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Statistical Analysis of the Influence of the Injection Molding Temperature on the Tensile Properties of Polypropylene

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ABSTRACT: Polypropylene (PP) and other polymers have been drawing increasing attention from the industry in recent decades, which has been translated to an ever-growing utilization world-wide. Besides, PP is one of the cheapest polymers in the market, being also suitable to large-scale and easy mechanical recycling. The PP structure is affected by high processing temperatures, which cause rupture of chemical bonding and degradation. This degradation ultimately affects the mechanical properties of the polymer, such as its response to tensile stress. In this work, PP is injection molded as standard tensile specimens. The injection temperature is varied between 160 and 200 ^{o}C and the influence of this parameter on the main tensile properties of the specimens is studied. Tensile tests were carried out according to ASTM D638 where Young modulus and tensile strength were measured for between 15 and 30 specimens for each temperature. A commercial software was used for the statistical analysis of the significant differences between mean values, in order to verify apparent trends identified in the variations. The drawn conclusions show that maximum values for the properties occur at different temperatures and differences between temperatures based only on mean values can not be readily inferred. The results clearly indicate that a more thorough examination of the experimental data, such as the one shown here, is necessary than what is usually presented in the related literature, which considers only mean and standard deviation values.

Keywords: polypropylene, injection molding, injection temperature.

1. Introduction

Polypropylene (PP), a semicrystalline polymer, is largely used for short-term packaging and engineering applications, such as containers, automotive parts (Guerrica-Echevarria et all, 1996), specially due to its combination of low price, low density and versatility (Bonelli et al, 2001).

One important issue nowadays is the minimization of the environmental impact of discarding materials in landfills, increasing the search for easily recyclable materials, such as PP, which is in fact extensively recycled, decreasing the need for new raw-materials (Wassermann et al, 2002).

When recycling plastics one has to bear in mind the possible degradation to which the material is exposed, and therefore it is necessary to verify the properties of interest to a particular application.

Although PP is highly susceptible to photo-degradation when exposed to environmental conditions (Bonelli et al, 2001), it is often used in artifacts such as packaging and automobile bumpers. The absorbed energy due to radiation and light cause rupture of C-H bonds of the tertiary carbon atom groups and reduction of molecular weight, with modification of the chemical structure. This modification considerably changes the mechanical (tensile, impact and flexural properties, for instance) and physical properties of the polymer (melting flow index etc.) (Kelen, 1983; Billingham et al 1983; Whiteley et al, 1982).

As expected, thermal degradation of PP can occur depending on processing conditions. This fact has been verified in the literature by authors such as Fujiama et all (2000) and Billiani et al (1990), who studied the influence of the injection temperature of PP. Actually, during injection molding, temperature, injection rate, and injection pressure are all parameters that must be controlled to minimize polymer degradation (Phillips et al, 1994). Kalay et al (1997) mention that these factors along with mold characteristics may affect the properties of the final part. They have also concluded that low injection rates and temperatures close to the melting point, favors molecular orientation, because the chains organize themselves on a slower rate promoting more spherulites and therefore polymers with increased cristallinity. Martins & Paoli (2002) also points out that if the injection temperature is low, the viscosity is high and therefore shear stresses will be more intense, causing micro-fractures of the molten polymer and low homogenization.

Most papers on the subject of thermo-mechanical degradation of PP during injection molding report mean values and standard deviations only. Although these informations are important, they may not properly represent the set of data and may lead to imprecise conclusions.

The main objective of this work is to carry out statistical analysis (variance analysis) of the variation of mechanical properties, namely tensile Young modulus and tensile strength, of PP specimens injected under different injection

temperatures and, by doing that, to justify the importance of a more detailed mathematical analysis when referring to this sort of data in the literature.

2. Methodology

A commercial polypropylene (melting flow index of 13.4 g/10min) was used in this work. Specimens were injection molded according to ASTM-D638, type II, for tensile tests.

Five injection temperatures, namely 160, 170, 180, 190 e 200ºC, were used. Temperature of the mold was kept relatively high (around 50º C).

Tensile tests were conducted in an EMIC DL10000 Universal testing machine according to ASTM-D638, at a strain rate of 5 mm/min. Deformations were recorded via extensometer.

Tensile strength (TS) and elastic modulus (E) were determined and mean and standard deviations values corresponding to 15 to 31 different specimens for each condition.

Films were obtained from the injected specimens for X-Ray measurements. The X-Ray difractometer Lab X XRD-600 Shimadzu, with a $Cu_{K\alpha}$ source. Data were acquired as a function of the 2θ angle using continuous scans at 2ºC/min. The equipment estimates material cristallinity indexes by calculating areas relative to the amorph and crystallyne regions of the sample.

A commercial software was used for the statitistical analysis of variance (ANOVA). The results of the tensile tests were analyzed using ANOVA/MANOVA for the Tukey test. Correlation coefficients between sets of data were also calculated.

3. Results and Discussion

Tab. (1) describes mean and standard deviation values for tensile stress (TS) and Young modulus (E) at different injection temperatures used. These values are also shown in Fig. (1) e (2), where the different labels (letters) are the result of the comparative statistical analysis of different set of data. On this nomenclature, different labels indicate statistically distinct populations, and when a column contains two letters, it means that there is no significant difference between that column and the other columns labeled with the same letters.

Table 1. Mean and standard deviation values for TS and E at different injection temperatures.

Temperature (ºC)	160	170	180	190	200
TS (MPa)	27.9 ± 2.4	29.9 ± 1.9	26.4 ± 3.6	22.1 ± 3.1	17.1 ± 2.4
E (MPa)	1668 ± 141	1795 ± 133	1900 ± 125	2027 ± 149	1972 ± 239

One can notice in Fig. (1) that the tensile stress values at 160, 170 and 180º C are not significantly different. Besides, the 170 and 180ºC data differ from each other and 190 and 200ºC differ from all others. The maximum mean value for TS is actually 29,9 MPa at 170ºC.

These facts are as expected because, according to studies of Billiani et al (1990), there is increase of molecular vibrations and breakage of covalent bonds causing significant decrease of molecular weight of the injected specimens as the temperature is increased much further from the melting point. This ultimately negatively affects the TS value.

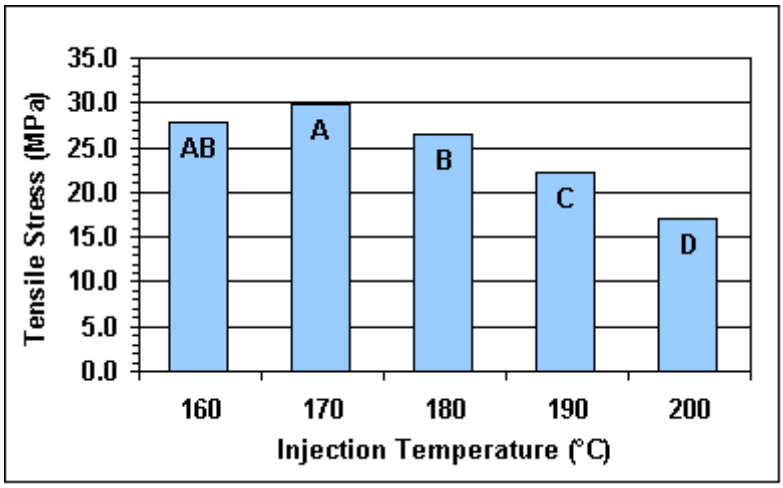

Figure 1. Mean tensile stress values at different temperatures.

The lowest temperature, on the other hand, showed a decrease of TS values since the material may not have melt or flown in a homogeneous way, and therefore being more prone to high shear stresses and chain breakage. It is important to remember though, that this decrease was not found to be genuine according to the statistical analysis.

Fig. (2) shows the mean Young Modulus values for the different temperatures, where it can be seen that the maximum value (2027 MPa) was found at 190°C. The data corresponding to 160°C differs from all others and 180 is not different from 170 and 200°C, although the last two differ from each other. The data for 190°C was not found to be statistically different from that for the 200°C.

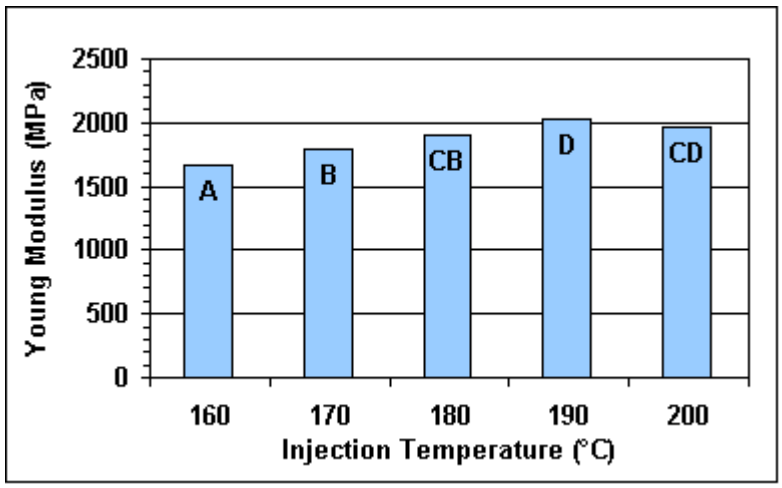

Figure 2. Mean Young Modulus values at different temperatures.

The observed increase of Young Modulus values from 160 to 190°C indicates that the increasingly higher temperatures not exceeding much the melting point, is likely to allow for a higher orientation of the crystalline portion of the polymer chains. X-ray results (Fig 3 and 4) in fact confirmed the hypothesis of increase in cristallinity, since it increased from 32.9% (160° C) to 39.6% (190°C).

Temperatures higher than 190°C, in this case, caused material degradation and a very brittle material as proved by a decrease of the Young modulus (Fig. 2) and development of a yellowish coloration.

The low cooling rate of the injected specimens due to a higher than usual mould temperature favored a lower solidification rate, allowing the maintaining of a higher cristallinity and therefore a higher Young modulus is found at all temperatures comparing to other literature values (Martins & Paoli, 2002; Guerrica-Echevarria et all, 1996).

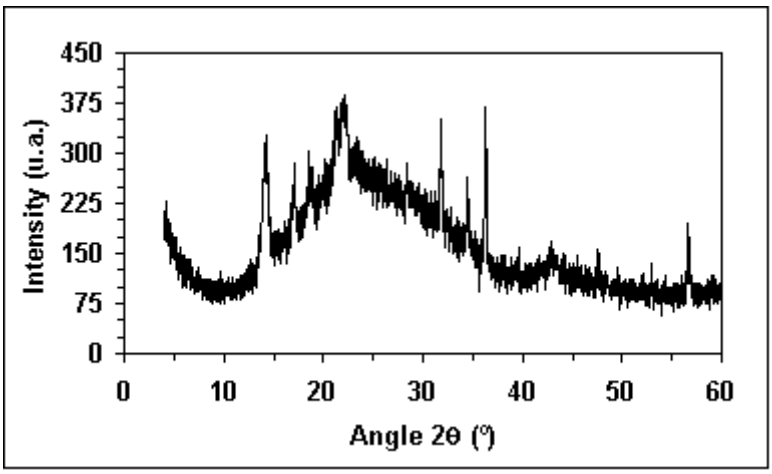

Figure 3. Polypropylene X-Ray analysis – Injection temperature = 160 ºC.

The already presented results corroborates what has already been mentioned by other authors. The important issue not usually mentioned in the literature is whether the differences (changes of tensile stress and Young modulus values) are actually significant or just due to the dispersion of results. In this work this subject is addressed by the results of the statistical analysis.

It is difficult however to imply that from the presented results in the literature, since it is only usually given mean and standard deviation values for the different properties of polymers. Drawn conclusions based only in mean values can me misleading unless a similar statistical approach is taken. In the case presented here, for instance, maximum TS

was obtained at 170°C, but this set of data is not statistically different from that for the 160°C. In other wprds, from the point of view only of the tensile stress, the injection temperatures of 160 and 170°C cause a similar effect on the PP. It is important to bear in mind, however, that each specific material or processing condition requires similar statistical analysis in order to identify individual actual property peaks. From a practical point of view, one may say that lower production costs could be achieved in processes like extrusion and injection molding in case similar analysis were carried out for each specific processing condition.

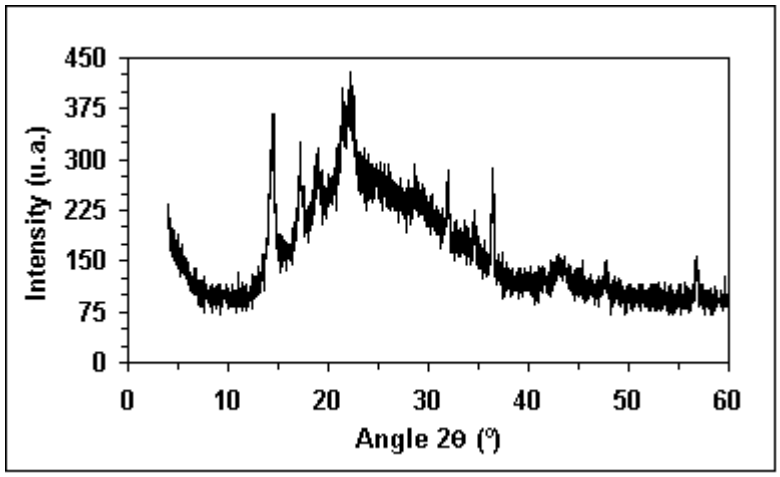

Figure 4. Polypropylene X-Ray analysis – Injection temperature = 160 ºC.

Tensile test results were also used to make dispersion diagrams. Fig. (5) shows this diagram for the set of data obtained at temperature equal to 170°C, where one can notice the very poor correlation between Young modulus and tensile stress. The complete correlation analysis is shown in Tab. (2), where low values indicate poor correlation at all temperatures.

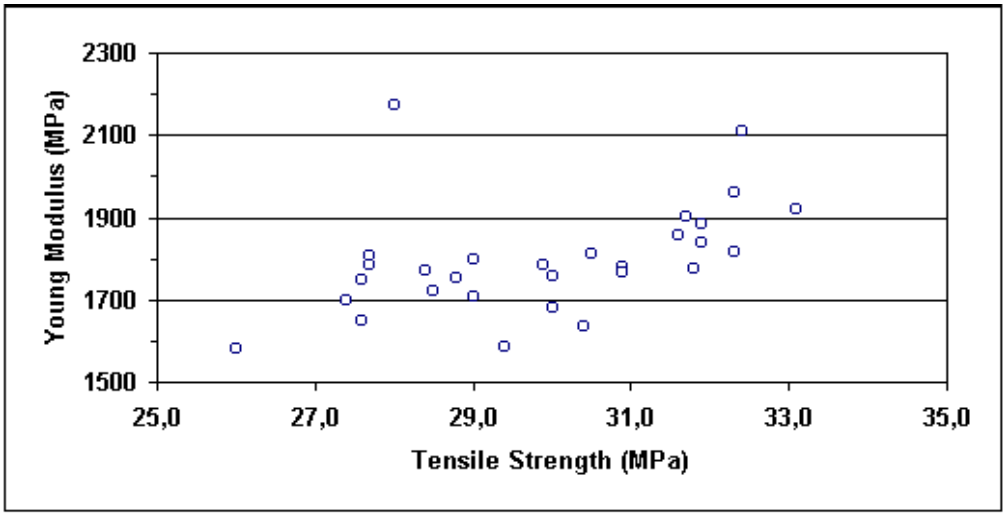

Figure 5. Dispersion diagram correlating Young Modulus and Tensile Strength at 170 ºC.

Table 2. Correlation coefficient between Young Modulus and Tensile Strength at different temperature.

Temperature (ºC)	160	170	180	190	200
Correlation coefficient	0.307	0.453	0.007	0,222	0,367

4. Conclusions

The measured properties, namely tensile stress and Young modulus, were found to vary with injection molding temperature following different patterns and these differences were mainly attributed to changes in cristallinity and polymer chain structure. Peak values occurred at different temperatures, even because these properties are not correlated.

Differences between temperatures based only on mean values were shown to lead to incorrect conclusions. The results clearly indicate that a more thorough examination of the experimental data, such as the one shown here, is necessary. This work therefore indicates that considerations based only on mean and standard deviation values, usually presented in the literature, have to be carefully studied before firm conclusions can be drawn.

5. Acknowledgements

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6. References

Guerrica-Echevarría, G.,Eguizábal, J.I. and Nazábal, J., 1996, " Effects of reprocessing conditions on the properties of unfilled and talc-filled popypropylene", Polymer Degradation And Stability, Vol. 53, San Sebastian, Spain, pp.1-8.

Bonelli, C.M.C., Martins. A.F., Mano, E.B. and Beatty, C.L., 2001, "Effect of Recycled Polypropylene on Polypropylene/ High Density Polyproplylene Blends", Journal of Applied Polymer Science, Vol. 80, pp. 1305-1311.

Wassermann, A.I., Espindola, L.C., Castilhos, A.F., Holtz, N. and Cardozo, N.S.M., 2002, "Análise da processabilidade na reciclagem de plásticos misturados", Sulmat Congresso em Ciências de materiais do Mercosul, Porto Alegre, Brazil.

Kelen, T., 1983, "Polymer Degration", Van nostrand Reinhold, New York, United States.

Billingham, N.C. and Calved, P.D., 1983, "An Introduction"- Degradation and Stabilization of Polyolefins, Aplie Science Publishers, Ed. N.S. Allen, London, cap.1.

Whiteley, K.S., Heggs, T.G., Koch, H. and Mawer, R.L., 1982, "Polyolefins" ,Ullmann's Encyclopedia of Industrial Chesmistry, Ed. B. Elvers et all, VCH, New York, United States 487 p.

Billiani, J. and Fleischamann, E., 1990, "Influence of Injection Rate and Melt Temperature on Polypropylene during Injection Moulding without Packing", Polymer Degradation and Stability, Vol. 28, England, pp 67-75.

Phillips, R., Herbert, G., News, J. and Wolkowicz, 1994, " High Modulus Poypropylene: Effect of Polymer and Processing Variables on Morphology and Properties", Polymer Engineering and Science, Vol. 34, Nº23

Kalay, G. and Bevis, M.J., 1997, "Processing and Physical Property Relationships in Injection- Molded Isotactic Polypropylene. 1. Mechanical Properties", Journal of Polymer Science, Vol. 35, pp. 241-263.

Fujiyama, M., Masada, I. and Mitani, K., 2000, "Melting and Crystallization Behaviors of Injection-Molded Polypropylene", Journal of Applied Polymer Science, Vol. 78, pp. 1751-1762.

Martins, M.H. and De Paoli, M.A., 2002, "Estudo que determina as melhores condições de processamento do polipropileno", Plástico Industrial.