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Abstract. This paper deals with the optical and colorometric properties of the recycled polymer during numerous internal reprocess. The effects of the number of grinding-injection cycles, three process parameters (material temperature, mold temperature, and injection rate) were investigated. One most limit for this kind of study is the large number of experiments that requires long time and significant investments. The idea is to vary five injection parameters (Tmaterial, Tmold, injection rate for five injection cycles using statistical approach. The five variables were investigated at three industrial used levels. The number of recycling varies from cycle 0 to cycle 4 at five levels. The complete matrix for screening was designed using D-optimal quadratic design. The experimental design was generated with the statistical software MODDE 10.1-Umetrics. A set of 42 experiments was carried out to determine the influence of injection parameters, polluant and recycling on the appearance properties of smooth and grained surfaces. The statistical software package Nemrodw® version 2007, LPRAI (Marseille, France) was used to analyze the experimental design.

Keywords: Recycling · Gloss properties · PP · Experimental design

1 Introduction

Nowadays, appearance properties of injected objects are one of the most defies for automotive industries. The necessity of recycling was also added for quality commitment. In fact, recycled polymer improve cost performance [1]. Nevertheless, this recycling process often brings unfavorable effects, e.g., an increase in appearance properties. In order to accomplish this compromise between recycling and good esthetic of produces, studies on the effect of recycling on the appearance properties of polymers are re-quired [2]. The effect of recycling on optical properties of polymer films has been few analyzed in the literature [3–5] Among other plastics, polypropylene

(PP) is a commodity polymer products and used in large quantities for automative applications [6, 7]. The main reasons for the success of PP are its quite good price/performance ratio, its excellent mechanical properties, and suitable optical characteristics [8–10]. In fact, the massive consumption of this polymer makes its recycling strategically very important for the environmental policy of industry [11] Conversely, appearance properties of polymer products are mostly fixed by the processing parameters. A low cavity surface temperature lead to a precipitately polymer melt freeze. Subsequently, a frozen layer will be formed during filling process at the inter-face between the hot polymer melt and the cold mould cavity. This phenomena conducts to a series of defects of the ultimate moulded parts, such as flow mark [12] weld mark [13], swirl mark, roughness [14], low gloss [11], and low replication accuracy [13].

The surface quality of plastic parts can be improved significantly by increasing mold cavity temperature [15]. A better replication of the mold texture can be achieved at a lower melt viscosity at higher shear rates and higher mold temperatures. This provides a higher gloss in smooth regions. The gloss properties had a significant effect on the color: all the factors lead to an increase in gloss showed concomitant effects of increasing the color coordinate b^* and of decreasing the lightness L^*. Studies were published showing that the processing parameters may affect the gloss of injection molded parts [9], the mold temperature commonly being considered the more important parameter to be controlled. It was shown that the mold temperature is the more relevant parameter and that a clear interaction exists between that parameter and the holding pressure.

This quantitative descriptor of appearance (gloss and color) is the consequence of a psychophysical phenomenon of visual perception correlated with a situation in which the light reflected from the surface of an opaque sample is either predominantly in the specular direction (gloss) or diffuse in all directions (color) [7]. the color depends on the illumination conditions, the observation angle, the optical characteristics of the material, the amount of the colorant present, the surface topography [6], and the gloss [3]. Work on polymeric surfaces has shown a decrease in gloss with increased roughness [15, 17, 18].

In this paper, the effect of process conditions (material temperature, mold temperature, injection rate) are investigated. The impact of recycling on the colorimetric properties of polypropylene containing 2 wt% of pigment using statistical approach were evaluated.

2 Materials and Techniques

2.1 Materials

The PP homopolymer SABIC® PP 575P produced by SABIC Europe was used in the experiments. SABIC® PP 575P is an homopolymer for injection molding. A masterbach supplied by Clariant was used to obtain a gray-beige complexion. The masterbach was characterized with EDS analysis by scanning electron microscopy. The analyze show the presence of: $CaCO_3$ (calcium carbonate), TiO_2 (titanium dioxide) and Sb_2O_3 (Antimony trioxide). Blending of PP with 2 wt% of pigments (masterbatch) was performed in the PEP "plastics technical center" with a co-rotating intermeshing twin-screw extruder (Clextral EVOLUM HT 32, diameter 32 mm, centerline distance 21 mm) with a length to diameter ratio L/D 44. The screw profile is made of conveying and kneading elements, also using opposite pitch to ensure melting, mixing, shearing, and a good dispersion of the components. The extrusion process was carried out with a screw rotation speed of 350 rpm, at a temperature of 210 °C and a throughput of 30 kg · h^{-1}. The colored pellets were also injection-molded to obtain samples for study.

2.2 Techniques

2.2.1 Polymer Processing

The mold was supplied by CFO company specialized on "Manufacturing Design Tools". The mold design was realized in C2MA to obtain samples of 100 mm 100 mm with a thickness of 2 mm. the samples show two different faces, a polished mirror surface and a rough surface. All specimens were prepared on a Krauss Maffei KM50-180CX injection-molding machine. The maximum clamping force is 50 t. During the course of experiment, corresponding to each set of experimental parameters, the 5 first injected samples are thrown away to ensure that the process was stable. PP was injected from zero to five steps of recycling. Samples are grinding with a Cutting Mill SM 300 to be reinjected.

In a purpose of understanding the effect of injection parameters and recycling on the appearance properties of polymers, several injection parameters and cycles numbers must be tested. One most limit for this kind of study is the large number of experiments that requires longtime and significant investments. The idea is to vary three injection parameters temperature of material (Tmaterial), Temperature of the mold (Tmold) and the injection rate for five injection cycles. The work material selected for the study was polypropylene because of its large use and the need to recycle it with the maintaining of good aspect properties.

The effects of four factors known to influence injection conditions, material temperature, mold temperature, injection flow and number of injection cycles were studied using a statistical approach. The three variables (materiel temperature, mold temperature and injection flow) were investigated at three industrial used levels (materiel temperature: 220, 240, 260 °C, mold temperature: 30, 50, 80 °C and injection flow: 10, 20, 40 cm^3/s). The number of recycling varies from cycle 0 to cycle 4 at five levels. The complete matrix for screening was designed using D-optimal quadratic design. The experimental design was generated with the statistical software MODDE 10.1-Umetrics. A set of 45 experiments was carried out to determine the influence of injection parameters and recycling on the appearance properties of samples. The statistical software package Nemrodw® version 2007, LPRAI (Marseille, France) was used to analyze the experimental design.

The response approach involving a D-optimal quadratic design was adopted for studied the effect of injection parameters and recycling on the final response (colorimetric and gloss). Each variable (material temperature, mold temperature, injection rate) was studied at three different levels (1, 2, and 3). The four recycling cycles was studied with four levels (1, 2, 3, and 4). All variables were taken at a central coded value of zero. The minimum and maximum range of variables investigated and the full experimental plan with respect to their actual and coded forms. The analysis of the data was carried out to obtain an empirical model defining the response Y (Eq. 1), which is the value of colorimetric (L*, a*, b*) or gloss (R20, R60, R85).

$$Y = b_0 + b_{1A} \cdot X_{1A} + b_{1B} \cdot X_{1B} + b_{2A} \cdot X_{2A} + b_{2B} \cdot X_{2B} + b_{3A} \cdot X_{3A} + b_{3B} \cdot X_{3B} + b_{4A} \cdot X_{4A} + b_{4B} \cdot X_{4B} + b_{4C} \cdot X_{4C} + b_{4D} \cdot X_{4D} \quad (1)$$

2.2.2 Colorometric Properties

CIELAB was intended to present a standard, approximately uniform color space which could be used by everyone so that color values could be simply compared. In a uniform color scale, the differences between points plotted in the color space correspond to visual differences between the colors plotted. The L* axis is the light-dark axis, and thus gives a measure of the relative brightness of the sample ranging from total black (L* = 0) to the white (L* = 100). The a* axis is the red-green axis (positive a* = red, negative a* = green), and the b* axis is the blue-yellow axis (positive b* = yellow, negative b* = blue) [15, 12] (Fig. 1).

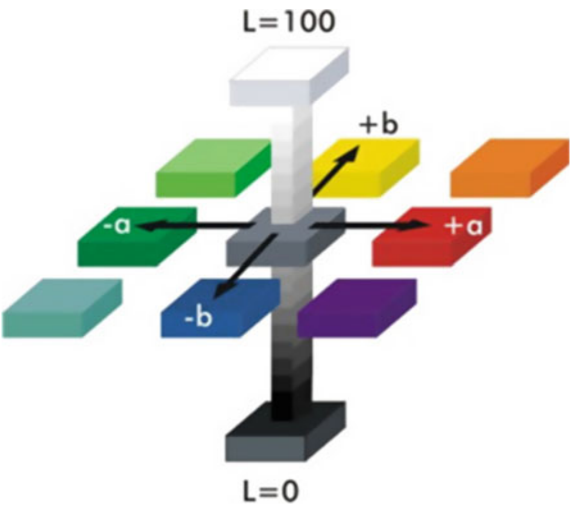

Fig. 1 (L*, a*, b*) representing CIELab color space

3 Results and Discussion

The results of colorimetric responses are analyzed with the statistical software package Nemrodw-version 2007, LPRAI (Marseille, France) and shown respectively in Fig. 2. The reference is taken on the value of the highest level (black bar). The influence of the injection temperature (220, 240 and 260 °C), the mold temperature (30, 50 and 80 °C), the injection rate (10, 20 and 40 cm^3/s) and the number of injection cycle (cycle 0 to cycle 5) on the gloss of different PP samples with grained surface is shown in Fig. 2.

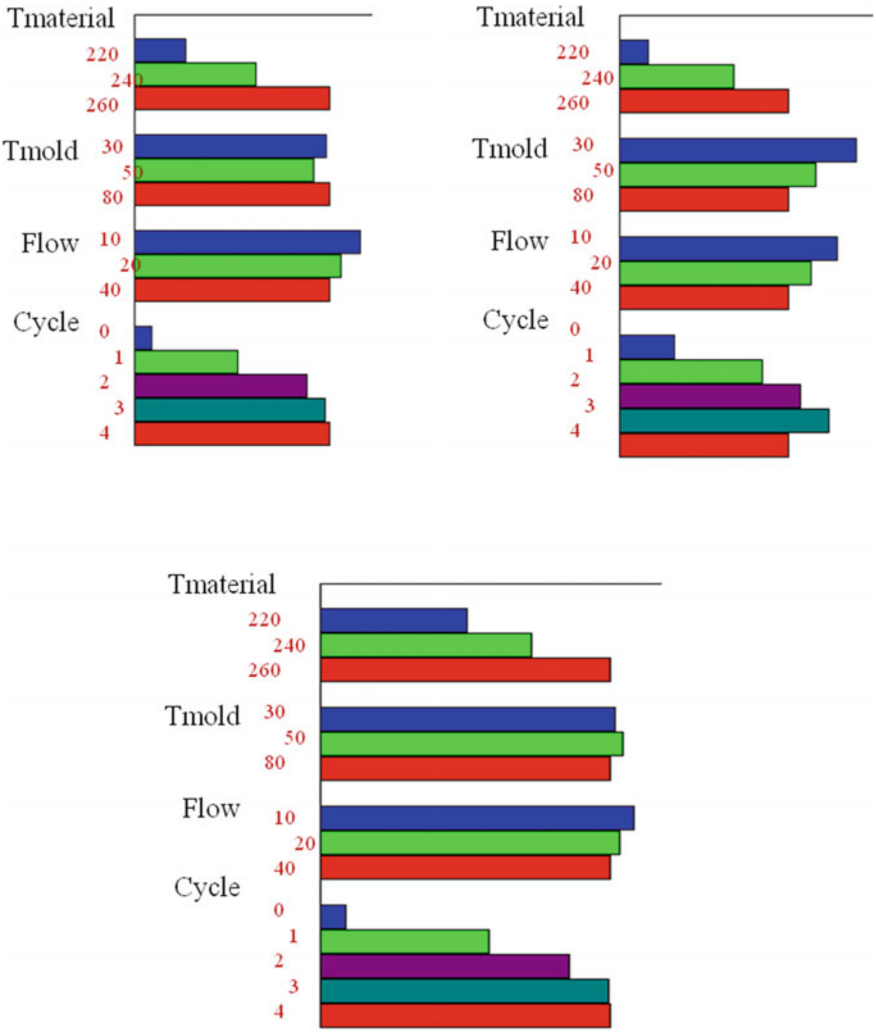

Fig. 2 Graphic representation of the difference in weight of the factors on total effect for the colorimetric response. **a** L*, **b** a*, **c** b*

The colorimetric property depends on variations in injection conditions and number of recycling cycles. Thus, the recycling and material tempera-ture have the most important effect on colorimetric. The variations of colorimetric parameters are obtained by changing the number of cycle and the Table 1.

The appearance properties of the injection molded samples showed that variations in injection parameters and number of cycles had a significant impact on colorimetric.

Table 1 The effect of injection parameters and number of cycles on the colorimetric (Pareto individual effect from response analyses with Nemrodw)

	L*	a*	b*
Cycle 0/4	**42**	24	**57**
Cycle 1/4	12	0	13
Cycle 2/4	0	0	1
Cycle 3/4	0	3	0
T_{mat}220/260	**35**	**46**	21
T_{mat}240/260	9	7	6
T_{mold} 30/80	0	11	0
T_{mold} 50/80	0	2	0
Flow 10/40	2	6	0
Flow 20/40	0	0	0

Generally appearance properties are related to the surface roughness. The differences appearance properties between samples were presumed to arise from the variation in the surface topography in ac-cording to the literature [9, 16].

4 Conclusion

The variations of properties due to numerous recycling are in the same range that is observed by changing processing parameters. In fact, the optical properties of recycled parts can be maintained during numerous internal recyclings. The number of cycle and the material temperature are the most important factors changing the colorimetric properties of the material. Thus, the aspect properties of recycled parts can be maintained during numerous internal recycling up to three cycles.

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