fact that iPP gel surface roughness increases as the treatment and TAIC addition are conducted, there is also an increase on the refractive index of the resulting material. Consequently it can be concluded that the material density increases after treatments.

DSC analysis are presented in Table 5 reveal for the crystallinity of the gamma-irradiated iPP does not change with different concentration of TAIC, but the relative level and the order of nanogel-phase increase. Under the controlled TAIC concentration, the thermomechanical deformation of iPP decrease, and the initial and final thermal degradation temperature of iPP rises up by 70 to 125°C higher, respectively, indicating that the modified iPP has higher thermal stability than the non-UV irradiated iPP. The tensile and impact strength, the elongation at break, and the Young's modulus of the modified iPP are enhanced, exhibiting the toughened and strengthened effects.

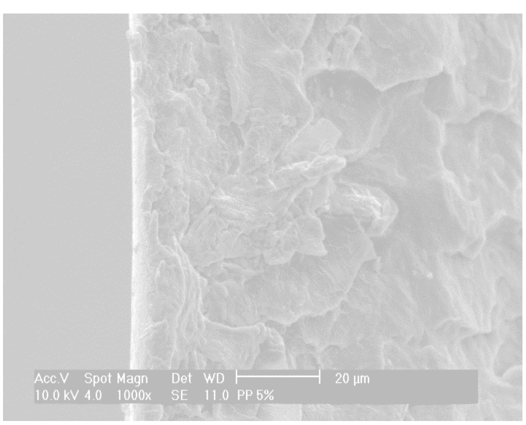


a) b)

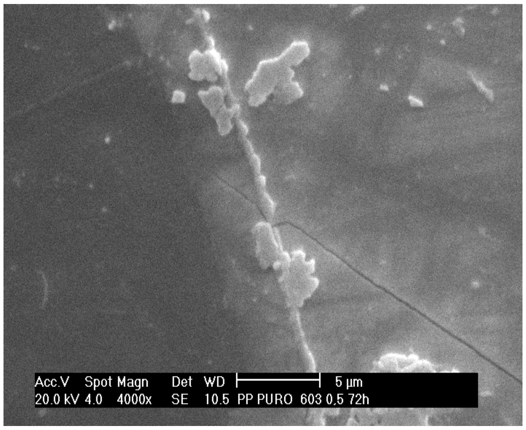
Figure 1: Optical Microscopy of iPP gels (iPP 603) in different magnification: a) 1500 x and b) 500 x.



a)

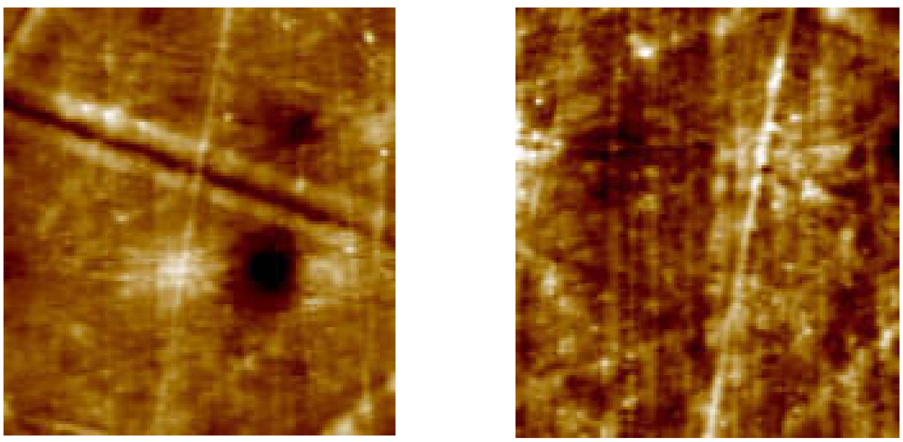


b)



c)

Figure 2: MEV micrograph of micro gel of pure and irradiated iPP with gamma beam source. a) i-PP without etching; b) pure i-PP after 100 hours of etching; c) modified with TAIC 5%; d) pure i-PP after 72 hours of etching.



a) **b)**

Figure 3: AFM images 10 x 10 μm of a) treated and b) pure iPP gels. Z = 5 μm

Table 3: Distances of higher and lower levels, and average on iPP gels surface measured by profilometry.

Type of polymer	**thickness of the iPP film**
Pure iPP 603	2,8 10^{-6}m (roughness larger difference) 0,9 10^{-6}m (average difference)
iPP 603 5 mmol	13,2 10^{-6}m (roughness larger difference) 0,6 10^{-6} m (average difference)

Table 4: Elipsometry data for pure and treated i-PP gels.

Type of polymer	Angstron	Refractive Index
Pure iPP 603	6472 +/-189	1,2
iPP 603 5 mmol	6.328 +/-1715	1,4

DSC

Table 5

Amostra	**ΔH de Fusão (mJ/mg)**	**% Cristalinidade**
PP puro	65.634	31.707
PP 0.5 TAIC	64.0536	30.943
PP 1.0 TAIC	54.4231	26.291
PP 1.5 TAIC	53.0036	25.605
PP 3.0 TAIC	66.4962	32.123
PP 5.0 TAIC	65.3473	31.568

ΔH de fusão PP 100% cristalino= 207 J/g

Conclusion

The structure and mechanical properties of isotactic polypropylene (iPP) functionalized by electron beam irradiation are investigated by differential scanning calorimetry, and mechanical measurements.According to SEM, AFM, profilometry and elipsometry analysis, the surface of nanogels prepared from iPP becomes rougher as the gamma irradiation and the addition of TAIC were performed. These treatments may have a strong impact on the properties of iPP gels. Future work consists on the calculations of statistical sizes of grains by X-ray diffraction and scattering, and light scattering.

The experimental results show that the degree of crystallinity nucleation, temperature and the dimensional stability increase with dose of gamma irradiation and the concentration of additives.

Acknowledgements

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