Evaluation of homogenization properties of masterbatches

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Abstract

Appearance is a very important property of different products, therefore it is important to investigate what kind of parameters and processing circumstances influence them. Proper investigation requires a reliable and repeatable measurement method. Recently a novel evaluation method was developed to evaluate the color inhomogeneity of injection molded specimens. In this work this novel inhomogeneity evaluation method was used to compare homogenization properties of different masterbatches. It was also shown that the homogenization properties of different masterbatch recipes can be evaluated and developed with this technique. The results this method produced were validated by a group of trained technicians and their results were correlated with the inhomogeneity levels derived from the new method.

Keywords: masterbatch, homogenization, injection molding, inhomogeneity evaluation, inhomogeneity sensation

1. Introduction

The appearance of injection molded products is crucial and it does not only mean the color shade itself, but the evenness of the color as well. It has been published by many authors [1, 2] that technological parameters of injection molding play a significant role in the color and gloss of polymer products. Sathyanarayana et al. [3] proved that the dispersion of the fillers and colorants are influenced by processing parameters, which could be the root cause of color inhomogeneities in polymer products. In industrial applications – where the color or its homogeneity is important – the CIELAB color space is widely used for the evaluations due to its device-independency [4-6] and good correlation with human sensation [7].

Mixtures are usually characterized by the intensity and scale of segregation. The intensity of segregation in a two-component system is described in Eq. (1)

$$I = \frac{\sigma^2}{\overline{ab}}, \tag{1}$$

where I is the intensity of segregation, σ^2 is the sum of the squared deviation from the mean composition, and $\overline{ab}$ is the product of the average concentration of components a and b. In this form I is a number ranging from zero to one, where zero means that the concentration distribution is totally homogenous, the concentration of a and b equals to the average concentration in every inspected sample, while if the intensity of segregation is one, means that all the inspected sample contained only a or b components, which is the maximum segregation.

The scale of segregation can be derived from the correlation coefficient, $\rho(r)$, which is in connection with the correlation function, $R(r)$. The correlation function is defined in Eq. (2).

$$R(r) = \langle [a_x - \bar{a}][a_{x+r} - \bar{a}] \rangle, \tag{2}$$

which can be interpreted as the deviation of concentration from the mean value at point x, multiplied by the deviation of concentration at point $x+r$, and averaged over the entire mixture. This can be written in a normalized form considering that in $R(0)$ the correlation function will equal the variance σ^2. This is represented in Eq. (3)

$$\rho(r) = \frac{R(r)}{R(0)} = \frac{R(r)}{\sigma^2}. \tag{3}$$

From Eq. (3) the linear scale of segregation is the following:

$$S_L = \int_0^\infty \rho(r) dr, \tag{4}$$

and the volume scale of segregation is:

$$S_V = \int_0^\infty \rho(r) r^2 dr, \tag{5}$$

which has practical importance, since it represents the size of the clumps in a mixture. Figure 1. illustrates the scale and the intensity of segregation in a mixture [8].

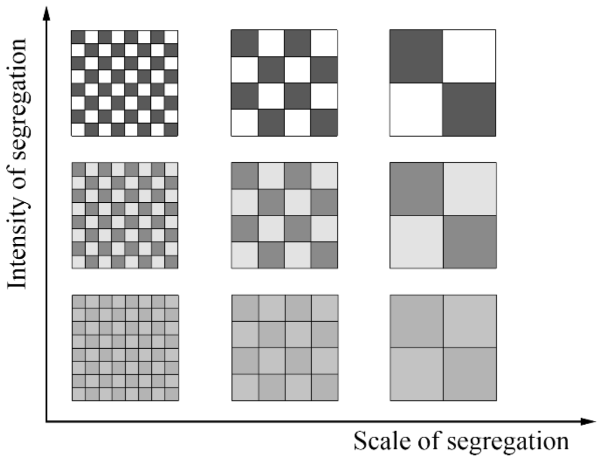


Figure 1: Illustration of the scale and the intensity of segregation [8]

In most applications the components need to be well dispersed and evenly distributed throughout the base material. Proper dispersion is a focus area in the case of nanoparticle-filled polymers as well [9], therefore several papers deal with this issue, such as the work of M. Salzano et al., who developed a novel method to generate nanoscale ZnO dispersion in HDPE [10]. Generally, the agglomerates deteriorate the mechanical and aesthetic properties, except for some special cases where the non-uniform distribution of the components is needed to reach a certain property such as in the works of Li et al. [11], who investigated the breaking properties of PP blends.

In the examination of injection molded parts, the results of evaluation methods based on sample variance of the concentration of components unfortunately do not correspond to human visual inspection – which is currently the most widely used method in industrial applications. Furthermore, it is very difficult to measure the concentration of the coloring agents if the homogeneity of the molded samples need to be quantified. Therefore in many industrial applications a digitized image of the sample is evaluated with various techniques [12-14]. In the available literature there are a lot of different methods used to calculate the homogeneity of images. According to H.D. Cheng et al [15] these methods can be put into the following categories: edge value based methods (or edge detection) [16], standard deviation (or variance) based calculations [17-19] and entropy based calculations [20, 21]. Edge value based methods typically apply a special gradient operator on the pixels of the images. The most typically used gradient operators are the Sobel operator, the Laplace operator or the Robert operator. Usually they apply a certain

threshold, and use the gradient values above this threshold. This way the image can be segmented or certain formations can be detected. Entropy based calculations are usually used to evaluate image segmentation or contrast enhancements. Mixing quality is typically evaluated by standard deviation or variance based methods. There are also calculations which apply a certain combination of the above mentioned procedures [22].
A new evaluation method was published by Zsíros et al. [23], which was applied to injection molded parts. The calculations of the homogeneity levels have been executed in the Lab color system, since the Euclidean distances in this color space are proportional to human color difference perception [24]. It was shown that the results of this method correlate well with the results of human visual inspections, and its standard deviation is low enough for one to investigate the processes and changes taking place in injection molding which might have influenced color inhomogeneity. To demonstrate this capability of the developed measurement system, Zsíros et al. [23] measured the effect of different injection molding parameters on color inhomogeneity. They also applied this evaluation method to compare the mixing efficiency of different mixing equipment used in injection molding [25]. Although this method seems to work well, it was only applied to one specific color, therefore it is recommended that it is tested and calibrated to a wider range of colors as well.
Based on the introduction the aim of this paper is to calibrate the novel inhomogeneity measurement method developed by Zsíros et al. [23] with various colors and to measure the differences between the homogenization properties of different masterbatch recipes. If the mixing properties of different masterbatches are known (how difficult it is to mix them homogenously into the base material), the exact need for mixing efficiency could be estimated more precisely.

2. Materials and methods

An Arburg Allrounder Advance 370S 700-290 machine (with a screw diameter of 30 mm) was used to produce 80 mm by 80 mm, 2 mm thick flat specimens. An HP Scanjet G4010 flatbed scanner was used to digitize the samples. The scanner was tested with different resolutions from 100 DPI to 600 DPI, and it was found that above 200 DPI the calculated homogeneity values did not change, but it took considerably longer to calculate higher resolution images. Therefore in further tests all images were digitized at 200 DPI. The images were evaluated with a software that we developed ourselves. It applies the calculation method published by Zsíros et al. [23].

Color plates were injection molded from 9 different masterbatches, and each of the masterbatches was tested with different processing parameters. Parameters were chosen that might have influence on inhomogeneity. The colors were red (R), yellow (Y), Orange (O), dark grey (DG), light grey (LG), green (G), blue (B), light yellow (LY), and pink (P). The test specimens were injection molded with the different masterbatches, which were mixed into ABS Styrolution Terluran GP 35. The various injection molding parameters can be seen in Table 1.

Table 1. Injection molding parameters

No.	Injection rate [cm^3/s]	Delay time [s]	Temperature [°C]
Series 1	55	0	225
Series 2	10	0	225
Series 3	100	0	225
Series 4	55	0	225
Series 5	55	10.5	225
Series 6	55	21	225
Series 7	55	0	190
Series 8	55	0	260

In the basic parameter set (Series 1) and in the control parameter set (Series 4) the injection speed was 55 cm^3/s, the delay time between the cycles was set to zero resulting in a residence time of 85 s, and barrel temperature was 225°C (Table 1). The other parameter sets were based on the first set but one specific parameter was modified at a time. Injection molding parameters that were not changed during the tests were clamp force set to 700 kN, and mold temperature set to 40°C. The switchover point was adjusted to reach the same filling (visual 99%) with each parameter combination.

Ten samples were produced with each parameter set. These samples were evaluated with the software and by six randomly chosen professional inspectors. Among the inspectors there were both males and females. The inspectors were instructed to evaluate the samples based on the scoring rule that a perfectly homogenous sample should have a score of zero – which theoretically does not exist – and the absolute worst sample should have a score of ten. For the correlation calculations the average of the six human scores was used.

3. Mathematical evaluation of color inhomogeneity

The masterbatch homogenization properties of all the masterbatches were evaluated with a new method developed by us [23]. The image analyzer software can characterize the color unevenness of injection molded products by using images of scanned samples.

Because the Lab color system approximates human vision, the RGB color coordinates of the images of the scanned samples were converted into the Lab color system (*P[L,a,b]*). During evaluation a moving window scans the picture, and at every position of this window the mean color coordinates are calculated. A matrix can be generated from the mean color coordinates as $\overline{\overline{A}}_{i,j,k}$ where the elements of the matrix can be calculated as follows (Eq. 6):

$$\overline{a}_{i,j,k} = \frac{\sum_{x=i}^{i+k-1} \sum_{y=j}^{j+k-1} P[L,a,b](x,y)}{k^2}, \quad (6)$$

where *i* and *j* are the position of the moving window within the whole picture, *k* is the width and height of the moving window, and *x* and *y* are the local coordinates within the moving window. The window size (*k*) could be varied from 2 to the maximum size of the picture (Figure 2).

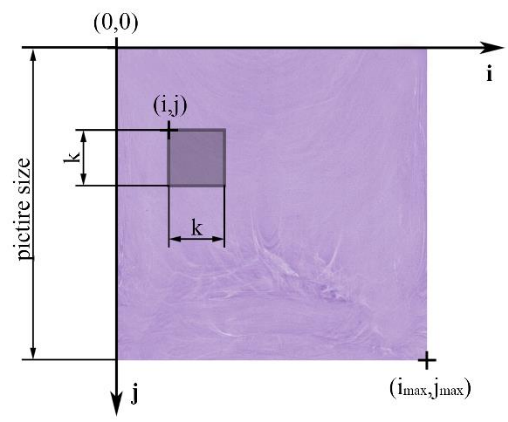


Figure 2. Parameters for the software calculations

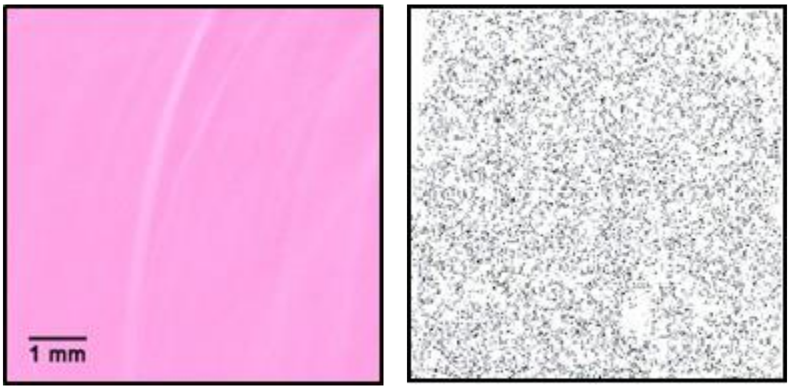
For all window sizes and positions the Euclidean distance of each pixel from the mean color coordinates were calculated, thus a 3 dimensional matrix (MD_k) can be generated (Eq. 7).

$$MD_{i,j,k} = \frac{\sum_{x=i}^{i+k-1} \sum_{y=j}^{j+k-1} \sqrt{\sum_{\varepsilon=L,a,b} \left\{P[\varepsilon](x,y) - A[\varepsilon](x,y)\right\}^2}}{k^2} \qquad (7)$$

The lower the $MD_{i,j,k}$ value is, the more even the color of the sample in the area covered by the moving window is. Moving the window pixel by pixel the software can locate the area having the highest MD_k value (HMD_k). If the size of the moving window is equal to the image size in pixels, a global MD value (GMD) can be obtained. The software calculates the HMD_k values for different window sizes and they can be compared to human evaluations.

4. Results and discussion

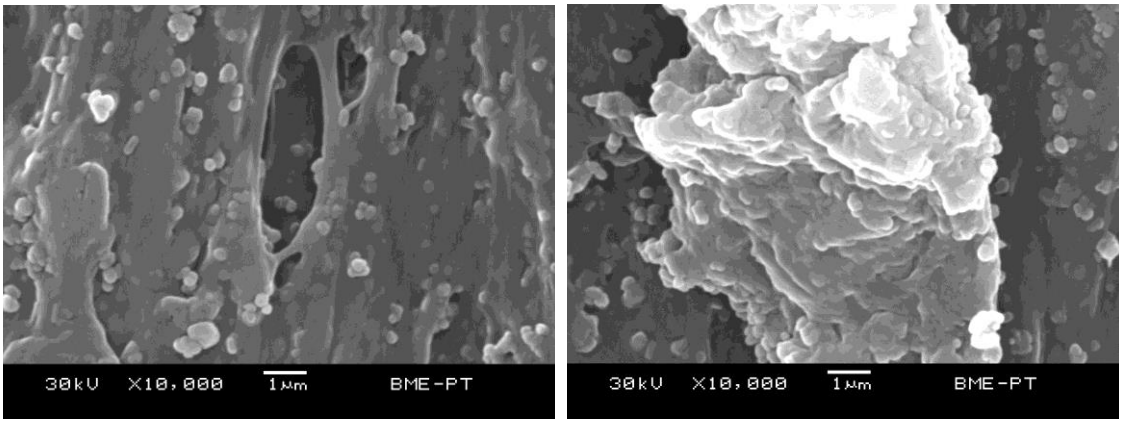
In the test specimens produced white stripes were visible in the case of certain masterbatches. To find the root cause of these color inhomogeneities, masterbatch samples were analyzed. Energy-dispersive X-ray spectroscopy (EDS) was performed with the help of a Jeol JSM 6380LA type electron microscope. The surfaces of the injection molded samples were investigated. The sample was coated with a very thin layer of Au/Pd alloy to avoid electrostatic charging. The EDS analysis of the samples showed the presence of Ti, which suggests that the samples contain a significant amount of TiO_2. This could be an explanation of the appearance of white stripes. Unfortunately, the results showed that the TiO_2 particles were evenly distributed around the area of the stripes (Figure 3) on the surface of the observed injection molded specimens.



A) B)

Figure 3. Microscopic (A) and EDS (B) analysis of the area of the stripes

Scanning Electron Microscopy (SEM) was performed on the cut surface of two different masterbatch pellets by using the same electron microscope as the one used or the EDS analysis with the same sample preparation method. Figure 4 shows two electron micrographs from the cut surfaces of two granules of different masterbatches. It can be seen that in certain areas the TiO_2 particles are well dispersed, while in other areas agglomerates can be found, which can be several microns or even tens of microns in size. Two pink masterbatches from different producers were examined for inhomogeneity and the presence of agglomerates in the electron micrographs. Unfortunately, while the color homogeneities of the injection molded samples from the two different masterbatches showed significant differences, both of them showed a similar number of agglomerates in the electron micrographs. Due to this fact, the idea of describing masterbatch homogenization abilities by the size, distribution and properties of agglomerates was discarded.



A) B)

Figure 4. TiO_2 in the ABS masterbatch: A) dispersed TiO_2 particles, B) TiO_2 agglomerate

Based on these results it was concluded, that the description of the homogenization properties of different masterbatches should not be based on agglomerate size distribution, but on something else.

In the case of the pink masterbatch the correlation of the HMD_k values with human evaluations changes as a function of the window size. Figure 5 shows that in the very small and very large window range – less than 5 pixels, and more than 250 pixels – the correlation decreases significantly. Window size did not influence correlation values between 5 and 250 pixels considerably, however, around window size of 28 pixels, correlation reached a maximum value of $R=0.86$ (Figure 5).

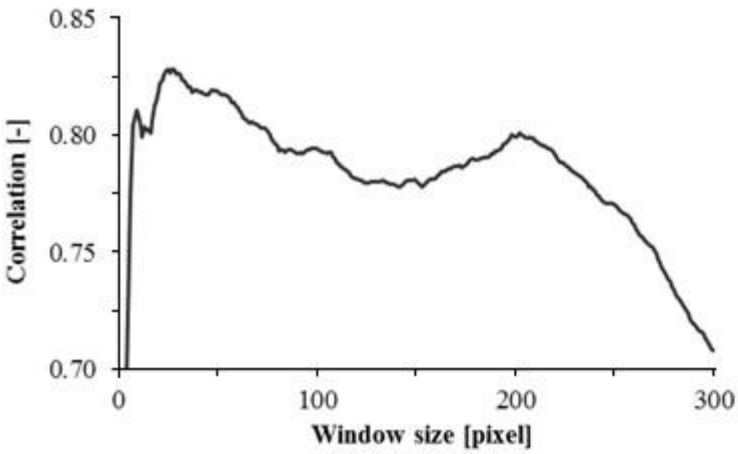


Figure 5. Correlation between human inspection and HMD_k values as a function of window size

Reference test specimens were injection molded from pre-colored materials. The reference tests showed that even though human eyes could not find any color inhomogeneities on the samples, the software showed a small, but consistent color inhomogeneity which resulted from the scanning process. These small color inhomogeneities from the pre-colored materials varied with the different colors. These values correlated well with the color inhomogeneity values calculated for the picture with the maximum window size (*GMD*). Therefore *HMD* values were corrected with *GMD* values to enable the comparison of the color inhomogeneity values of different colors. By depicting the corrected software scores (*CMD*=*HMD*-*GMD*) against the human scores (Figure 6), it can be stated that a correlation does exist, although not linear. In Figure 6 CMD values were calculated for 35 pixel window size, because CMD values showed a maximum correlation with human scores at this window size with a value of R=0.86.

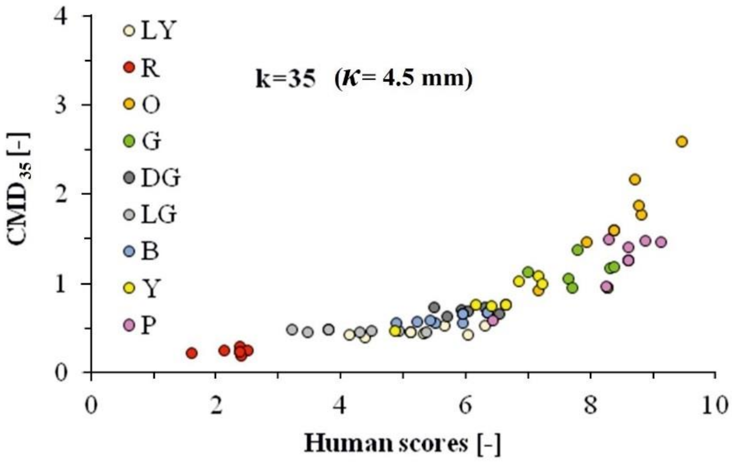


Figure 6. The corrected software scores of each color as a function of human scores

For a mathematical description of this non-linearity the software scores have been correlated to the human scores with different transformation functions. In the case of the tested scores, the logarithmic transformation showed the highest correlation coefficient (R) values (Figure 7). This finding is also in correlation with the Weber-Fechner law, which states that human sensation is proportional to the logarithm of the stimulus intensity [26].

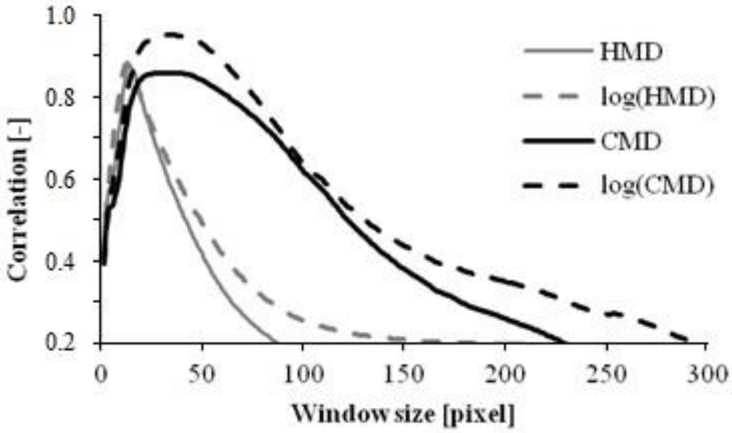


Figure 7. The software scores and the corrected software scores with and without logarithmic transformation

Correlation peaked at the window size of around 34-35 pixel, having a correlation value of *R*=0.95. In Figure 8 the *IHS* (inhomogeneity scores), which are a linear transformation of the corrected logarithmic values, were plotted as a function of the human scores. This linear transformation is represented in Eq. 8.

$IHS = C_1(log(CMD) + C_2)$, (8)

The constants C_1 and C_2 didn't have any effects on the correlation values. The aim of these constants were only to pull the *IHS* values to a similar scale as the human scores. The constant values, which are needed to make the scale of the software scores similar to that of human inspections, are dependent on several factors such as the noise and sensitivity of the flatbed scanner, and the color and inhomogeneity preferences of the human inspectors. In Figure **8** values $C_1=5$, $C_2=1$ were applied to calculate the *IHS* values (Eq. 8). Figure 8 illustrates well that human inhomogeneity sensation is proportional to the *IHS* (logarithm of the standard deviation of the Euclidean color differences in Lab color space).

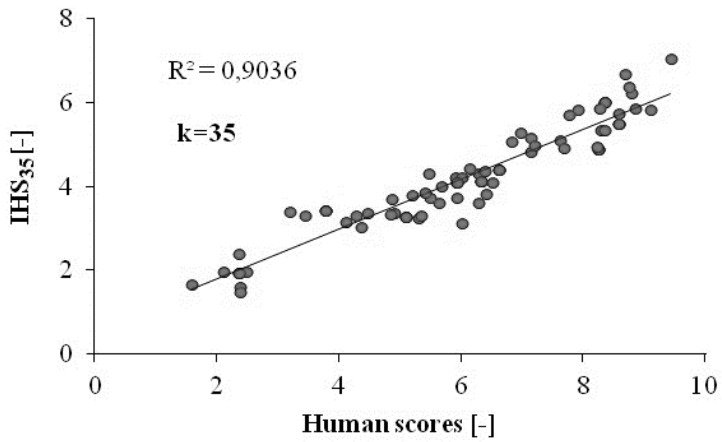


Figure 8. The correlation of the human scores with the logarithm of the corrected software scores at a window size of 35 pixels

Based on these findings, the *IHS* values at a window size of 35 pixels were used to characterize the homogenization properties of the different masterbatches. In Figure 9 the inhomogeneity levels of the different masterbatches are represented. The dark columns mean the minimum inhomogeneity levels, while the light columns represent the influence of the technological parameters on the different colors. This generally means that the higher the value is on this chart, the more difficult it is to produce homogenous injection molded parts from that masterbatch. Figure 9 also illustrates the influence of the technological parameters on color inhomogeneity in the case of the different masterbatches. It shows that certain masterbatches are more sensitive to the processing parameters, while others show certain inhomogeneity levels which are independent of the injection molding parameters, as in the case of two masterbatches of high inhomogeneity levels, such as the green and the pink masterbatches. It can be seen that the tops of the light bars represent an almost equal IHS, but the dark bar of the pink masterbatch is significantly shorter. This means that in the case of the pink masterbatch, the optimization of the injection molding parameters can improve homogeneity by much, while the inhomogeneity of the green masterbatch can only be modified by a little.

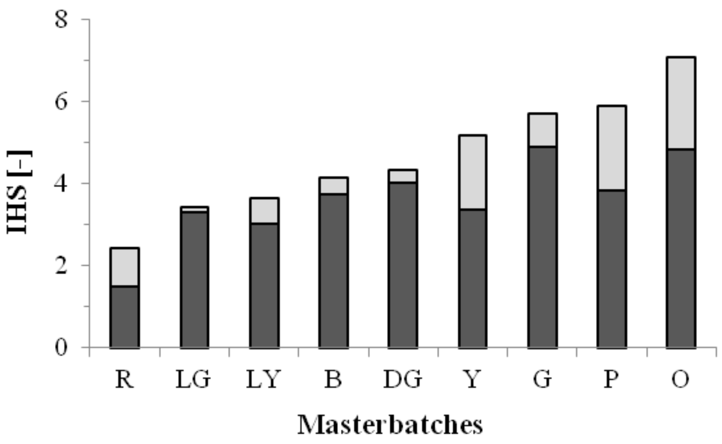


Figure 9. The minimum levels (dark bars) and the influence of technological parameters (light bars) on color inhomogeneity of the test specimens colored by different masterbatches

The worst masterbatches considering homogenization are the orange, the pink, the green and the yellow masterbatches. On the other end of the scale are the red, the light yellow, and the light gray masterbatches.

To analyze the homogenization property differences of the masterbatches nine different monobatches were investigated. Each of these monobatches contained only one type of colorant and the carrier. The colorant types and their concentrations can be seen in Table 2.

Table 2. The colorant types and their concentrations in the investigated monobatches

		Number of sample								
		1	2	3	4	5	6	7	8	9
Red	dye	1.00%								
	organic		5.00%							
	Mono 1			10.00%						
	Mono 2				10.00%					
	inorganic					10.00%				
Blue	dye						1.00%			
	organic							5.00%		
	Mono								10.00%	
	inorganic									10.00%

The results of this measurement can be seen in Figure **10**. Some differences can be noticed between the components. When these differences are compared to the variations experienced between the different masterbatch recipes in Figure 6, it was concluded that the differences between the various masterbatch recipes cannot be explained by the homogenization properties of their components.

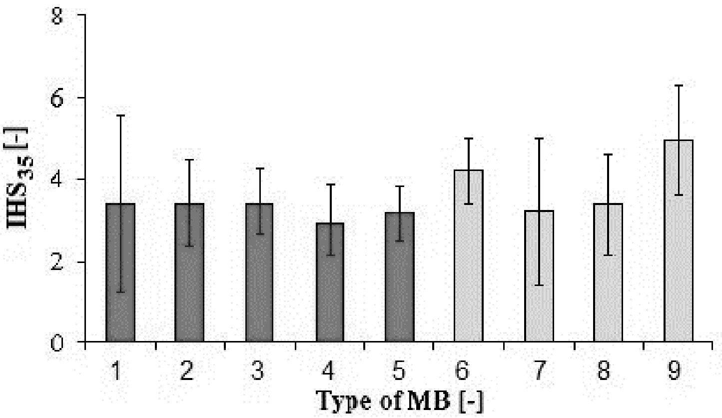


Figure 10. The homogenization properties of the monobatches

The effects of the interactions of the components were tested by altering the recipe of the pink masterbatch, which was tested earlier. In the test series only one component or its concentration in the masterbatch was modified. The variations of the original pink recipe can be seen in Table 3. As can also be seen, sample No. 5 is identical to sample No. 1, except for granule size. Since the comparison of the results did not show any significant differences, it can be stated that granule size in masterbatches does not affect color homogeneity.

Table 3. Variations of the recipe of the original pink masterbatch

Sample Nr.	TiO_2		Organic [%]	Inorganic [%]	Dye [%]	Wetting agent [%]	Additive		Granule size
	Type A [%]	Type B [%]					Type	[%]	
1	-	35	-	<0,5	<0,5	2-3	A1	0,5	STD
2	-	40	0,5-1	-	-	2-3	A1	0,5	STD
3	35	-	-	<0,5	<0,5	2-3	A1	0,5	STD
4	40	-	0,5-1	-	-	2-3	A1	0,5	STD
5	-	35	-	<0,5	<0,5	2-3	A1	0,5	Mini
6	43	-	0,5-1	-	-	-	-	-	STD
7	10	-	-	<0,5	<0,5	2-3	A1	0,5	STD
8	12	-	0,5-1	-	-	2-3	A1	0,5	STD
9	40	-	0,5-1	-	-	10	A1	0,5	STD
10	35	-	-	<0,5	<0,5	2-3	A2	0,5	STD
11	-	-	-	-	1,5-2	2-3	A1	0,5	STD
12	-	-	6-7	-	-	2-3	A1	0,5	STD
13	35	-	-	<0,5	<0,5	2-3	A3	0,5	STD
14	35	-	-	<0,5	<0,5	2-3	A1	7-9	STD

Generally, it can be stated that the type of TiO_2 and the applied various additives had no effects even if applied in higher amounts, but the organic pigment-based recipes were systematically better than the solvent-based ones with both TiO_2 types (Figure **11**). The significance of the differences were proved by two-sample t-test.

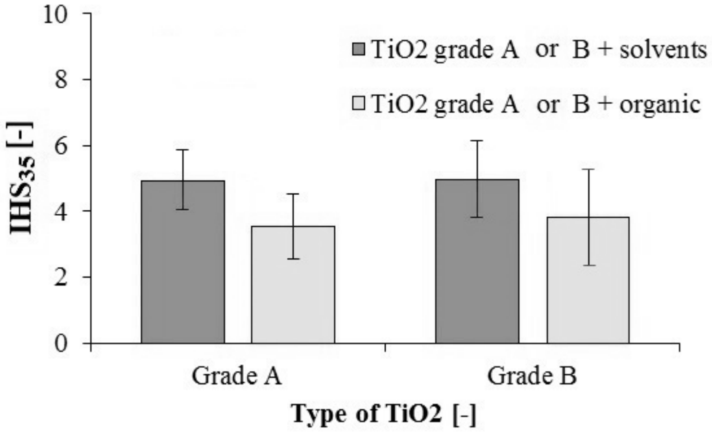


Figure 11. The effect of organic pigments on color homogeneity in the case of various TiO_2 grades

The organic-based recipe was further investigated by altering the amount of wetting agent in the case of the grade A TiO_2 based version. It can be seen from Figure 12 that higher amounts of wetting agent resulted in lower inhomogeneity scores. Although tests were executed only on grade A TiO_2 based masterbatches, it can be assumed from the results seen in Figure **11** that grade B TiO_2 recipes would show very similar results if they were tested. Furthermore, it can be stated that the lowest inhomogeneity scores were obtained in the case of the organic pigment-based recipe with the highest amount of wetting agent.

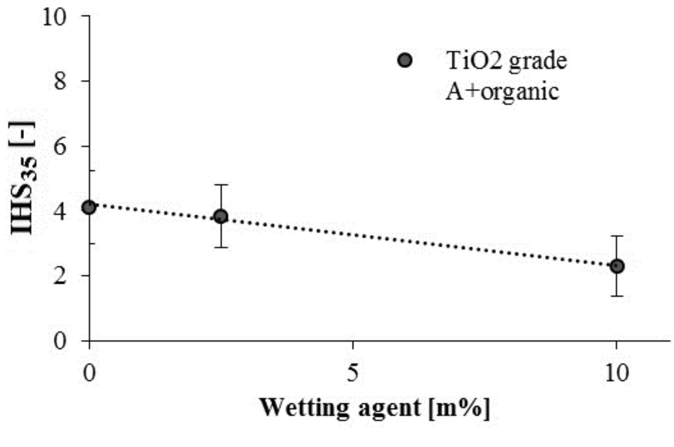


Figure 12. The effect of wetting agent on color homogeneity in a TiO_2 and organic pigment based masterbatch

5. Conclusions

It was shown in this paper that the inhomogeneity scores – calculated with the method developed by us earlier – correlate well with human evaluations even in the case of masterbatches with large color differences. This could be further improved with the logarithmic transformation of the corrected software scores (R=95%). This high correlation in the case of more than 1000 evaluations proves that human inhomogeneity sensation is proportional to the logarithm of the standard deviation of the Euclidean color differences in the Lab color space. This finding is in line with the well-known Weber-Fechner law, which states that human sensation is proportional to the logarithm of the stimulus intensity [26].

It was also shown that an objective characterization of different masterbatches is possible with this method. Nine masterbatches were evaluated and compared to each other. The masterbatches substantially differed in color, recipe and thus in homogenization properties as well. These masterbatches showed different inhomogeneity changes as the processing parameters changed. While some masterbatches were quite sensitive to injection molding conditions, others showed little or negligible changes.

Furthermore, it was shown that in the case of various masterbatch recipes, it is not the individual homogenization properties of the components which drive the final properties of the masterbatches, but the interactions between the different components. It was also concluded that among the tested recipes the organic pigment-based masterbatch containing the highest amount of wetting agent had the best homogenization properties.

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